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KEYNOTE ABSTRACTS

1 Fatigue life extension by crack repair using double stop-hole technique

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Drilling holes in the vicinity of the crack tip turns the crack into a notch and reduces the crack tip stress intensity factor. In this paper a new idea is used, in which instead of a single hole, two symmetric and interconnected holes are drilled at the crack tip. The main concept of double stop-hole method is to diminish the stress singularity at the crack tip and also to reduce the stress concentration at the edge of stop holes in the cracked structural elements. The double stop-hole method can be used to increase the fatigue life of cracked components. The fatigue crack growth retardation is examined using a combined experimental-numerical investigation on the efficiency of proposed double stop-hole method. The distance between the hole centers is considered as the main parameter affecting the efficiency of this method. Pure mode-I loading conditions have been applied to the single edge-notch tension (SENT) specimens made of high strength steel S690 according to the EN 10025 standard. The numerical and experimental results both show that the fatigue life extension caused by the double stop-hole method is significantly more than the conventional single stop-hole method.

2 Intermetallic phases in new steels

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Al containing intermetallic phases have been investigated with respect to their application potential in various bcc and fcc steels. In hot working tool steels attractive properties have been obtained, meeting industrial requirements on one hand and reducing resource critical alloying elements on the other hand. In high C bearing steels, Al alloying leads to the formation of kappa-carbides and a reduction of density; the benchmark properties of conventional bearing steel could not been met because of the limitation for the heat treatment conditions. Al alloyed low carbon steels offer a significant density reduction and reasonable formability after cold rolling and batch annealing. In high Mn steels, the formation of the kappa phase have been monitored by synchrotron X-ray diffraction. The very regular arrangement of nm size precipitates results in the MBIP phenomenon (MicroBand Induced Plasticity). These precipitates contribute to a significant strengthening, while the ductility and the strain hardening remain high. It is concluded that Al-containing intermetallic phases provide an interesting tool for the design of new steels. The adjustment of the properties follow different design rules. A thorough understanding of the boundary conditions for the intermetallic phase formation is unavoidable.

3 Synergistic action of hydrophobic and hydrophilic zirconium phosphate nanofillers for efficient mechanical reinforcement of perfluorosulfonic acid membranes

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Improvement of mechanical properties of fuel cell membranes has recently been recognized as an important factor to avoid the membrane premature failure, caused by the stress the membrane has to withstand as a consequence of hydration changes during fuel cell operation. In this work we investigated the possibility of using a double filler, made of a hydrophilic and a hydrophobic component, to improve the mechanical strength of a perfluorosulfonic acid ionomer (PFSA), without prejudicing the ionomer transport properties. To this aim, nanosized zirconium phosphate (hydrophilic component) and its organically modified derivative bearing fluorinated alkyl chains (hydrophobic component) were used as the double filler of a short side chain PFSA. In comparison with the unmodified ionomer, the membrane loaded with the double filler has similar proton conductivity, in spite of lower hydration, and significantly improved mechanical properties, with an increase up to 300% in the elastic modulus and up to 95% in the yield stress. The double filler membrane shows better fuel cell performance as well in terms of higher OCV, lower hydrogen crossover and greater power density both at 80 and 110 °C in hydrogen/air (0.82 and 0.70 W cm⁻², respectively, with 50% relative humidity). Interestingly, the enhancement of mechanical properties achieved with the double filler is much greater than that observed for the best composite membranes loaded only with the hydrophilic or the hydrophobic component, thus showing the synergy between the two filler components.

4 Revisiting the hardening precipitates in high strength aluminum alloys by atomic-resolution electron microscopy

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Developments of high-strength aluminum alloys have always faced a difficult problem: owing to their small size, the early-stage strengthening precipitates are difficult to characterize in terms of composition, structure and evolution. Here we employ atomic-resolution transmission electron microscopy (TEM) imaging and first-principles energy calculations to address these problems. Recent years, we have investigated tens of typical high strength aluminum alloys, such as 2xxx (AlCuMg) and 7xxx (AlZnMgCu) alloys, with different compositions and with varying thermal processes for their property-structure-process correlations. Using advanced aberration-corrected high-resolution TEM (HRTEM) and aberration-corrected scanning TEM (STEM), much of our attention has been paid to revisit the strengthening precipitates in these important alloys and to clarify the controversies left in the past about their precipitation behaviors. Our study demonstrates the followings:

(1) Atomic-resolution imaging in HAADF-STEM can provide straightforward structure models at the atomic-scale, whereas atomic-resolution imaging in HRTEM with rapid quantitative image simulation analysis can provide the refined structures with high precision beyond the

resolution limitation of the microscope. The combination of the two techniques can be more powerful in solving difficult structure problems in materials science. (2) Most of the early-stage precipitates are highly dynamic in both composition and structure. Typically, having their characteristic genetic skeletons to guide their evolution, these dynamic precipitates initiate, mature and grow with thermal aging following characteristic evolution paths. The fine precipitation scenarios revealed are rather different from previous understandings in the textbooks and literatures published thus far.

5 Advanced engineering intermetallic titanium aluminides

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After almost four decades of intensive fundamental research and development activities intermetallic titanium aluminides have found application in automotive and aircraft engines. The advantage of this class of innovative high-temperature materials is their low density in combination with good strength and creep properties up to 800°C. A drawback, however, is their limited ductility at room temperature, which is reflected in a low plastic fracture strain. Advanced engineering TiAl alloys are complex multi-phase materials which can be processed by ingot or powder metallurgy, precision casting methods as well as additive manufacturing, e.g. electron beam melting. Each production process leads to specific microstructures which can be altered and optimized by thermo-mechanical processing and/or subsequent heat-treatments. The aim of these heat-treatments is to provide balanced mechanical properties, i.e. sufficient ductility at room temperature as well as creep strength at elevated temperature. In order to achieve this goal, the knowledge of the occurring solidification processes and phase transformation sequences is essential. To account the influence of deformation and kinetic aspects sophisticated ex- and in-situ methods have been employed to investigate the evolution of the microstructure during thermo-mechanical processing and subsequent heat-treatments. Summarizing all results a consistent picture regarding processing and mechanical properties of advanced engineering intermetallic TiAl alloys can be given. Finally, future TiAl development strategies will be outlined.

6 Mapping the precipitation kinetics in compositional space: a combinatorial approach to microstructure characterization

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Alloy design traditionally relies on the cumbersome, time-consuming fabrication a discrete set of alloys with different compositions, followed by their processing and characterization. With such a methodology, finding an optimum of alloy composition in a complex engineering material is a formidable challenge, because of the interactions of chemical composition and microstructure evolution during the thermo-mechanical processing steps. The presentation will show how combinatorial approaches can succeed in mapping the microstructure development in compositional space. The methodology involves first to fabricate compositionally gradient

materials with a gradient extending over several mm, e.g. using Linear Friction Welding, followed by high temperature diffusion treatments and rolling.

The second and step is to characterise the microstructure evolution with the combination of time and space resolution. When studying precipitation, we will show that Small-Angle X-ray Scattering answers these two requirements. In the first case study, the effect of Co content on the precipitation of Co in Cu will be demonstrated. The precipitation kinetics map in compositional space is used to compare with a precipitation model. This comparison will make it possible to assess the robustness of the model and will provide with values of its physical parameters. In the second case study, we will demonstrate the effect of Mg on the precipitation sequence and associated kinetics in an Al-Cu-Li alloy. The access to a full range of Mg concentrations and associated precipitation kinetics makes it possible to determine how much of Mg is necessary to change the precipitation sequence in this alloy.

7 Application of the additive manufacturing by selective laser sintering for constituting implant-scaffolds and hybrid multilayer biological and engineering composite materials

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The keynote lecture is focusing on the special micro and nanocomposite materials designed mainly for use in regenerative medicine and regenerative dentistry. The hybrid additive technologies of structure and properties formation of newly created metal microporous materials (100-600 nm). For forming the newly obtained implant-scaffolds is applied the selective laser sintering/laser melting (SLS/SLM) combined with chemical treatment, by surface etching, and an innovative manufacturing technologies of composite materials by depositing an internal surface of micropores. Completely innovative implant-scaffolds were developed, which are comprised of a solid zone and a microporous hybrid zone fulfilling the functions of scaffolds ensures appropriate osteosynthesis of bone implants with bone stumps, enables the living tissue to outgrow across the porous zone after implantation, creating a durable and firm joint of an implant with a living tissue. An innovative generation of rigid and elastic hybrid multilayer biological and engineering composite materials for regenerative medicine were developed which consist of at least 4 layers: a layer of cultured living cells, a layer of engineering biomaterial made of metallic, ceramic or polymeric material, also of polymer nanofibers, intelligent amyotrophic/transition layer or a few of them degraded or detached under the influence of one or several active environmental factors controlled by a therapist from a layer of the cultured biologically active cells once the entire composite fulfils the therapeutic function, and an external textured layer of an engineering material providing more space for cells culturing and enabling to create three-dimensional cellular structures.

8 Phase formation and properties of advanced metastable metallic materials

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A drawback of nearly all current high strength metallic materials is that they lack ductility (i.e. are brittle and hard to form) - or on the opposite side, they may be highly ductile but lack strength. Hence, it is mandatory to develop new routes for creation of tailored metallic materials based on hierarchical hybrid structures enabling property as well as function optimization. One starting point along these lines is the design of monolithic amorphous materials or bulk micro-, ultrafine- or nano-structured composite structures with intrinsic length-scale modulation and phase transformation under highly non-equilibrium conditions. This can include the incorporation of dispersed phases which are close to or beyond their thermodynamic and mechanical stability limit thus forming hierarchically structured hybrid and ductile/tough alloys. Alternatively, the material itself can be designed in a manner such that it is at the verge of its thermodynamic/mechanical stability.

This talk will present recent results obtained for metallic glass-based hybrid structures with transformation effects at different length-scales and microcrystalline-grained hybrid structures based on elastic instabilities and modulated length-scale. The deformation behaviour and possible phase transitions during deformation will be related to the intrinsic properties of the phases as well as the microstructure of the material including heterogeneities and length-scale modulation in order to derive guidelines for the design of macroscopically ductile high-strength materials. Finally, the results will be critically assessed from the viewpoint of possible scaling-up for technological applications and the use of simple and cost effective processing technologies.

9 Spark plasma sintering of ceramic powders: From evidence of specific effects to the elaboration of complex architectures and shapes

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Pulsed Electric Current Sintering (PECS) techniques have known a huge development over the last two decades. In particular, SPS is an extremely powerful technique to sinter all classes of materials as well as their composites.

In recent investigations on different ceramic powders (LiF, NiO, and YAG) local melting of the particle and nano-particle surfaces have been clearly shown, confirming the formation of spark and plasma during the SPS. Due to the lower temperature and the shorter durations achieved during SPS, interdiffusion or reaction between two adjacent materials can be limited, favoured or controlled. The potentialities of this technique to design new materials or architectures will be illustrated through few examples on layered multimaterials.

Recently, the modeling of Spark Plasma Sintering by finite element method has known drastic development. Coupling three main physics, Electric Thermal and Mechanic (ETM), it allows now to predict the temperature, grain size and porosity during the process. The electrical and thermal parts of the ETM model are used to calculate the temperature at any point of the SPS

tool and column. Sintering models (Olevsky and Abouaf) are used to predict the densification of an alumina nano-powder. Last, a grain growth law was coupled with Olevsky's densification model which has shown the great effect of the grain growth at the end of the sintering stage. Finally this type of modeling allowed us to define the optimized SPS parameters and tool geometry in order to minimize the porosity gradient in a complex shape part.

10 Metal matrix composites developed by severe plastic deformation: challenges and stakes

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The present talk will focus on the specific features of the processes of severe plastic deformation (SPD) in order to elaborate Metal Matrix Composites (MMCs). The problematic will be illustrated by various MMCs developed by different thermomechanical processes.

MMCs are structural and functional materials which are already widespread in various industrial sectors. However, the MMCs stakes lie in the development of novel materials with advanced properties for ever more demanding applications. These necessary levels of performances are governed by the respective nature of the components, by the uniformity and fineness of the composite architecture and by the quality of the component interfaces. The SPD elaboration processes present some advantages essentially due to their solid state nature and the occurrence of mechanical alloying which pave the way for promising out-of-equilibrium materials. Concerning MMCs elaborated by SPD, the final composite design results not only from the choice of the adjoining constituents which controls both their chemical affinity and their gap of mechanical behavior but also from their initial layout and their processing conditions. These advantages and challenges, and the problem of the thermomechanical stability of the composites elaborated by SPD will be demonstrated. The development of MMCs is rendered easier by the continuous progress in the procedure of materials selection and by the improvement of the elaboration processes assisted by always more powerful techniques of numerical simulation and of microstructural characterization.

At last, the MMCs promises are all the more extended that their use is aided by the development of novel processes.

11 Effect of high temperature deformation on the texture development in alloys and oxides

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Texture control is one of the key technologies to achieve high performance materials, especially the materials with high anisotropy in properties. Deformation texture and recrystallization texture are well known textures, which are produced by cold working and the combination of cold working and annealing, respectively. Since the recrystallization texture can be related to the deformation texture, how to develop deformation texture has been the basic issue.

Deformation texture varies primarily depending on deformation mode and slips system, hence, once material and deformation mode are chosen, the outline of the texture is fixed. This means the difficulties in producing new textures by cold working or the combination of cold working

and heat treatment. As a process to produce new textures, high temperature deformation is promising, because not only new slip systems might appear but also additional deformation mechanisms such as grain boundary sliding and dynamic recrystallization might be activated. In addition, the activation of these kinds of deformation mechanisms might make it possible to produce textures in the materials which cannot be deformed at room temperature such as intermetallic compounds and oxides. Furthermore, dislocation structures different from room temperature deformation can be given in some kinds of solid solution alloys, which vary the levels of stored energy in the alloys. In this presentation, the effectiveness of high temperature deformation to produce textures in aluminum based and iron based solid solution alloys and oxides is introduced. Effects of texture on the improvement of properties are also shown.

12 Strengthening of low alloy steel by nano-scale precipitation of alloy carbide/nitride

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Recently, interphase precipitation during ferrite transformation attracts lots of attentions as a promising microstructure for high strength and high ductility in sheet steels. Furthermore, surface hardening by internal nitriding is widely used for improvement of wear resistance of machinery parts with precipitation. In both cases, microalloying of strong carbide/nitride forming elements is effective for precipitation strengthening of ferrite matrix. In recent years, the present authors have made efforts to characterize nano-scale precipitation of such alloy carbide/nitride in low alloy steels. In this presentation, characteristics of nano-scale precipitation and resulting mechanical properties are discussed.

Alloy carbide dispersion obtained by interphase precipitation is finer as ferrite transformation temperature is lowered or the amount of microalloying is increased. It has been confirmed that effective nucleation sites of carbide are ferrite/austenite interphase boundaries with irrational orientation relationships. Such carbides are responsible for Orowan-type dispersion strengthening of ferrite without suffering ductility. In surface hardening, small addition of titanium or vanadium results in formation of metastable clusters before stable nitride precipitation. It has been demonstrated that addition of strong nitride-forming element induces precipitation of relatively weaker nitride-forming elements such as aluminum or chromium. Thus, combined addition of those elements results in substantial increase in surface hardening.

13 The formation of kink bands in a Mg alloy with synchronized LPSO structure

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Mg alloys with synchronized long period stacking order (LPSO) structures exhibit unique mechanical properties in strength and ductility. When they are subject to compressive deformation along the direction nearly parallel to the basal plane, abrupt kinking of basal plane occurs to form a microstructure called "kink bands". The formation of kink bands is often observed in crystalline materials with highly anisotropic plasticity, and it is not only a plastic deformation mechanism but also regarded to be an essential mechanism enhancing the strength

of LPSO phase since the activation of basal slip should be prevented by the kinking of basal plane. However, the deformation behavior and strengthening mechanism of kink bands have not been fully clarified yet. In the present study, the detailed morphology and formation behavior of kink bands were examined, and then the strengthening mechanism is discussed. Particular attention is paid to the distribution of plastic strain around kink bands.

14 Joining techniques by sintering of nanoparticles derived from metal oxides

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High specific surface energy of nanoparticles lowers sintering temperature, and enables low-temperature sintering joining of materials using nanoparticles as a filler material. After joining the joint has superior high-temperature properties coming from the high melting point of sintered layer. We have achieved joining of Au, Ag, Ni and Cu using Ag nanoparticles as a filler material. To improve the bondability more, and to reduce the cost of the filler material, we have developed a new bonding process using Ag₂O paste consisting of Ag₂O particles mixed with a reduction solvent. Ag nanoparticles formed at approximately 130°C through the reduction of Ag₂O. The Ag nanoparticles were immediately sintered to one another and bonded to a metal substrate. The Ag₂O paste was applied to joining of Au, Cu and Al. Au and Cu were successfully joined through the sintered Ag layer without any oxide layers at the bonding interfaces. On the other hand, the Al was bonded to the sintered Ag layer through a thin Al oxide layer. Therefore, Ag nanoparticles derived from reduction of Ag₂O were found to be bonded to the aluminum oxide. We also applied a CuO paste to Cu-to-Cu joining as a filler material. Cu nanoparticles formed from the reduction of CuO particles by a reduction solvent, and Cu-to-Cu joining was achieved through the sintering of Cu nanoparticles. The Cu substrate was directly bonded to the sintered Cu layer.

15 Precipitates in Al alloys across and between industrially common compositions

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The morphology, structure and strengthening properties of age-hardening precipitates depend on alloy composition and thermo-mechanical history of the material. 2xxx, 6xxx and 7xxx aluminium alloys get the majority of their strength from precipitation hardening, but have very different precipitation sequences. The main hardening phases in the three systems are theta'/S, beta''/Q' and eta', respectively. These are coherent with the aluminium matrix along different planes and directions and form plates and/or needles. Alloys which are between two systems are referred to as hybrid alloys. The effects of cross-system substitutions in these three systems are investigated and compared, regarding solute influence on precipitate distribution, structure, interfaces and morphology. When adding zinc to 2xxx alloys, this element will go into the S phase and eventually form eta type particles. When instead adding silicon, the S particles are replaced with disordered needle shaped particles based on the silicon network. With all

elements combined in an alloy, silicon seems to be the element which controls the precipitation sequence. In such an alloy, upon overaging, the disordered needles act as nuclei for six different phases from all three alloy systems. In general, we see that disordered precipitates are the most efficient for increasing materials strength. The main technique we use is advanced (aberration corrected) transmission electron microscopy including high angle annular dark field scanning TEM imaging. Stability of the structures in the hybrid alloy systems is investigated through ab-initio calculations.

16 Improving creep resistance of magnesium alloys

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Magnesium alloys are one of the first choices when light weight design is of importance for applications. Especially in the area of transportation, weight reduction becomes important to reduce CO₂ emissions, to improve fuel efficiency or to increase either range or payload. Mg applications are already fairly well introduced in numerous applications but using Mg at elevated temperatures and under stress remains still a challenge. However, there are suitable Mg alloys available as well as metal matrix composites based on Mg alloys. This contribution will give a review on the state of the art on creep resistant Mg alloys. Furthermore recommendations for alloy development will be given to make Mg alloys competitive to other materials e.g. Al alloys.

17 Development and research of low-cost titanium alloys, especially case of Japan

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Titanium exhibits many attractive properties. It is considered to be ubiquitous since it has the 9th-highest Clarke number of all the elements. However, the principal beta-stabilizing elements for titanium can be very expensive, e.g. Mo and V, making many titanium alloys expensive. Iron and Manganese are beta stabilizers for titanium alloys and it is also considered to be ubiquitous since they have the 4th-highest and 11th-highest Clarke number of all the elements, respectively. Furthermore, since iron has higher diffusion coefficient in beta phase of titanium, precipitation of omega phase becomes faster by iron addition. The behaviors of Ti-Mn and Mn-Fe alloys during heat treatment have been investigated and it was found that in some alloys the isothermal omega phase is precipitated. Because this phase can lead to brittleness, it is very important to suppress its precipitation. We investigated the effect of adding aluminum using Ti-8.1mass% Mn-1mass% Fe-0, 1.5, 3.0 and 4.5mass% Al alloys by performing electrical resistivity, Vickers hardness, and X-ray diffraction measurements. In solution-treated and water-quenched all alloys, only beta phase was identified. The resistivities at room and liquid-nitrogen temperatures were found to increase monotonically with increasing Al content, while Vickers hardness decreased up to 3mass% and then kept that value. Isothermal omega precipitation was suppressed by aluminum addition, while alpha precipitation was accelerated by Al addition. Moreover, I also describe an investigation of the influence of the Mn content on the phase constitution and heat-treatment behavior of Ti-8 to 10Mn-1Fe-3Al (mass%) alloys.

18 What controls temperature dependence of yield stress in L1₂-ordered intermetallic compounds?

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Deformation of L1₂ compounds is usually carried by $\langle 110 \rangle$ dislocations gliding on (111) octahedral or (001) cube slip planes. $\langle 110 \rangle$ dislocations are known to dissociate into partials in two different schemes; (i) the APB type with $1/2\langle 110 \rangle$ dislocations separated by an APB and (ii) the SISF type with $1/3\langle 112 \rangle$ type partials separated by SISF. In the APB type dissociation, further sub-dissociation of each of $1/2\langle 110 \rangle$ superpartials is possible, involving a CSF in between. Which of the two is preferred is considered to be determined by the relative energy of APB and SISF on the octahedral plane. More importantly, dislocation dissociation schemes are believed to determine the temperature dependence of yield stress through the core effects. While the core of $1/2[110]$ dislocations dragging APB on octahedral plane is believed to be planar, the core of $1/2[110]$ dislocations dragging APB on cube plane as well as that of $1/3[121]$ dislocations dragging SISF on octahedral plane is believed to be non-planar, resulting in a rapidly decreasing yield stress with the increase in temperature. However, our recent study clearly indicates that the SISF dissociation does not exist even the SISF energy is significantly low. While the APB energy anisotropy and elastic anisotropy determines the presence or absence of yield stress anomaly, the CSF energy controls deformation of L1₂ compounds carried by APB-coupled superpartials. The classification of L1₂ compounds in terms of the temperature dependence of yield stress is discussed in relation to the CSF energy and the APB energy anisotropy and elastic anisotropy.

19 An artistic approach to thermal spray

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“Art is everywhere” to quote Ben, a renowned French contemporary artist. However, there are some areas in which art is more prevalent. Thermal spray is one of them, as this presentation seeks to demonstrate. For this, each of the arts (according to their official classification) is shown to correspond to a specific key point of the thermal spray process for coating: e.g., coating build-up, additive manufacturing, deposition onto brittle and/or temperature sensitive materials (glass, wood, fabrics, polymers), powder optimization, and adhesion. Both modeling and experimental aspects are discussed, focusing on the study of particle-to-particle or particle-to-substrate interfaces, shock phenomena and advanced investigation techniques such as X-ray microtomography or high-speed instrumentation. Plasma spray and cold spray provide the relevant examples that this contribution elaborates. They relate to different industrial sectors such as aircraft-aerospace, luxury, biomedical and the automotive industry. Beyond anecdotal evidence, the discussion aims to show that an artistic approach to thermal spray does help to understand better this powerful coating process.

20 Dynamic transformation during plate and strip rolling

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Torsion simulations were carried out of both plate (long interpass times) and strip (short interpass times) rolling. Both isothermal and continuous cooling conditions were employed. The dynamic transformation of austenite to ferrite was observed under all conditions and at all temperatures within the austenite phase field. The critical strains for the initiation of dynamic transformation were determined by double differentiation. These fell in the range 5 - 10 %. About 8 to 10 volume percent ferrite was formed in a given pass, leading to about 40 - 60 % ferrite at the end of selected simulations. During the interpass intervals, some retransformation to austenite took place, the amount of which increased with holding time and temperature and decreased with the addition of alloying elements. The implications with respect to rolling load (i.e. mean flow stress) and microstructure (carbon partitioning) are also discussed.

21 The role of microstructure in creep strength of 9-12%Cr steels

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Tempered martensite lath structure (TMLS) plays a vital role in creep resistance of 9-12%Cr steels. Under creep conditions the TMLS is stabilized by three agents: (i) a dispersion of boundary $M_{23}C_6$ carbides and Laves phase; (ii) a dispersion of $M(C,N)$ carbonitrides homogeneously distributed within ferritic matrix; (iii) substitutional alloying element within ferrite. The boundary particles exert Zener drag force which effectively hinders boundary migration. A dispersion of $M(C,N)$ carbonitrides hinders dislocation glide and climb that suppresses knitting reaction between lattice dislocation and low-angle boundaries. This reaction is responsible for decreasing long-range elastic stress field originated from lath boundaries. In addition, $M(C,N)$ carbonitrides provide high threshold stress. Substitutional elements as W and Mo effectively slowing down diffusion in ferritic matrix retard climb of lattice dislocation that also prevents the aforementioned knitting reaction. Depletion of W and Mo from solid solution leads to the occurrence of static recovery and precipitation of Laves phase at boundaries under long-range aging. This process is responsible for creep strength breakdown. Under creep the formation of Z-phase at the expense of V-rich $M(C,N)$ carbonitrides decreases threshold stress and leads to the transformation of lath boundaries to subboundaries. Well-defined subgrain structure appears instead of TMLS. Ostwald ripening of boundary $M_{23}C_6$ carbides and Laves phase decreases Zener drag force and migration of subgrains starts to occur. The relation of these processes with material softening and creep strength is discussed.

22 LPSO structure and its related high strength magnesium alloys

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Magnesium alloys are very attractive in such applications as automotive, railway and aerospace technologies. However, their low ignition temperature and low mechanical strength have restricted their use. New high-strength magnesium-base alloys with heat resistance and flame resistance have been developed in Japan and are now the focus of wide attention in many parts of the world. These new alloys are strengthened by a unique phase having long-period stacking ordered (LPSO) structure. These alloys are therefore called LPSO-type Mg alloys. The LPSO structure, which is formed in Mg-M-RE alloys (M is Co, Ni, Cu or Zn, and RE is limited to Y, Gd, Tb, Dy, Ho, Er or Tm), features synchronization with respect to chemical and stacking modulations. The LPSO phase has better mechanical properties than a-Mg phase and undergoes kink deformation. This kink deformation drastically improves the mechanical properties of LPSO phase. This kinking can be a new concept for the strengthening mechanism of metals.

23 Microscale mechanical behavior of unique ultrafine-grained materials

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The processing of metals through the application of high-pressure torsion (HPT) provides the potential for achieving exceptional grain refinement in bulk solids. These ultrafine grains in the bulk metals usually show superior mechanical and physical properties. Especially, the development of micro-mechanical behavior is observed after significant changes in microstructure after processing and it is of great importance for obtaining practical future applications of these ultrafine-grained metals. Accordingly, this presentation demonstrates the evolution of microstructure and small-scale deformation behavior through nanoindentation experiments after HPT on various metallic alloys including a ZK60 magnesium alloy, a Zn-22% Al eutectoid alloy, a high entropy alloy and a metal matrix nanocomposite. Special emphasis is placed on demonstrating the essential microstructural changes of these materials with increasing straining by HPT and the evolution of the micro-mechanical responses in these materials by measuring the strain rate sensitivity.

24 Shear stress and hydrostatic pressure effect in severe plastic deformation

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Most of severe plastic deformation (SPD) processes, e.g. equal channel angular pressing and high-pressure torsion (HPT), are involved in high pressure and giant shear strain. In general, the amount of applied shear strain in SPD is regarded as a primary processing parameter to control the degree of grain refinement. On the other hand, stress or hydrostatic pressure is

regarded as an additional controlling parameter to prevent damage or fracture under giant applied strain. This presentation is concerned with giant strain deformation under the high pressure behavior of metallic materials as exemplified by copper under HPT. To that end, the evolution of microstructure was considered in terms of a dislocation density-based constitutive model embedded in a finite element code. The variations of the specimen geometry, the hydrostatic pressure state, the equivalent strain, and the dislocation density were examined by numerical simulations. The simulated results of the dislocation density and the grain size were shown to be in good agreement with the experimental data for copper. The effect of hydrostatic pressure on the grain refinement was explained in terms of self-diffusivity and elastic dislocation energy. This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP) (No. 2014R1A2A1A10051322).

25 Effect of C and N on deformation behavior and stacking fault energy of Fe-Cr-Mn austenitic stainless steels

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TRIP (transformation-induced plasticity) and TWIP (twinning-induced plasticity) have received much attention as a major deformation mode in austenitic steels, and several attempts have been made to interpret TRIP versus TWIP in terms of change in stacking fault energy (SFE). In spite of many investigations on the compositional dependence of SFE, there remain inconsistencies regarding the effect of interstitial alloying elements, especially nitrogen and carbon. In this presentation, a correlation among SFE, N+C contents and deformation mode in high-interstitial-alloyed austenitic stainless steels will be reviewed.

The austenitic Fe-Cr-Mn stainless steels with different N or N+C contents were designed based on thermodynamic calculation and were fabricated by vacuum induction melting under nitrogen atmosphere. The deformation mode gradually changed from TRIP to TWIP as N+C increased. In order to understand the SFE dependence of deformation mode, SFE were evaluated using the neutron diffraction profiles. A linear relation between measured SFE and N+C contents could be established. Depending on the SFE, deformation mode changed from TRIP to TWIP and dislocation glide. The deformation bands formed under plastic deformation had distinct substructures and their particular intersecting behavior resulted in the formation of different types of products (secondary ϵ martensite, α' martensite and secondary twin) at the intersecting regions. Finally the equation which can predict the relation between SFE and the contribution of C and N was suggested.

26 Temperature and strain rate effects on strengthening of metallic materials

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The strength of metallic materials is widely determined by the interaction of moving dislocations with obstacles at different length scales. The interaction at smallest distances is given by dislocation glide in the periodic potential of the crystalline lattice leading to the so-called Peierls stress. On larger length scales, interactions become prominent with other dislocations, solute atoms, grain boundaries or clusters of atoms and precipitates. The dislocations can overcome these obstacles either by glide (shearing or bypassing) and/or by climb requiring a thermal activation. Thus, a significant temperature and strain rate dependence of the strength must follow from any kind of strengthening model. In this keynote, state of the art in yield stress modeling for crystalline metallic materials is reviewed with focus on state parameter based approaches. Particularly interesting is that some of the parameters characterizing strengthening show a time dependent behavior. This is observed, e.g., in the course of grain and precipitate coarsening, or accompanying stress relaxation around misfitting precipitates.

To describe the latter phenomenon, a novel model for particle strengthening with local stress relaxation around incoherent precipitates by creep and diffusion has been developed. In addition to the effect on strength, the volumetric misfit of precipitates accommodated by elasticity may induce high stresses, which can significantly affect precipitation. An instructive example of growth kinetics of M_3C carbides in an Fe-Cr-C matrix demonstrates the significant influence (within several orders of magnitude) of the matrix creep properties.

27 The development of superplasticity in ultrafine-grained magnesium alloys

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The processing of magnesium alloys through the application of severe plastic deformation provides an opportunity for achieving exceptional grain refinement. These materials are often superplastic at elevated temperatures with maximum elongations up to more than 3000%. This presentation examines the reports of superplasticity in a range of magnesium alloys processed by either equal-channel angular pressing (ECAP) or high-pressure torsion (HPT). It is also shown that the flow behavior of these materials may be conveniently displayed by constructing deformation mechanism maps.

28 Additive manufacturing – paving the way to industrial application

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Laser-based additive manufacturing, either using powder bed, powder- or wire-based direct material deposition processes has recently made its way into industrial applications. In particular aerospace applications, such as GE's recently announced fuel nozzle or Airbus' bionic bracket, have attracted major attention. These examples indicate that additive manufacturing has a great potential to open new horizons reaching beyond the limits of conventional manufacturing.

The present paper will highlight recent achievements in additive manufacturing from a materials and processing point of view indicating that an indepth understanding of their interrelationships is key for component performance. Future challenges are, among others, seen in the fields of quality assurance and reliable processes. In this context, the effects of defects must be understood in detail.

29 Structural evolution of metals at high temperature, pressure and plastic deformation: In-situ and real-time investigations with neutron and synchrotron quantum beams

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In-situ neutron and synchrotron X-ray diffraction deliver unique and complementary insight into the microstructural evolution of metals at high temperature, during thermo-mechanical processing or under high pressure. Neutrons illuminate a larger bulk volume and reveal quantitative phase abundance, bulk texture, lattice parameter changes and other ensemble averaged quantities. Applications are presented on phase transformation and the defect kinetics in metals at high temperature. In contrast, fine-bundled high-energy X-rays deliver reflections from a number of individual grains. For each constituting phase, their statistics and behavior in time reveal information about grain growth or refinement, subgrain formation, static and dynamic recovery and recrystallization, slip systems, twinning, etc. The concept of the Materials Oscilloscope has been developed, where multi-dimensional diffraction patterns are streaked in time, distinguishing such physical processes in a variety of metallic systems, undergoing room- and high temperature plastic deformation, in co-existing phases and across phase changes.

30 The natural aging and precipitation hardening behavior of Al-Mg-Si-Cu alloys with different Mg/Si ratio and Cu addition

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The natural and artificial aging behavior of Al-Mg-Si-Cu alloys with different Mg/Si ratios and Cu additions were investigated by Vickers microhardness measurements, differential scanning calorimetry (DSC) analysis and transmission electron microscopy (TEM) characterization. It was found that the excess Si and Cu addition enhance hardening ability during natural and artificial aging. Low Cu alloys with high Si have a higher precipitation hardening than Mg-rich alloys during artificial aging, while precipitation hardening in high-Cu alloys is less dependent on Mg/Si ratio due to the similar precipitates distribution. L, Q' and β'' phase are the main precipitates contribute to peak-aging hardness. In the over-aged condition, all the β'' phase had transformed into Q' phase. Besides, Mg-rich high Cu alloys have finer and more numerous precipitates distribution than their Si-rich equivalents due to the preferential precipitation of L phase. The combination of excess Mg and high Cu results in an alloy with relatively low hardness in T4 temper and higher hardness after the paint bake cycle, suggesting good potential for auto body panel applications.

Keyword: Al-Mg-Si-Cu alloy; natural aging; artificial aging; precipitates

31 Process-property relations in bulk metallic glasses

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Bulk metallic glasses (BMGs) are a new class of alloys which can be manufactured as massive amorphous pieces by rapid cooling from the melt. They possess excellent mechanical properties, such as high strength and good elasticity, and usually corrode homogeneously due to a lack of galvanic corrosion and the absence of grain boundaries. Processing of BMG-forming liquids is limited by the temperature-time interval of the undercooled liquid range. BMG forming is thus generally carried out via rapid cooling of the melt to bypass the “nose” of the time-temperature-transformation (TTT) diagram for crystallization. However, detailed information on how the cooling rate affects melt flow behavior during casting is lacking.

In this talk, we present ways to monitor the casting process via high-speed thermography and illustrate how different processing routes (including processing in hydrogen-containing atmosphere) generate modified glass-forming ability and mechanical properties. We also present recent results using high-speed thermography to describe BMG deformation on the microscopic scale, and illustrate how fast differential scanning calorimetry at heating and cooling rates of several 10^4 K/s can be used to understand the thermophysical properties of undercooled liquids in more detail. This understanding can in turn be used to optimize the casting process of BMG-forming liquids and thus to improve processing-property relationships in BMGs.

32 Optimization of fabrication routes for ferritic ODS steel cladding tubes: Metallurgical approach combined with thermo-mechanical simulations

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Oxide Dispersion Strengthened (ODS) ferritic alloys are developed as prospective cladding materials for future Sodium-Cooled-Fast-Reactors (GEN IV). These advanced alloys exhibit a good resistance to irradiation and a high creep rupture strength due to a reinforcement by the homogeneous dispersion of hard nano-sized particles (Y_2O_3 , $YTiO$). ODS alloys are fabricated by powder metallurgy, consolidated by hot extrusion and manufactured into cladding tube using the cold pilgering process. They exhibit low ductility and high hardness at room temperature which implies intermediate softening heat treatments. Fabrication route optimization is needed to ensure a reliable manufacturing and reduce the cladding tube anisotropy. A better understanding of the deformation paths during hot extrusion and HPTR cold pilgering is obtained through detailed finite element simulations. Such calculations require adequate constitutive laws, which were obtained through appropriate mechanical testing, namely multi-axis compression and hot torsion tests. In cold pilgering conditions, it is shown that the predicted strain path is very sensitive to the constitutive law, which would impact the development of crack initiation criteria. The specific metallurgy of ODS materials is investigated by looking at microstructure evolutions during hot deformation, or heat treatments. Recrystallization after cold pilgering is difficult to trigger, especially when the material is strongly textured. On the other hand, hot extrusion involves continuous dynamic recrystallization and grain refinement, without damage. Hot torsion tests indicate furthermore a mechanism of strain accommodation at grain boundaries, with low dislocation activity in the bulk of the grains, and development of damage at large strains.

33 Mechanically-induced grain coarsening in Gradient nanograined Cu

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Mechanically-induced grain coarsening dominated plastic deformation has been observed in many nano-grained (NG) materials. However, the intrinsic mechanism for mechanically-induced grain coarsening is not well explored yet. Here in this study, we focused on the gradient nano-grained (GNG) surface layer on coarse-grained (CG) Cu substrate and provided systematic studies of the effects of temperature and strain rate on the tensile properties as well as microstructure stability of the GNG Cu layer during tensile deformation, respectively. It is demonstrated that continuous and homogenous grain coarsening with increasing strain was detected with a variety of temperature and strain rate. However, at a given strain, the extent of grain coarsening was less significant at lower deformation temperature and higher strain rate, although higher strengths were achieved in GNG Cu layer at these two conditions.

The results demonstrated clearly that the homogenous grain coarsening accompanying the plastic deformation of GNG Cu layer was thermally activated and not exclusively controlled by the stress.

34 Innovation for the next generation of health solutions

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Over the last 50 years, biomaterials, prostheses and implants saved and prolonged the life of millions of humans around the globe. Today, nanotechnology, nanomaterials and surface modifications provides a new insight to the current problem of biomaterial complications, and even allows us to envisage strategies for the organ shortage. In this talk, three distinct but complementary applications will be targeted with the overall aim to envisage today how far innovation can bring tomorrow medical devices. They provide short, medium and long-term solutions for health, respectively. First, how to improve functional nano-coatings for medical devices will be addressed. The importance to develop functional anti-bacterial coatings for advanced health applications will be firstly discussed with special focus on how to improve the stability of the bactericidal action after repeated cycles of use, cleaning, and sterilization. Second, the potential of nanostructured metallic degradable metals to provide innovative solutions at medium term for the cardiovascular field will be rapidly depicted. Finally, a new approach for processing materials and cells directly into scaffolds will be described. The potential of dynamic cell culture in 2D and 3D will be discussed. The intrinsic goal of this talk is to present an extremely personal look at how nanotechnology can impact the innovation in materials, surfaces and interfaces, and how the resulting extreme properties allowed biomedical functional applications to progress, from the glory days of their introduction, to the promising future that nanotechnology may or may not hold for continuing improve the quality of the life of millions worldwide.

35 Revision of ISO 27306 for CTOD toughness correction for constraint loss

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As the result of the international standardization work done in Japanese IST project, ISO 27306 were published in 2009 for constraint correction of CTOD fracture toughness for fracture assessment of steel components. ISO 27306 employs an equivalent CTOD ratio, $\beta = \text{deruta}/\text{deruta (WP)}$, where deruta and deruta (WP) are CTODs of the standard fracture toughness specimen and the wide plate component, respectively, at the same level of the Weibull stress. β is less than 1 for structural components subject generally to tension, which leads to more accurate fracture assessment than by a conventional fracture mechanics approach. On the occasion of the 1st periodical review, the revision of ISO 27306 has been proposed from Japan to achieve the following aims; expansion of range of use in terms of yield-to tensile strength ratio of structural steels, improvement of accuracy of surface crack assessment and the harmonization with new BS7910-2013. This paper presents the key contents of the revision of ISO 27306. A case study is included on the fracture assessment of a wide plate component with FAD (failure assessment diagram) specified in BS 7910-2013.

36 Development of polymer-based composite coatings for the gas exploration industry

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Inorganic oxide can be synergistically beneficial for organic coating, if it has the ability to impart anti-corrosion ability and as an additive to enhance physical or chemical properties. The aim of this study was to evaluate the anti-corrosion ability of nano nickel zinc ferrite (NZF) in solution and paint film. First step includes the evaluation of anti-corrosion ability of NZF extracts in 1 molar H₂SO₄ and 3.5 wt % NaCl using electrochemical techniques (EC). During second step, time dependent anti-corrosion ability of NZF/chlorinated rubber (CR) nano-composite coatings (0.1% -1.0% NZF) applied on API 5L X-80 carbon steel (CS) were characterized by EC such as open circuit potential (OCP), electrochemical impedance spectroscopy (EIS), linear polarization resistance (LPR) and potentiodynamic (PD). Characterization of corrosion layer was done by removing coatings after 216 hrs of immersion in 3.5 wt % NaCl. Optical microscopy, EC data suggests electrochemical activity by metallic cations on surface during corrosion process which results improvements in the anti-corrosion properties of CS. Moreover, surface techniques shows compact corrosion layers coatings and presence of different metallic oxides phases for nano-composites coatings.

37 Anelastic phenomena preceding the melting of pure metals and alloys

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Precursor effects of melting in pure metals (In, Pb, Bi and Sn) and alloys of the systems Pb-Bi and In-Sn with different compositions have been investigated by means of Mechanical Spectroscopy (MS), i.e. dynamic modulus and damping measurements. MS tests carried out during heating evidenced a sharp drop of dynamic modulus E in a temperature range ΔT of 10-20 K before the formation of the first liquid. The modulus variation ΔE and the corresponding temperature range ΔT depend on the specific metal or alloy under examination.

The modulus drop is consistent with a relevant increase of interstitialcy concentration (self-interstitials assuming the dumbbell configuration), as predicted by the Granato's theory of melting. The increase of damping in the same temperature range of modulus drop supports this explanation. Owing to their dumbbell configuration self-interstitials interact with the flexural vibration of samples and the periodic re-orientation under the external applied stress leads to energy loss and damping increase.

The increase of self-interstitials has the effect of weakening interatomic bonds (modulus drop) that precedes the collapse of crystal lattice (melting).

38 Combined effects of grain boundary convection and migration in dynamic phase transformations

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During large strain deformation of polycrystals, grain or interphase boundaries are driven by the material flow, which is a convection movement. By contrast, upon static recrystallization or grain growth, their motion takes place with respect to matter, which is referred to as grain boundary or interphase migration. During hot working, where dynamic phase transformations, as for instance in some two-phase titanium alloys, or dynamic recrystallization commonly occur, convection and migration operate simultaneously. According to local geometrical (e.g., prescribed velocity field, grain boundary curvature) and physical (e.g., grain boundary mobility, dislocation densities) conditions, they can reinforce or oppose each other, but generally combine in more complex ways.

The aim of this work is to analyze such effects on the basis of simple analytical approaches. Dynamic growth and shape changes of a second phase particle during compression or simple shear are analyzed. Calculations are then extended to grain shape changes associated with growth and shrinkage of grains during discontinuous dynamic recrystallization. In particular, length ratios and orientations of the principal axes are derived. The results suggest that second phase particles or grains dynamically generated (i.e. during straining) exhibit approximately equiaxed shapes. Finally, some comparisons of the predictions are made with metallographic data.

39 Complex cell physiology on topographically and chemically designed material surfaces

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A crucial factor for ingrowth of permanent implants in the bone is the rapid cellular acceptance. The topographical features often follow mechanical aspects for implant stability. But several of these implants fail due to insufficient cell adhesion. Cells are able to perceive the physico-chemical properties of their surrounding and to pass these signals into the cell to modulate their adhesion structures, growth or production of extracellular matrix. However, the complex cell physiology at the material interface is not yet fully understood, particular on stochastically structured topographies resulting from industrial production. We could find out that corundum blasted titanium hampered the organization of actin filaments inside the cells, clustered adhesion components, e.g. beta-1 integrins and tensin, and the cells bridged the valleys which reduces cell-substrate contacts. These morphological changes strongly diminished the mineralization of osteoblasts. To shed light on cause and effect we reduced the physical complexity of the material surface by introduction of regular micro structures (pillars, grooves) using deep reactive ion etching. Now it was more obvious what cells are doing on sharp edged topographies - the actin filaments were clustered around the pillars, which is seen impressively by correlative microscopy. As a result the intracellular calcium signaling and the protein synthesis were impaired. Recent findings indicated an attempted phagocytosis of the micro pillars by osteoblasts. Therefore we conclude that implants used in orthopedic surgery should

avoid any sharp-edged topographical features that could induce phagocytosis by the surrounding cells, which is an unnecessarily energy consuming process.

40 Enhancement of mechanical biocompatibility of metastable beta-type titanium alloys by deformation-induced transformation

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Metastable beta-type titanium alloys are highly suitable for structural biomaterials for the use in hard tissue, i.e., cortical bone (hereafter bone), replacing implants. Their mechanical functionalities, such as Young's modulus, balance of strength and ductility, fatigue strength, resistance against fatigue crack propagation, fracture toughness, etc. are necessary to be improved to be compatible with those of the bone. Metastable beta phase in the metastable beta-type titanium alloy is transformed into various phases such as α' martensite, α'' martensite, and omega phases by deformation according to the stability of the metastable beta-phase. In addition, twinning is also induced by deformation as well. The deformation twinning is effective to enhance the work hardening in the metastable beta-type titanium alloy leading to increasing strength and ductility accompanied with the other deformation-induced transformation including deformation-induced martensite and omega phase transformation. The enhancement of the mechanical biocompatibilities of newly developed metastable beta-type Ti-29Nb-13Ta-4.6Zr (referred to as TNTZ) and Ti-Cr alloys for biomedical applications as well as conventional ($\alpha + \beta$)-type Ti-6Al-4V ELI, which is widely used for biomedical applications, using deformation-induced transformations mentioned above will be discussed.

41 Ultrafast heating of advanced high strength steels

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Heat treatment of advanced high strength steels with very high heating rates could be a valuable alternative for improving of both strength and elongation of steel sheets. The enhanced nucleation density and suppressed grain growth of austenite after ultrafast heating without isothermal soaking can contribute significantly to the grain refinement and in this way to the increase of the strength and toughness.

The current state-of-the-art in the field of ultrafast heating is commented and the effects of the fast and ultrafast heating rates on the microstructure and the properties of several steel grades are reviewed. The influence of the interaction between the recrystallization and the phase transformation on the microstructure formation in cold rolled sheets is analysed by monitoring the changes in the hardness of samples quenched after heating to various temperatures with heating rates from 10°C/s to 7000°C/s. It was found that for most of the investigated steel grades the ferrite to austenite phase transformation starts on partially recovered but not recrystallized matrix if the heating rate is higher than 400°C/s and that the grain refining effect is dependent on the degree of cold rolling reduction. The isothermal soaking time after ultrafast heating has a critical influence on the grain size and the properties. It was found that isothermal soaking times longer than 30 s can erase completely the effect of ultrafast heating.

The recent developments in this field are discussed together with critical analysis of the possibilities for rescaling the ultrafast heating from laboratory experiments to industrial applications.

42 Plasticity induced grain boundary migration

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Grain boundary migration is an important phenomenon during hot working of materials. It is a key to understand the microstructural evolution during the deformation process and is an essential mechanism for microstructural design of materials. This phenomenon of grain boundary migration during hot forming has been a central research area in the material science in the last century. It was long time assumed that grain boundary migration during cold deformation does not occur. The generation of ultrafine-grained and nanocrystalline material by low temperature plastic deformation has been introduced during the last two decades. To understand the minimum grain size which can be achieved by these processes, it becomes quite evident that grain boundary migration plays a key role. A central question of this grain boundary movement is: "What are the driving forces and what controls the resistance against the movement of the grain boundaries at these low temperatures?" Further important points resulting from this question are: "What is the role of thermally activated processes and what is the contribution of stress and strain induced transfer of materials from one grain to the other?" The presentation will focus on these central questions by analysing the grain boundary movement as a function of deformation temperature, strain rates, strain paths, impurities, and alloying elements. It will be shown that grain boundary movement during heavy plastic deformation at low temperature has a similar importance as in the case of dynamic recrystallization during hot working, only the driving forces are different.

43 Neutron diffraction analysis of light alloys

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The development and application of low density alloys, such as Al and Mg alloys, has rapidly increased in the automotive sector in recent years. This necessitates advanced characterization techniques to assess features of the alloy during component manufacturing. For many Al and Mg alloys, challenges (e.g. hot tearing, dimensional distortion) arise in casting and processing. Understanding the mechanism of evolution of these defects is important in ensuring their minimization. Neutron diffraction has provided a method to determine the factors that trigger hot tearing in Al and Mg alloys. Neutron diffraction analysis is effectively used in determining factors compromising integrity of powertrain components (e.g. engine blocks, cylinder heads). In addition, neutron diffraction has been applied to examine the phase evolution during solidification of Al and Mg alloys. This novel approach enabled better understanding of solidification characteristics with inoculants and solute additions, resulting in improved castability. This presentation will highlight the frontiers of neutron diffraction analysis.

44 Diamond based Schottky photodiode for radiation therapy dosimetry

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Diamond has long been considered as a suitable material for the fabrication of radiation detectors due to its outstanding physical properties. Even more so in the specific case of radiation therapy dosimetry applications, where the near-tissue equivalence radiation absorption, good spatial resolution and radiation hardness are required.

Recently, a synthetic single crystal diamond dosimeter was developed at "Tor Vergata" University in cooperation with PTW-Freiburg, showing excellent dosimetric properties. Such a device was thus commercialized (PTW microDiamond) and widely accepted by the medical physics community, due to its reproducibility, reliability, accuracy and versatility.

In this work, a novel diamond based dosimeter for in-vivo application developed in our laboratories will be presented. The new diamond dosimeter, consists of a small unwired detector and an external reading unit that can be connected to commercial electrometers for getting the detector readout after irradiation.

A basic dosimetric characterization of detector performances was performed under irradiation with ^{60}Co and 6MV photon beams. Response stability, short and long term reproducibility, leakage charge, fading effect, linearity, dose rate dependence, temperature dependence and angular response were investigated. The detector response was found to be reproducible and dose rate independent in the range between 0.5 and 5 Gy/min. Its temperature dependence is within 0.5% between 25 and 38 °C, its directional dependence is within 2% from 0° to 90° and negligible fading effect was observed. The obtained results indicate the proposed novel diamond device as a promising candidate for in vivo dosimetry in radiation therapy application.

45 Metallurgical aspects affecting thermomechanical processing of Ti based microalloyed steels

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Thermomechanical processing is a well-defined route to achieve adequate combinations of strength/toughness for a wide range of applications. Inside this family, that based on accumulated strain in austenite followed by proper accelerated cooling strategies is probably one of the most selected, mainly due to its good combinations of grain size refinement and other strengthening mechanisms. Usually this has been achieved with steels with Nb additions and other microalloying combinations. Recently, it has been observed an increase in the relevance of Ti as microalloying element. In addition to the classical approaches based on its availability to avoid grain growth, Ti provides, mainly in near-net-shape technologies, additional possibilities in relation to austenite conditioning and precipitation hardening. Nevertheless, multiple factors intervene simultaneously (solidification rates, Ti amount, Ti/N ratio, interaction with Nb...) leading nowadays, to important difficulties to properly predict its behavior. This manuscript will focus on some of these issues taking into account how they can affect final microstructure and properties.

46 Kinking and Disclinations in Plastically Anisotropic Materials

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Disclinations in solid structures are defects of rotational type, which are responsible for mutual inclination and misorientation of adjacent parts of the material [1-3]. Disclinations become important when simple translational plasticity that is related to dislocations is restricted or prohibited, e.g. in the case of LPSO structures, for which kinking is the dominating mechanism of plastic deformation. The present talk deals with the analysis of kinking and related phenomena on the basis of the disclination approach. We discuss the properties of disclinations in comparison to those for dislocations. Definitions and designations: Volterra and Somigliana dislocations, Frank (rotation) vector of a disclination, wedge and twist disclinations are introduced [1]. Screened disclinations, i.e. loops, dipoles, defects in small particles etc. with diminished energies are considered [2]. The methods and results of calculation of elastic stresses and energies for screened disclinations, for example, wedge disclination dipoles and quadrupoles are presented. We consider the models describing the structure evolution in plastically deformed materials on the basis of disclination approach [3]. The kink bands in plastically anisotropic materials and the bands with misorientated crystal lattice in metals are described as a result of partial wedge disclination dipole motion. It is shown that the same model is valid for the description of deformation twinning phenomena and elastic domain formation. Other disclination models are successful in the explanation of work-hardening at large strains and in the analysis of grain boundaries and their junctions in polycrystals. For nanocrystals, where nonuniform anisotropic plasticity is realized via grain boundary sliding disclination approach allows to explain the peculiarities of the flow stress dependence on the grain size.

47 Cooling curve based estimation of mechanical properties in high strength steel welds

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The application of high strength steels in welded structures relies on easy to use quality assurance concepts for the welding process. For ferritic steels, one of the most common methods for estimating the mechanical properties of welded joints is the cooling time concept $t_{8/5}$. Even without experimental determination, the calculation of cooling time with previously introduced formulas based on the welding parameters leads to good results. Because high strength structural steels and weld metals with a yield strength of 960 MPa contain higher quantities of alloying elements, the transformation start temperature A_{r3} is found to be outside of the range of 800 °C to 500 °C. This leads to inadequate estimation results, as the thermal arrest caused by the microstructural transformation in this case is not considered. In this work the usage of the well-proven cooling time concept $t_{8/5}$ is analyzed using high strength fine grained structural steels and suitable welding filler wires during gas metal arc and submerged arc welding processes. The results are discussed taking into account the microstructure and the transformation behavior. Based on the experimental work, an improved concept is presented.

48 Surface modifications for engineering the Properties of Inorganic Two-dimensional Nanostructured materials

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Nanomaterials with quantum confined electrons show the special tunable band gap, surface activity and planar conductivity. Increased surface activity enhances the chemical and physical reactivity and also influences the 2D-wave function. Surface chemical modification helps in engineering the intrinsic physical properties of 2D materials. The surface modification can be achieved by incorporation of hydrogen, water, dangling bonds or by creating defects such as pit, vacancy, disorder or by changing strain, superlattice and grain boundary of the materials. These surface modifications in 2D inorganic materials (MoS_2 , CeO_2 , Graphene- V_2O_5 , ZnS , ZnO , Graphene- ZnO etc.) changes the intrinsic physical properties like electronic mobility, carrier concentration, spin polarization, photo/electro-chemical reactivity and intrinsic magnetism. The surface modification provides the inherent stability and non-destructive nature of chemical modification. Hydro/solvothermal synthesis methods were appealing approaches to prepare non-layer 2D nanostructures, in which the experimental parameters such as the concentration of precursors, solvent, reaction temperature and surfactants were important factors. Owing to their large lateral size and ultrathin thickness they are the ideal materials for surface-active applications such as photo-catalytic water splitting, catalytic oxidations and conversions, super-capacitor applications as energy storage devices, photo detector, photo thermal therapy etc. Systematic studies were needed to optimize the experimental conditions to achieve the synthesis of new non-layer structured 2D nanostructures. It is also important to find a general and effective way for controlled synthesis of ultrathin non-layer structured materials. Further rationalize the design of a material with desired lateral size, thickness, defect, structure and crystal phase are very much important.

49 Hierarchical design and synthesis of nanomaterials to enable high capacity rechargeable battery electrodes

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Sulfur-based cathodes have been considered as one of the strongest candidates for next generation Li-ion batteries because of their high theoretical specific capacity. However, practical application of sulfur-based cathodes is currently hindered by three major challenges: (i) being an ionic and electronic insulator, (ii) large volume change during cycling, and (iii) dissolution of long chain polysulfide products (Li_2S_x , $4 < x \leq 8$) into the electrolyte. In this talk we will describe a novel concept of a hierarchical structure with a Li_2S -carbon black ($\text{Li}_2\text{S}/\text{CB}$) nanocomposite core encapsulated by a conductive shell. The CB network inside the composite core allows most of Li_2S to participate in electrochemical reactions, while the outer conductive shell can serve three functions: i) offering a highway for both electron and Li-ion transport, ii) confining the volume expansion and shrinkage within the shell, and iii) preventing the dissolution of polysulfides into the electrolyte. Using this concept we have demonstrated the highest initial discharge capacity (1,029 mAh/g Li_2S which is equivalent to 1,476 mAh/g S) at the 0.2C rate among all of the reported works for the encapsulated Li_2S and S cathodes. We

have further demonstrated that by control of interfacial chemistry during the formation of the conductive shell a uniform shell can be formed which greatly enhances the capacity retention of Li_2S cathodes over charge/discharge cycles. To conclude our talk, we will discuss the future directions to make Li_2S cathodes with high energy density, high power and long cycle life.

50 Microstructure and mechanical properties of twin roll cast magnesium alloy sheets

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There has been an increasing demand for the high performance Mg alloy sheets in automotive and transportation industries to reduce car weight and to increase the fuel efficiency. However, the development of high performance Mg alloys is difficult to achieve due to limitation of deformation modes and high production cost related to processing of Mg sheets. Recently, the twin roll casting (TRC) process is recognized as an efficient process for production of Mg alloy sheets with lower cost. Although the TRC process has received a great attention, only limited Mg alloys have been produced due to solute segregation and poor mechanical properties. In order to expand the application of Mg alloys, development of high performance Mg alloys with controlled microstructure and good mechanical properties is required. In the present study, new TRC Mg alloys were developed by controlling solute segregation. Magnesium alloy sheets with various compositions were produced using the twin roll casting process. The microstructure and texture were characterized by optical microscopy and XRD. The mechanical properties were evaluated by tensile tests. New TRC magnesium alloy sheets have successfully been developed with reduced segregation and good mechanical properties.

51 Texture: The “fingerprint” of deformation mechanisms in nanomaterials

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With decreasing grain size different deformation mechanisms determine the plasticity of materials. In nanomaterials (grain size <100 nm) grain boundary mediated processes like emission and absorption of dislocations by grain boundaries and grain boundary sliding predominate (Skrotzki et al., 2013). Here, texture provides a “fingerprint” of the deformation mechanisms because it accumulates the information on the activity of the slip and twinning systems as well as on grain boundary sliding processes. This will be demonstrated on Pd-Au solid solutions deformed by high pressure torsion.

Skrotzki, W., Eschke, A., Jonas, B., Ungar, T., Toth, L., Ivanisenko, Yu. and Kurmanaeva, L., 2013. New experimental insight into the mechanisms of nanoplasticity. *Acta Mater.* 61, 7271 – 7284. DOI: 10.1016/j.actamat.2013.08.032.

52 Microstructure and creep behaviour of similar martensitic 9% chromium steel electron beam welds

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Advanced martensitic 9%Cr steels have been welded using the electron beam welding (EBW) process. The influence of welding parameters on the development of the microstructure within the weldment was investigated in order to find a welding procedure leading to the optimal microstructure for creep resistance. A decisive aspect when welding martensitic 9% Cr steels is the width and the microstructure of the heat affected zone (HAZ) and particularly the presence of a grain-refined zone which is considered as the weakest zone in weldments during creep loaded service (due to type IV cracking). The microstructure in the different zones of the weldment, and particularly in the HAZ, was analysed before and after post weld heat treatment (PWHT). Uniaxial creep tests of base material as well as electron beam welded joints were carried out at 650°C at different stress levels. Creep tests reveal that the EB welded joints exhibit a lower creep strength compared to base material. Further microstructure examinations of crept welded samples have shown that failure always occurs along prior austenite grain boundaries in the HAZ. However, when comparing creep results obtained with conventional welding processes, EBW process offers very promising results.

53 On the calculation and impact of phase boundary energies on precipitate kinetics in complex alloys at high temperatures

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Mechanical and technological properties of alloys are significantly affected by precipitates in the microstructure. The presented work deals with aspects of modelling the kinetics of the particles. Models going beyond phenomenological calculation of phase fractions usually incorporate the energy of the phase boundary between a precipitate and the surrounding matrix. This energy contributes to the formation energy of the initial nucleus, it retards the growth of the particles and acts as driving force for coarsening processes. Therefore, it is of utmost importance to be able to model the phase boundary energies in precipitate kinetic simulations. If this quantity is not known, it has to be substituted by an according fit parameter, which is limiting the predictive capability of the according model. This work summarizes the individual approaches contributing to the General Broken Bond (GBB) concept and demonstrates its applications and limits in complex alloys. GBB takes into account thermodynamic data from precipitates and matrix, their chemical compositions, orientation and curvature of the interfaces and concentration gradients across the interface at high temperatures. Since the model is designed for multicomponent materials, it has been applied to Al-alloys, ferritic and austenitic steels and Ni-base alloys amongst others. It is discussed to what extent GBB was successfully applied, what limitations were experienced, and what kind of improvements can still be implemented.

54 Grain size dependence on the yielding behaviour of iron

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Discontinuous yielding has been believed to be characteristic of low carbon steels but such a discontinuous yielding is also found in polycrystalline metals with fine grained structure. In low carbon steels, it was confirmed that a Hall-Petch relationship stands up in the relation between grain size and lower yield stress. This means that yield stress is governed by the mechanism of grain refinement strengthening. On the other hand, it is sure that the discontinuous yielding is always followed by yield point elongation. In this presentation, the deformation behaviour after yielding will be discussed in connection with the change of dislocation density. The material used in this investigation is a commercial low carbon steel (0.15%C) and grain size is controlled in the range from 1 μ m to 5 μ m. The contents in this presentation are as follows: 1) Even at same strain, the introduction of dislocations is promoted with reducing the ferrite grain size. 2) The yield stress of cold worked specimens follows an identical Bailey-Hirsch line regardless of the ferrite grain size and the charged strain. 3) Yield point elongation can be explained by the plastic instability which appears in the process of strengthening mechanism shift from grain refinement strengthening to dislocation strengthening.

55 Advanced Plasma Processing for Surface modifications of Materials

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Plasma processes started to be applied for surface modification of materials in the 70's, in the fields of microelectronics and semiconductors. Since then, enormous advancements in the basic, diagnostic and experimental aspects of plasma sciences have been made, so that many other science areas and industrial fields have been permeated by plasma processes: polymers, textiles, biomaterials, microfluidics, composite materials, paper, packaging, automobile, waste treatment, to mention but a few. The advantages of plasma lie in the ability to generate active species that are chemically and physically actives (excited atomic, molecular, ionic and radical species) at low temperature. We begin with a brief overview of the physics and chemistry of cold plasmas. Next, interaction mechanisms between plasma and a material will be examined: these include the 3 main effects, namely, (i) cleaning/ablation, (ii) modification of surface-chemical structure, and (iii) plasma enhanced chemical vapour deposition (PECVD). In this presentation, we will report recent advances in the successful use of low pressure plasmas for surface modifications and thin film growth. Considering the actual environmental constraints, emphasis will also be put on the atmospheric pressure plasma jet process, as a promising technology for surface treatments.

56 Thermomechanical Processing of Medium Manganese Steels

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As third generation advanced high strength steels (AHSS) managing both high strength and good ductility/formability, medium manganese steels containing 3-7 wt% Mn have attracted attentions recently. However, the fundamental microstructure evolution during thermomechanical processing and heat treatments in medium-Mn steels is still unclear. In the present study, changes in microstructure and mechanical properties during various heat treatments and thermomechanical processes of 4-5%Mn-0.1%C(-2%Si) steels were systematically studied. It was clarified from dilatometric measurements that ferrite transformation in 4Mn-0.1C steel was quite slow, so that fully martensitic structures were obtained in many cases after cooling from austenite. On the other hand, hot-deformation of austenite greatly accelerated ferrite transformation, and dual phase microstructures composed of ferrite and martensite could be obtained. The dual phase steel showed good combinations of high strength and adequate tensile ductility.

57 Recent findings in bulk nanostructured materials produced by SPD processing

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During the last decade the use of severe plastic deformation (SPD) for nanostructuring of materials to control their properties has developed into one of the most dynamic topics in modern research on nanomaterials science and engineering.

The present report deals with new findings of superstrength without sacrificing significant ductility and superductility with enhanced strength, which have been revealed recently in a number of SPD-produced ultrafine-grained alloys. Another interesting and novel discovery is observation of simultaneously superior strength and high electrical conductivity in Cu and Al alloys after SPD. The nature of these unusual phenomena is associated with development of grain boundary engineering of bulk nanomaterials and these new approaches are represented in detail in the paper.

The report concludes with new trends in research and development on the scale-up of SPD processing for its practical applications in engineering and medicine.

58 Neutrons for materials characterization under extreme conditions

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Advances in neutron flux, neutron instrumentation, and sample environments over the past years allowed the development of unique techniques to characterize material synthesis and processing. Here, we present capabilities and results to characterize materials in situ at temperatures above 2000°C as well as using recently developed energy-dispersive neutron imaging and tomography applied to optimizing single crystal growth in situ.

Temperatures in excess of 2000°C are not readily achieved and neutrons are one of a few probes to characterize materials under these conditions. Research in refractory materials, phase diagram studies or accident scenarios for nuclear materials are research areas where such extreme conditions are required. As an example, we present the formation of UC_x from UO_{2+x} and graphite in situ using high temperature neutron diffraction with particular focus on resolving the conflicting reports on the crystal structure of non-quenchable cubic UC_2 .

Energy-dispersive neutron imaging and tomography utilize isotope-specific neutron absorption resonances to visualize the distribution of elements in the bulk. Furthermore, so-called Bragg-edges allow measurements of lattice strains and thus imaging of e.g. the stresses in a sample. Homogeneous distribution of dopants, such as europium, is essential for the performance in the detector application while controlling and minimizing the stresses reduces mechanical failures. As with many other neutron techniques, sample environments to e.g. study crystal growth by the Bridgman technique in-situ are possible, providing direct feedback on how processing parameters affect material properties such as dopant distribution, mosaicity, or stresses.

59 Ionic conducting polymer electrolytes for electrochemical energy technologies

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Many electrochemical devices using solid conducting membranes were developed in the past two decades, such as electrolyzers, fuel cells, and redox flow batteries. Ion exchange materials have great potential to be applied in new energy conversion devices and membrane reactors, which are highly efficient, energy saving and friendly to the environment.

The future of this type of technology depends greatly on the enhancement of membrane stability. The polymer electrolyte membrane must be improved in terms of durability; most importantly, it must operate in an aggressive environment and under harmful experimental conditions, and it must present a reduced permeability. In general, anion exchange membranes show lower permeability than cation exchange membranes. Unfortunately, most commercial anion exchange membranes do not have sufficient chemical stability and present a low conductivity. We have in recent years concentrated on improvement of existing aromatic polymers introducing Van der Waals bonds (organic-inorganic hybrids) or covalent bonds (cross-links). The continuous optimization of synthesis parameters, the choice of different polymers and the improvement of treatments of membranes, led to good results in terms of ionic conductivity, selectivity and stability. [1] H Hou, ML Di Vona, P. Knauth ChemSusChem. 2011, 4, 1526-1536 [2] H Hou, ML Di Vona, P. Knauth J Membr Sci 2012, 423, 113-127

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60 Thermo-mechanical training of Fe-Mn alloys to improve damping capacity

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A high damping capacity is considered as one of the important functional properties of shape memory alloys. Those properties are related to a martensitic transformation. Since training treatments in the shape memory alloy are known as a useful method to improve the shape memory effect, the effects of training treatments on damping capacity of Fe-Mn damping alloys are studied. As training treatments, thermal training (only thermal cycling) and the thermo-mechanical training (thermal cycling with rolling deformation or bending deformation) are carried out. Since vibration manner in internal friction measurement is bending mode, the thermo-mechanical training featured by bending mode is performed. It is found that thermal training and thermo-mechanical training improve the damping capacity of the Fe-Mn alloys with increasing the number of training cycles. Comparing improvement ability of the damping capacity between both training treatments, thermo-mechanical training can improve it more effective. The epsilon martensite phases have a preferred orientation in the specimens with thermo-mechanical training with bending deformation. An anisotropic damping capacity is also observed for the samples subjected to bending mode thermo-mechanical training. It is found that the trade-off between the damping capacity and hardness can be overcome by the thermo-mechanical training. The thermo-mechanical training treatments, particularly training with bending deformation, are useful for enhancement of the damping properties and hardness of Fe-Mn alloys.

61 Large scale metal additive manufacture for engineering parts

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The additive manufacture of large engineering metal structural parts is currently of great industrial interest and provides significant challenges. Foremost amongst these is achieving material properties equivalent to or better than those provided by other manufacturing routes and with high integrity. The Wire + Arc Additive Manufacture (WAAM) process has been investigated in detail for these applications due to the major cost saving it provides compared to the current manufacturing processes. Furthermore WAAM can be easily combined with in-process cold work to produce material properties better than those of forged material. This presentation will highlight the material properties that can be achieved by the WAAM process both with and without cold work. Examples will then be given to demonstrate how it has been used to produce large engineering structures. Finally details of the expected cost saving for some specific examples will be given.

62 Reactions and pressure-induced phase transitions in the diamond anvil cell

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Synchrotron-based diffraction experiments in diamond anvil cells allow us to monitor pressure-induced changes up to Mbar pressures. Such high pressures often lead to significant changes in the electronic structure, such as high-spin to low-spin transitions. Heating compounds at very high pressures often leads to new compounds or novel polymorphs, thus allowing us to significantly expand our knowledge of structure-property relations. In my contribution, I will discuss the use of the laser-heated diamond anvil cell to synthesize new compounds, such as transition metal borides, carbides and nitrides. I will summarize studies addressing pressure-induced spin-crossover transitions and will conclude with an outlook towards recent advances in detector development, using the LAMBDA detector as an example. This detector will allow time-resolved studies with millisecond time resolution using hard x-rays, and I will present first measurements with such a detector.

63 Development of high-temperature shape memory alloys above 673 K

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High-temperature shape memory alloys (HTSMAs) has been expected to use in aerospace, automotive, chemical and power plants industries. However, shape recovery becomes small with increase martensitic phase transformation temperature due to plastic deformation. Some promising alloys are found to be used around 473 K, alloys which can be used above 573 K have not been developed yet.

We have focused on TiPt, TiPd, and TiAu alloys as potential HTSMAs because of their high martensitic transformation temperatures of above 843 K. The martensitic transformation from cubic B2 in austenite to orthorhombic B19 martensite occurs in all of TiPt, TiPd and TiAu. The shape recovery of binary alloys was very small due to plastic deformation above A_f temperature. Then, solid solution hardening by addition of alloying element has been performed to improve shape recovery. In this study, shape recovery was investigated by loading-unloading compression test and thermal cyclic test. Training effect such as thermo-mechanical treatment was also investigated using thermal cyclic test. Although the perfect shape recovery is difficult, we found some interesting trend to improve shape recovery. The effect of yield strength of austenite and martensite, detwinning strength of martensite, and microstructure change by thermomechanical treatment on shape recovery will be discussed together with history of development of HTSMA.

INVITED ABSTRACTS

64 Magnetic shape memory - polymer hybrids

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Martensitic Ni-Mn-Ga alloys are known for the Magnetic Shape Memory (MSM) effect, which can generate a strain of up to 6 % or even up to 12 % by applying external magnetic field. The MSM effect occurs by rearrangement of the martensite variants and the maximum strain level depends on the microstructure of the martensite being most advantageous in single crystals. As single crystals are rather tedious to produce, there have been attempts to achieve MSM effect in polycrystals. However, in polycrystals the magnetic field induced shape change has remained low so far. As an alternative to the former, hybrid MSM materials offer several advantages. When compared to single crystals, hybrids have extended freedom of shaping, lower raw material price, relatively large MSM strain and easier manufacturability. Embedding MSM particles into a suitable polymer matrix results in actuation function or good vibration damping performance. In the present study we report on the mechanical, structural and magnetic properties of MSM polymer hybrids, which are prepared by mixing gas-atomized Ni-Mn-Ga MSM powder into epoxy matrix and aligning the magnetic particles in a magnetic field.

65 Artificial intelligence approach to predict strain-stress curve of steels

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A combined approach of Bayesian inference and artificial neural network was performed to predict strain-stress curve of steels from the view point of material genome-based AI materials science. As descriptors, microstructural features such as metric and topological features were taken into consideration.

66 Modelling and simulation of pore formation in chromium steels during creep

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A model has been developed to describe the nucleation and growth of creep pores in steels under service conditions. The nucleation model is based on classical nucleation theory by using Helmholtz free energy, while for the growth of pores, a vacancy flux model towards existing pores is utilized. The model is able to describe nucleation and growth rates of pores, in the matrix (homogeneous nucleation), at grain boundaries, at triple and quadruple grain boundary points and at particles/inclusions. Nucleation and growth of pores in creep process is determined to be a function of; uniaxial or multiaxial external stress, internal stress due to the residual stresses, working temperature, local microstructure (nucleation and growth of particles), nucleation sites, interfacial energy of grain boundaries and phase boundary energies, diffusion rates in different paths and pore geometry which are all considered in this investigation.

The aim of the present study is modelling and simulation of pore formation/growth under 66 and 90 MPa uniaxial creep loading at 650°C after 0, 2000, 4000, 6000 and 8000 hours for 9Cr-1Mo martensitic (ASME Gr.91) steels. The model results are then compared to experimental findings of pore number densities which shows good agreement between experiment and simulation results.

67 Change in mechanical strength and bone contact ratio of beta-type TNTZ subjected to mechanical surface modification

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Ti-29Nb-13Ta-4.6Zr (TNTZ), which is one of metastable beta-type Ti alloys, was recently developed as a representative biomedical Ti alloy for next generation. As-solutionized TNTZ has a low elastic modulus less than 60 GPa close to that of cortical bone along with very low cytotoxicity and good bone biocompatibility. Solution treatment and aging (STA) is a typical heat treatment for improving the mechanical properties of beta-type Ti alloys. However, STA also drastically increases the elastic modulus. Therefore, this study investigated the effects of fine particle bombarding (FPB) or friction stir processing (FSP) on the mechanical properties of TNTZ subjected to more severe thermomechanical treatment in order to maintain a relatively low elastic modulus. The bone contact characteristics of TNTZ samples subjected to surface modification and cancellous bone were also compared.

Vickers hardness of TNTZ subjected to FPB was significantly increased within 20 µm from the very edge of the specimen surface. The trend of change in Vickers hardness was almost identical to FSP. The fatigue strength of TNTZ subjected to FPB increased in the high cycle fatigue life region only. The fatigue limit was around 400 MPa. The bone contact ratio of TNTZ subjected to fine particle bombarding was better than that of TNTZ with the mirror surface.

68 Evolution of homogeneity in oxygen-free copper processed by ECAP and HPT

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Oxygen-free copper having a commercial purity of 99.95 wt.% was processed at room temperature by two techniques of severe plastic deformation: equal-channel angular pressing (ECAP) and high-pressure torsion (HPT). The HPT processing was performed under an applied load of 6.0 GPa for up to 10 turns while ECAP billets were processed through a die having an internal angle of 110° using route Bc for up to 8 passes. The evolution of microstructural homogeneity for each process was investigated by measuring the microhardness across the diameters of HPT discs and across the cross-sectional planes of the ECAP billets. Also, the distributions of microhardness measurements were recorded on the cross-sectional planes of the HPT discs and ECAP billets using a rectilinear grid pattern to produce color-coded contour maps.

69 Analytical sub-angstrom scanning transmission electron microscopy of alloys and steels

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Field-specific development of new materials requires the fundamental understanding of the atomic scale effects which drives the micro- and nano-structure morphology and chemical composition of various phases. Recent improvements of electron microscopes facilitate such studies at sub-Angstrom resolution.

This paper presents a couple of characterization methods for the investigation of different material types ranging from Al based alloys to chromium rich steels. Energy filtered TEM (EFTEM) and scanning TEM (STEM) provides insight into the material's crystallography and chemistry quantitatively and at atomic resolution. By using imaging with the high angular annular dark field detector (HAADF) and both X-ray (EDX) and/or electron energy loss spectrometry (EELS) [1,2] high resolution images and corresponding spectra can be acquired. Therefore, localization and identification of alloying elements with very low concentration (for example Sr, Sc and Ag in Al-alloys) which might be present as a single column, in clusters with a low number of atoms [3] or enriched within precipitates and at their interface with the matrix, was possible. Furthermore the nucleation and evolution of some precipitates during heat and creep treatment due to pipe- and substitutional- diffusion could also be studied.

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70 Radiation effects in ionic crystals: To create or not to create metallic colloids?

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The formation of point defects and their aggregates in wide band-gap materials has attracted the attention of scientists for more than a century. The ability of ionizing radiation to generate such defects and the fascinating change of coloration induced by trapping electrons or holes into vacancies and interstitials in conjunction to the simple crystalline structure make alkali and alkaline earth halides promising candidates to investigate the effects of ion irradiation at the electronic interaction regime. In the past, radiation-induced alterations were mainly investigated by using ion beams with energies in the range between keV and MeV, where the penetration depth of such ions is rather limited and do not allow macroscopic tests of bulk materials. Here, a series of experiments with beams of relativistic, high-energy heavy ions (energies beyond GeV) are reported aiming to discuss the necessary conditions for the creation of metallic colloids. Particularly, the question why the cubic fluorite structure (e.g. CaF₂) readily develops the colloids, while this is not the case for the cubic rocksalt structure (e.g. LiF). The experiments include optical absorption, Raman spectroscopy, resonant ultrasound

spectroscopy, electron paramagnetic resonance, thermo-luminescence, calorimetry, sputtering, bleaching and annealing.

71 Hardness homogeneity of an AZ80 magnesium alloy processed by high-pressure torsion

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Experiments were conducted on an AZ80 magnesium alloy, by processing by high-pressure torsion (HPT) at room temperature (296 K) for up to 10 turns under an imposed pressure of 6.0 GPa. Measurements of Vickers microhardness along diameters and through the disk thickness were recorded after HPT to evaluate the evolution towards homogeneity. The results show the hardness increases up to the factor of approximately 2 and the deformation is more homogeneous along the disk diameter than through the disk thickness.

72 Microstructure and friction behavior of AISI52100, D2 and H13 steels subjected to ultrasonic nanocrystalline surface modification (UNSM) technique at a high temperature

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In this study, AISI 52100, D2 and H13 steels were subjected to ultrasonic nanocrystalline surface modification (UNSM) technique at high temperature (up to 500 °C). The objective of this study is to characterize the microstructure and to investigate the effectiveness of modified surface on the friction and wear under dry conditions. The friction and wear behavior of the specimens against AISI52100 bearing steel ball with a diameter of 12.7 mm was carried out using a micro-tribo tester. The mechanical properties, in particular, hardness and compressive residual stress with respect to depth from the top surface were measured using a microhardness and X-ray diffraction. The change in the microstructure of the specimens before and after UNSM treatment was characterized by scanning electron microscopy (SEM) and the chemical composition was studied by energy-dispersive X-ray spectroscopy (EDS). The cross-sectional SEM analyzes revealed that the top surface of the specimen was plastically deformed which is a reason for the increase in hardness and improvement in friction and wear behavior of the specimens. The findings from this preliminary study are expected to be implemented to the bearings and tools to increase the efficiency and performance of the components.

73 Microstructure evolution and deformation mechanisms of harmonic structure designed materials

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Through many years, conventional material developments have emphasized on microstructural refinement and homogeneity. However, "Nano- and Homogeneous" microstructures do not, usually, satisfy the need to be both strong and ductile, due to the plastic instability in the early stage of the deformation. As opposed to such a "nano- and homo-" microstructure design, we have proposed "Harmonic Structure" design. It is a new material design paradigm allowing the enhancement of structural performance. The harmonic structure has a heterogeneous microstructure consisting of bimodal grain size together with a controlled and specific topological distribution of fine and coarse grains. In other words, the harmonic structure is heterogeneous on micro- but homogeneous on macro-scales. In the present work, the harmonic structure design has been applied to pure-Ti, Ni, Cu, Fe, Ti-6Al-4V alloy, SUS304L stainless steel and Co-Cr-Mo alloy via a powder metallurgy route consisting of controlled severe plastic deformation of the corresponding powder (pre-alloyed in the case of Ti-6Al-4V and SUS304L) via mechanical milling or high pressure gas milling, and subsequent consolidation by SPS. At a macro-scale, the harmonic structure materials exhibited significantly better combination of strength and ductility, under quasi-static loadings, as compared to their homogeneous microstructure counterparts. This behavior was essentially related to the ability of the harmonic structure to promote the uniform distribution of strain during plastic deformation, leading to improved mechanical properties by avoiding or delaying localized plastic instability.

74 Preparation of high corrosion resistance Ni-based amorphous alloy and their thermal spray coatings

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Many amorphous alloys with high corrosion resistance have been prepared mainly in forms of ribbons and thin films. The Fe-based amorphous coating films with high chromium content had been formed by a thermal spraying with oxyacetylene gas flame. Thermal spray gun of oxyacetylene type is a simple and lightweight thermal spraying gun and has popularized widely. Recently, the Fe-based spray film is obtained by study of spray parameters and the Fe-based amorphous flame spray film is marketed. However, the spray film with higher ductility and corrosion resistance is needed.

Therefore, we considered production of the sprayed coating of Ni-based amorphous alloy. The Ni-Mo-B ternary alloys are developed for flame spray coating. The Ni-Mo-B amorphous alloy exhibited high corrosion resistance (corrosion rate: 10^{-2} mm/year) in aggressive HCl solutions. And the ductile Ni₆₆Mo₁₅B₁₉ films were successfully coated the transporting iron-rolls by a thermal spraying with gas flame. Furthermore Ni-Cr-Nb-P-B with high ductility and high corrosion resistance are developed for spray coating. And the productions of the thermal spraying coating of Ni-Cr-Nb-P-B amorphous alloy with ductility were also successful by thermal spray and HVOF method.

75 Additive manufacturing of parts from advanced materials by 3D screen printing

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3D screen printing is an additive manufacturing technology whereby a part is built up layer-by-layer from a metal powder containing printing paste. Just as in ordinary screen printing processes, the paste is pushed through a structured screen by a squeegee. This way, a printed layer of about 20 μm thickness is formed on the substrate underneath the screen. After curing, the printed layer itself serves as the substrate for the next printed layer. The finished printed part is debinded and sintered, resulting in a fully metallic, ceramic or composite part. 3D screen printing allows for intricate features below 100 μm in size, as well as high aspect ratios above 100. Due to the powder metallurgical process route, a wide range of materials is accessible such as copper, steel, titanium, metal matrix composites, refractory metals, as well as ceramics. Examples of different materials and different geometrical features attainable by 3D screen printing will be given along with their potential areas of application.

76 Activation stress of slip systems in magnesium single crystals by pure shear test

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Pure shear tests were carried out to evaluate critical resolved shear stresses (CRSS) of slip systems in magnesium single crystals. As a result, the CRSS for the basal slip was 0.35 MPa, and it was close to conventional values. When the shear stress of 90 MPa was applied in parallel to the prismatic plane, the $\{10\text{-}12\}$ twin deformation occurred only, and the prismatic slip deformation did not occur. Therefore, the CRSS for the prismatic slip is expected to be over 90 MPa.

77 A comparative study of molecular motion cooperativity in polymeric and metallic glass forming liquids

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Recently, much attention has been devoted to understand the mechanism of glass transition and structural relaxation in supercooled liquids. In particular, many authors have studied the cooperativity and heterogeneity that develop in the supercooling process. Among these, the Bond Strength-Coordination Number Fluctuation (BSCNF) model developed by the authors describes the relaxation process in terms of the mean values of the bond strength, coordination number and their fluctuations of the structural units that form the melt. The BSCNF model has been applied successfully to analyse the temperature dependence of transport properties in

different systems. From these analyses, important clues, such as the number of structural units involved in the transport processes, behaviour of elastic properties across the glass-liquid transformation region, etc. were obtained. In order to gain further understanding on the behaviour of the molecular motion cooperativity in glass forming systems, we performed a comparative study between polymeric and metallic systems based on the BSCNF model. Preliminary result indicates that the degree of molecular motion cooperativity differs between the two systems. Specifically, the cooperativity in polymeric systems was around 5 times larger than that in metallic systems. The result found in the present study could be of interest from the application point of view, because many materials of technological interests exploit their relaxation properties.

78 Materials to control biological cells function: A focus on microtopography influence

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Surface modifications of materials are used to monitor and evaluate cellular function for several decades. An example of this is the ability to control tissue integration of medical prostheses by changing their composition, surface topography, chemistry, energy, or their mechanical properties. Similarly, control of the surface of materials is also a key element in the effectiveness of diagnostic tools, devices for cell culture, bio-sensors or drug delivery systems. The cells have the ability to discriminate and specifically react to surface characteristics of the materials considered at the micrometer scale as well as the nanometer scale. In this talk, I will focus on the response of cells to topography at their own scale. Our last experience on living cells behavior on microfabricated surfaces with 2D or 2.5D motifs will be detailed.

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79 Microstructural and micromechanical characterization of damage initiation in DP steels

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Typical microstructures of dual-phase (DP) steels consist of hard martensite particles dispersed within a ductile ferritic matrix. These microstructures possess a complex network of grain and interphase boundaries, which, together with the high contrast of the mechanical properties between the different constituents, control micro-fracture mechanisms, induced under deformation. Accordingly, in this study we analyze the influence of individual microstructural features and interfaces on damage nucleation and progression in DP steels. This detailed analysis aims at obtaining the essential information to optimize the processing parameters, in order to enhance the fracture toughness of the bulk material. To this end we studied the fracture evolution under cyclic and monotonic loading. Scanning electron microscope (SEM) – based

techniques were utilized, including electron backscatter diffraction (EBSD), electron channeling contrast imaging (ECCI), and focused ion beam (FIB) milling. Statistically relevant results illustrate favored micro-void / -crack nucleation sites, with respect to the applied strain level. One of the prominent fracture mechanisms is cracking of martensite, where prior austenite grain boundaries (PAGBs) are highly favorable for nucleation and propagation of cracks. This special behavior of PAGBs might be attributed to grain boundary segregation of some chemical constituents, or to residual elastic stresses stored in the vicinity of these boundaries. Currently, atom probe tomography (APT) and auger electron spectroscopy (AES) are employed for a detailed chemical analysis of PAGBs. Additionally, a novel in-situ micro-mechanical bending experiment has been used to examine the fracture toughness of individual grain and interphase boundaries.

80 Anelastic dislocation behaviour of an interstitial free steel

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One of the major issues that automotive industry has to deal with in forming Advanced High Strength Steels (AHSS) is springback -strain relaxation after release of the forming stresses-. Experimental evidence has shown that the magnitude of springback is dependent on an anelastic contribution, and hence, on dislocations. However, how do the dislocations cause this anelasticity? Already in the pre-yield regime, the dislocations bow out and cause the so-called anelastic strain in addition to the elastic lattice strain. This additional strain component is responsible for the non-linearity that is often observed in the stress-strain curve below the yield stress. After plastic deformation and during unloading, similar mechanisms as those in the pre-yield regime occur, albeit in a reversed manner, and lead to the springback phenomenon. Thus, better comprehension of the anelastic dislocation behaviour is essential for accurate springback prediction. In this work, the dislocation behaviour of an interstitial free steel after plastic deformation is analysed and compared to that of a low-alloy steel at room temperature. A physically-based model that characterises the dislocation structure through two variables, the dislocation density and the effective segment length, is used in combination with mechanical tests and advanced characterisation techniques to describe and quantitatively explain this behaviour.

81 Microstructure observation of Al-Zn-Mg alloys with different Zn, Mg concentration

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7xxx series alloy (Al-Mg-Zn) has a high strength compared to another Al alloy due to its good age-hardenability. The major elements of 7xxx series alloy are Mg and Zn, their ratio (Zn/Mg) is 0.5~1.5 for the commercial grade 7xxx series alloys. Only few reports as to precipitation hardening are available for low Zn/Mg ratio alloys. In this research, Al-Mg-Zn alloys that have different Mg, Zn amount but the ratio of Zn/Mg is fixed to 0.25 were prepared using casting

and subjected to aging treatment to observe their aging behavior. As aging time increase, alloys were hardened. Tensile tests showed that ultimate tensile stress (UTS) increased with increase of Mg, Zn amount in alloys. Transmission electron microscopy (TEM) observation was conducted using peak-aged samples. With increase of Mg, Zn amount, precipitates became finer and the number of density increased.

82 Neutron studies of geometrically frustrated layered manganese oxides

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Materials which exhibit strong geometric frustration are of research interest due to the possibility for the evolution of exciting magnetic states including incommensurate and noncollinear spin arrangements. Layered manganese oxides, exhibiting triangular lattices, have been the subject of extensive research within the literature due to the frustrated nature of the MnO_6 layers. The layered manganese oxide, $\text{Ca}_2\text{Mn}_3\text{O}_8$ is related to the triangular lattice with MnO_6 layers separated by Ca^{2+} . In contrast with triangular lattices $\text{Ca}_2\text{Mn}_3\text{O}_8$ exhibits ordered Mn^{4+} vacancies. However, whilst this material has received extensive attention in the literature as a potential catalyst the magnetic and electronic properties have remained largely unexplored. This may be due to difficulties in the preparation of high quality single phase materials.

Recently we synthesised high quality bulk $\text{Ca}_2\text{Mn}_3\text{O}_8$ materials. SQUID measurements indicate antiferromagnetic behaviour ($T_N \sim 52\text{K}$). Furthermore, a deviation from Curie-Weiss behaviour is observed at $\sim 130\text{K}$. Critical scattering is observed below 150K in powder neutron diffraction data suggestive of short range magnetic ordering. Below 50K magnetic Bragg peaks are clearly evident and the material can be determined to exhibit a '2 up - 2 down' antiferromagnetic spin configuration. In this paper we present an in-depth structural and magnetic characterisation of $\text{Ca}_2\text{Mn}_3\text{O}_8$ using powder neutron diffraction and neutron inelastic scattering.

83 Effect of microalloying and thermo-mechanical processing on the properties of quenched-and-tempered X65 seamless pipe

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The need to search for oil and natural gas in more severe environments, including both arctic and deep-water regions, results in Customer specifications that require increased strength and higher toughness in seamless pipe. To meet these demands, tubular manufacturers usually use a combination of chemistry modifications, namely increased alloying elements, and thermo-mechanical-processing techniques (TMCP) to achieve the desired customer requirements. However, Seamless Pipe Mills are equipped with various rolling facilities that affect the properties of seamless pipes due to directionality of rolling sequences. The present study addresses the effect of TMCP processing practices (directionality of rolling) as well as microalloying on the mechanical properties of an API Specification 5L Grade X65 quenched-and-tempered seamless pipe.

84 Tunnel defect in friction stir welded joint: Analysis and consequences for the assembly

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Friction Stir Welding (FSW) was discovered in 1991. It quickly became clear that FSW enabled to overcome many problems associated with traditional welding processes for the joining of aluminium alloys. Indeed, being a solid state process, FSW avoids many of the classical welding defects, but it also creates some specific ones. Standards struggle to accurately describe these specific defects, which is a big issue since not every company owns its own FSW quality standards. However, companies need to have a mean to assess the quality of friction stirred welds for a given application and to be able to define the weld quality level required in the production stage. In an industrial context, tunnel defects may appear for a butt joint configuration. The present work aims at determining the mechanical properties of the joint according to the size of the tunnel. The consequences of this defect for the assembly are also addressed. Experimental work shows that the tunnel defect may or may not be critical depending on the assembly loading configuration.

85 High speed in-situ X-ray tomography applied to advanced materials processing and development

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Synchrotron X-ray sources are capable of delivering high flux of X-ray photons capable of penetrating realistic thicknesses of engineering materials such as aluminium, steel, concrete, or stone. Recent advances in imaging, control, and data processing technology; sophisticated experimental design; and novel mathematical methods for tomographic reconstruction now make it possible to observe the development of internal structures during processing and testing of such materials. With such 'four-dimensional' time-resolved tomography at Diamond Light Source, we have been able to observe microstructural development and to follow changes on the scale of seconds, and even down to 100 millisecond time scale, for such processes as solidification, additive manufacturing welding, crack growth, and fluid transport. Examples from recent work will illustrate the ability of such imaging to reveal previously unobserved behaviour at the microstructural scale.

86 Quantitative atomistic analysis of mechanical relaxation in metallic glasses

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Anelastic relaxation behavior of metallic glasses correlates with useful engineering properties, such as ductility. We have recently determined relaxation-time spectra for metallic glasses from quasi-static and dynamic-mechanical measurements [1,2]. These reveal an atomically quantized

hierarchy of shear transformation zones consisting of ~14 to 33 atoms, exhibiting excellent agreement with Argon's analysis of Eshelby inclusions [3], the activation free energy of which is proportional to their volume. The insights gained from these results have led to important conclusions: a) Even when time-temperature superposition suggests a single activation free energy, it masks a range of discrete values; b) both alpha and beta relaxations can be explained with the same STZ mechanism, each corresponding to a different size range; c) Approximate measurement of the spectrum of activation free energies that relies on the temperature-stepping method [4] leads to a systematic underestimate of the high end of the spectrum.

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87 Supercritical fluids-based technologies for advanced materials

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Nowadays supercritical fluids-based technologies offer fast, sustainable and scalable routes towards high quality nanostructures (NCs) and thin films.

This presentation will be focused first on a brief introduction to supercritical fluids and the associated technologies. The interest of the processes based on a chemical transformation will be discussed thanks to the chemistry in near- and supercritical water/alcohols for the design of advanced nanostructured materials (high quality and unique nanooxides). The need to open the black box of the near- and supercritical processes brought us to develop numerous tools for in situ characterizations. After the discussion will be focused on the understanding of the nucleation and growth of high quality nanooxides in our continuous near- and supercritical processes based on recent research works on three systems giving access to unique NCs: i) the nucleation & growth in supercritical water / ethanol of $\text{Ba}_x\text{Sr}_{1-x}\text{Ti}_y\text{Zr}_{1-y}\text{O}_3$ ($0 \leq x \leq 1$ - BST, $0 \leq y \leq 1$ - BTZ) coupling ex situ and in situ analyses, ii) the formation mechanism of exciton luminescent ZnO NCs exhibiting the optical properties of single crystals and iii) the formation mechanism of CeO_2 NCs in supercritical alcohols.

This knowledge on the nucleation and growth of nanostructures can be transferred to film deposition; this is the supercritical fluid chemical deposition (SCFD) process. SCFD has been used to deposit various metal/oxide nanoparticles and films onto a wide range of substrates for microelectronic, optical and catalytic applications; this will be the last part of this lecture.

88 Growth of polycrystalline diamond films on Cu/CF composite materials using combustion CVD method

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Diamond coatings are investigated for thermal management, wear protection and corrosion resistance in harsh environments. In power electronic industries, copper (Cu), which shows high thermal conductivity, is considered as a promising substrate for such diamond based heat-

spread materials. However, the coefficient of thermal expansion (CTE) mismatch between diamond and Cu induces thermo-mechanical stresses that affect the integrity of the diamond-Cu assembly. In fact, diamond films deposited on Cu substrates, by combustion flame chemical vapor deposition (CVD), tend to peel-off upon cooling due to the compressive stresses present at the diamond-Cu interface. Therefore, a novel approach to ensure the adhesion between Cu and diamond is required.

This investigation is focused on the growth of diamond thin films on Cu reinforced with carbon fibers (Cu/CF) composites. It is expected that the insertion of carbon fibers into the Cu matrix could help anchoring diamond films on Cu by tailoring the CTEs of the Cu/CF composites.

It is found that an increased carbon fiber fraction in the Cu/CF composite leads to a reduced CTE improving, hence, the adhesion between the diamond film and the substrate and reduces residual stresses. At a carbon fiber concentration of 40%, the residual stress of the diamond film deposited on the Cu/CF composite is lower than that on bare Cu. The compressive residual stress on a diamond film deposited on a Cu/CF composite surface was found independent of deposition time and kept constantly below 1 GPa.

89 Recrystallization and grain growth behaviour of an Mg-La alloy after hot-rolling

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The recrystallization and grain growth behaviour of an Mg-1.33La (wt.%) alloy after hot-rolling was investigated using optical microscopy and micro-hardness measurements. Mg-1.33La (wt.%) alloy was hot rolled at 420 °C to two different thickness reductions (20% and 53%) and then annealed at 450° C up to 8 days.

The deformed alloy exhibited minimal dynamic recrystallization that was hindered by the second phase ($Mg_{17}La_2$) precipitation throughout the matrix during the deformation. During annealing, the recrystallized grain boundaries were strongly pinned by the $Mg_{17}La_2$ precipitated phase and this pinning effect was also effective during subsequent grain growth. Annealing treatments at 450°C up to 8 days resulted in normal grain growth. The grain growth kinetics could be described by $D^n = d_0^n + kt$, where n value was found to fall in the range of tabulated data in the literature. The alloy's hardness increased with increasing deformation strain and normal drop of this hardness was illustrated after annealing treatment.

90 Extraordinary structural stability and hardness by decomposition of metastable nanocrystalline solid solutions

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In recent years it has been shown that the formation of non-equilibrium solid solutions in various immiscible binary alloy systems with a positive heat of mixing can be achieved by severe plastic deformation (SPD). In the current study, SPD is used to produce a single phase nanocrystalline supersaturated Cu solid solution in the Cu-Co system, in which up to 26 at.%

Co is dissolved in the fcc Cu phase in the as-deformed state. The work focuses on the phase decomposition process and the microstructural evolution of the metastable solid solution during annealing as well as on its influence on the mechanical properties. The as-deformed and annealed microstructures are studied by transmission electron microscopy, including electron energy loss spectroscopy, and atom probe tomography. The mechanical properties are characterized by nanoindentation. It is shown that the two phases separate by spinodal decomposition during annealing at elevated temperatures, which proceeds on different length scales: The decomposed structure consists of a fine-scaled, compositionally modulated structure in the grain interior and larger, nanometer-sized fcc Cu and Co phases located mainly at the grain boundaries. This unique architecture achieved by phase separation and the way how the Cu and Co phases are distributed can be used as a tool to tailor the mechanical properties of the material. Simultaneously, a high structural stability is maintained in the nanocrystalline material even for annealing durations of several days.

91 The effect of alloying elements on static recrystallization and interphase precipitation behaviors during hot rolling process

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In current, it is of great technical importance to develop hot rolled steel products with high strength and good formability for automobile body parts due to the demand for weight reduction without sacrificing safety of vehicles. According to recent studies, interphase precipitation (IP) hardening can be a promising solution to achieve excellent high strength and superior formability in low alloy steels. However, it is not easy to achieve the IP hardening in hot rolling process because it is not clearly revealed the influence of hot rolling process parameters on interphase precipitates formed during phase transformation. Thus it is also required to evaluate the effect of alloying elements on the IP hardening in hot rolling process. In this study, we aim to clarify the role of the carbide forming elements on the IP hardening through recrystallization behaviors during hot rolling process. Double-pass hot compression deformation tests using a Gleeble thermo simulation machine were conducted to determine the effective rolling temperature for inducing recrystallization with different carbide former elements, such as Ti, Nb and V between rolling stands. Using the results, hot rolled steel sheets were fabricated in pilot scale and the mechanical properties were estimated. Then the optimum conditions of hot rolling process for the IP were discussed.

92 Order and disorder in some photovoltaic materials

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Perovskite solar cells have lately attracted considerable attention due to their high conversion efficiency and low cost. High performance is likely instigated by high optical absorption and good transport properties. The latter property usually correlates with long diffusion lengths, which directly depends on the level of structural order. Diffraction and scattering are among the principal experimental techniques for characterizing crystallite order, disorder, and

associated strains. In particular, pair distribution function (PDF) reveals both short-range and intermediate-range structural order/disorder. Moreover, diffraction line broadening analysis yields information on strains and defects in the structure and Rietveld refinement gives occupational disorder/deficiency of cation crystallographic sites. We will present some experimental and computational results obtained on several photovoltaic samples.

93 Synthesis of SiO₂-CaO-K₂O-Al₂O₃-B₂O₃ glass-ceramics for dental applications

Jae Chul Bang*
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Glass-ceramics with special properties are suitable for application in dental implants. In this study, we synthesized glass-ceramics based on the SiO₂-CaO-K₂O-Al₂O₃-B₂O₃ system using two different methods, sol-gel and conventional melting. The processing methods, compositions, and heat treatment were studied for their effect on the formation of the crystalline phases and on the resulting properties, such as transmittance over the visible spectra, hardness, fracture toughness, and bending strength. Various colors in Vita shades were achieved by the addition of various metal oxides such as Er₂O₃, Fe₂O₃, and Mn₂O₃.

94 IN792 DS superalloy: Optimization of EB welding and post-welding heat treatments

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Electron beam (EB) welding has been used to realize the seams on 2 mm thick plates of directionally solidified (DS) IN792 superalloy. A grid of the samples has been prepared by varying the pass speed v from 1 to 2.5 m/min, while the other process parameters (power $P = 1$ kW, acceleration voltage $T = 50$ kV, beam current $I = 20$ mA) were kept constant. Experiments were carried out both at room temperature and with pre-heating at 200 °C or 300 °C. Once found the best process conditions (pre-heating at 300 °C; $v = 2.5$ m/min) the effect of post-welding heat treatments at 700 and 750 °C for increasing time up to 2 hours has been investigated.

95 Mathematical model of microstructural evolution of hot rolled wire rods for Nb microalloyed steels

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Wire rods are supplied for end users manufactures of products with increasing complexity and mechanical properties requirements. Therefore, wire rods for demanding end users field applications must provide high yield stress, ductility and, in certain cases, toughness. These

properties can be improved simultaneously if appropriate rolling technique and alloy design are used. It has been known for decades in the flat rolling industry that microalloyed steels can be produced achieving the requirements just mentioned. The technology, however, is less often seen applied at the long products industry. In order to obtain a metallurgically sound product, the steelmakers need to run industry scale trials. These however are time consuming, costly and results are not always easy to analyze given the number of variables involved. Often mathematical modelling is an alternative away by which the number of industry trials may be reduced, generating cost savings. There are some models already published in the literature for the particular case of rolling wire rods. They were more concerned in fact with modelling the thermomechanical parameters of the schedule and not with alloy design. This paper presents a mathematical model for wire rod rolling of microalloyed steels in which alloy design is taken into account. The model indicated best alloy composition and rolling practice. These in turn were put to work in mill trials. The predicted and the mill trials results were compared. Reasonable agreement was found proving the model to be a valuable tool in both schedule and alloy design.

96 Routes for increased strength and ductility of Fe-TiB₂ high modulus steels

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Weight saving is a major goal for material design. Lightweight structural metallic materials not only require a lower density (ρ), but also sufficient stiffness (E) in respect to the base material, benchmarked by the specific modulus (E/ρ). As a density reduction is usually coupled with a drop in Young's modulus, conventional alloying offers only limited potential even at elevated contents. Hence, a decreased stiffness by alloying needs to be covered by the material thickness, which reduces the lightweight potential. In order to overcome this detrimental relationship, the most promising material design strategy is to blend stiff and lightweight phases such as borides, oxides, nitrides or carbides with a ductile Fe-base matrix, ideally by in-situ liquid metallurgy synthesis, thus creating high modulus steels (HMS). However, while the physical properties E and ρ can thereby be substantially improved, the implementation of such particles typically deteriorates the steels' mechanical properties. We present an overview on the example of the model system Fe-TiB₂ how strength and especially ductility and toughness of HMS can be significantly improved by optimized co-deformation processes between particles and matrices. Effects of matrix constitution and particle morphology are outlined and discussed together with novel production strategies to achieve such microstructures offering a superior profile of both physical and mechanical properties.

97 Grain boundaries and their junctions by atomistic and mesoscopic simulations

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Grain boundaries and their junctions influence numerous processes in materials science. Grain boundaries are the most important features of a polycrystal, control diverse phenomena and

influence decisively the properties of materials. For mesoscopic grain sizes, the volume occupied by grain boundaries exceeds by orders of magnitude that of other structural elements. However, this difference becomes insignificant in nano-sized materials as triple lines, being the most frequent topological element, can occupy as much volume as grain boundaries in a microstructure. Despite extensive investigation and substantial progress, these crystal features and their influence on microstructural evolution remain in the focus of interest. The present contribution is dedicated to the investigation by MD-simulations of atomistic mechanisms of grain boundary migration in bi- and tri-crystals. The mechanisms are correlated to experimental findings and their effect implemented in mesoscopic simulations of grain growth to predict their impact on microstructural development.

98 Human liver microtissue spheroids in hollow fiber membrane bioreactor

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The development of bioartificial devices that are able to favour the liver reconstruction and to modulate cell behaviour is an important challenge in liver tissue engineering.

As strategy we realized liver microtissue spheroids in a crossed hollow fiber membrane bioreactor, which was developed for the in vitro long-term perfusion. The bioreactor consists of two types of hollow fiber (HF) membranes with properties cross-assembled in alternating manner: modified polyetheretherketone (PEEK-WC) and polyethersulfone (PES), which perform different functions. PEEK-WC HF provide cells nutrients and metabolites whereas PES HF removes catabolites from cell compartment mimicking in this way the in vivo arterious and venous blood vessels. The combination of these two fiber set creates three compartments: two intraluminal compartments of PEEK-WC HF and PES HF in which the medium flows and one extraluminal compartment represented by extracapillary network formed by the fibers in which spheroids are cultured. The geometry of the bioreactor ensured optimal perfusion conditions, which plaid a crucial role together with the surface and transport properties of the HF membranes in guiding the self-assembling of liver microtissue spheroids. Indeed, adjacent spheroids fuse in the extracapillary space forming liver microtissues that expressed liver specific functions in terms of urea synthesis, albumin production and diazepam biotransformation at high levels up to 2 weeks. Furthermore, the bioreactor provided an adequate oxygenation of liver microtissue spheroids maintaining oxygen concentration levels well above the in vivo liver periportal zone concentration.

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99 A Study on the microstructural characterization of René 142 deposited atop René 80 processed through scanning laser epitaxy

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Advancements in the design, optimization and manufacture of turbine engine hot-section components during the past few decades have contributed enormously to the improvement in power-ratings and efficiency levels of gas-turbine engines. Nickel-base superalloys are extensively used to produce the hot-section components as this class of alloys offer improved creep strength and higher fatigue resistance compared to other alloys due to the presence of precipitate-strengthening phase such as Ni_3Ti or Ni_3Al (γ' phases) in the normally face centered cubic (FCC) structure of the solidified nickel. Although this second phase is the main reason for the improvement in properties, it also results in increased processing difficulty as these alloys are prone to crack formation. In this work, we demonstrate powder-bed additive manufacturing of René 142 onto René 80 substrates through scanning laser epitaxy (SLE). René 142 is a high strength, nickel base directionally solidified (DS) alloy that has high rupture strength, excellent resistance to grain boundary cracking, and superior high-velocity oxidation resistance. Successful deposition of René 142 on René 80 provides an avenue to repair legacy hot-section components by depositing superior quality alloys at the damage locations. The René 142 is demonstrated to follow the polycrystalline or EQ morphology of the underlying René 80 substrate. The SLE processed René 142 reveals dense, crack-free deposits and microstructure refinement along with superior yield strength compared to the cast substrate. This work is sponsored by the Office of Naval Research through grants N00014-11-1-0670 and N00014-14-1-0658.

100 Influence of material microstructure and thermal residual stresses on macroscopic fracture parameters and elastic properties of metal-ceramic composites

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This paper investigates the interplay between material microstructure and processing-induced thermal residual stresses (TRS) in particulate bulk MMC's with the main objective to explore their combined effect on the macroscopic fracture toughness and material properties (E modulus, bending strength) of the composite. The considered materials are hot pressed chromium-alumina bulk composites doped with rhenium (motivated by their potential applications in transport and energy sectors). The reported research includes the processing of MMC by powder metallurgy techniques (HP and SPS), microscopic analysis of microstructure with special focus on micro-CT scanning, measurements of TRS by neutron diffraction (ND) method and numerical modelling of TRS by FEM based on micro-CT images of real material microstructure. Several compositions of $\text{Cr(Re)/Al}_2\text{O}_3$ system and different particle sizes were used in the sintering process to assess the effect of microstructure on the TRS. Spatial distributions of TRS measured by ND are taken as supporting information when interpreting the results of K_{IC} measurements in a four point bending test. Numerical micro-CT based models

are constructed to predict the TRS, Young's modulus and bending strength with account of TRS-induced damage of the ceramic phase. A good predictive capability of these TRS models is achieved which may be important considering the cost of beam time for ND experiments. Finally, the large pool of experimental data and modelling results is discussed and conclusions are drawn as to the TRS/microstructure effect on the fracture toughness of the MMCs in question.

101 Correlations between defect content, mechanical properties and fractographic investigation of AlSi9Cu3(Fe) alloy reference castings

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High Pressure Die Casting (HPDC) is a foundry process particularly suitable for high production rates and applied in several industrial fields, but the amount of scrap, caused by defects or incomplete filling, is sometimes very high. For this reason it is important to know which are the main causes of defect formation and their effects on microstructure and mechanical properties. This paper presents the qualitative and quantitative results of a study conducted on AlSi9Cu3(Fe) alloy castings, referred to as Horse-shoe Reference Castings, specifically designed to generate different kinds of defects with different severity levels. The work focuses on the correlations obtained between the casting mechanical properties, their defect content in terms of porosity and oxide films and the process parameters adopted, mainly first and second phase plunger velocities, switch point and intensification pressure. The three point bending test was applied to two appendixes of the casting. The fracture surfaces were studied by scanning electron microscopy (SEM) and optical microscopy (OM) revealing the presence of oxide films and other casting defects, the porosity content and the eutectic fraction distribution over the cross section, focusing on the half subjected to tensile strength. A global quality evaluation of the casting was obtained, comparing two areas located at similar distances from the in-gate and supporting it with the simulated temperature distribution and the infrared images acquired. The second phase plunger velocity showed the main effects on the casting defect content, leading to fewer oxide films and inclusions and more reliable mechanical properties.

102 Development of advanced flux cored wires for modern thermal power plants

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The efficiency of thermal power plants can be improved by using higher steam parameters. Therefore, appropriate steels and welding consumables with improved creep strength at elevated temperatures are necessary. Worldwide research activities within the last decades enabled an increase of the operation temperature of modern power plants up to 620 °C.

As flux cored wires for gas metal arc welding offer several technical and economic advantages they become more and more popular and also matching flux cored wires for welding P91 and P92 are already available for several years. The latest development is a matching flux cored

wire for welding the Co-alloyed cast steel CB2, which is used for turbine and valve casings. With enhanced properties of the materials the microstructure became more complex and the demands on accurate welding processes and high quality welding consumables are increasing. Requirements of $Mn+Ni \leq 1.2$ wt% or even ≤ 1.0 wt% were progressively included into customer requirements in order to ensure PWHT below the A_{c1} temperature. Furthermore, requirements of a low amount of trace elements and reduced content of diffusible hydrogen emerged, and forced modifications of the flux cored wires. In this work the necessity of these requirements is discussed and the characteristics of modified, advanced flux cored wires are shown.

103 Bulk ultra-fine grained materials obtained in explosive welding process

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The aim of this work was to investigate the microstructure and mechanical properties of Ti6Al4V alloy and aluminium alloy 2519 plates cladded by the method of explosive welding in order to obtain bulk materials with the ultrafine grained structure. The samples were processed in two versions: with and without an aluminium alloy 1050 interlayer plate. In both types of samples high grain refinement was observed in aluminium zones. The results demonstrated that both Ti6Al4V/AA2519 and Ti6Al4V/AA1050/AA2519 composite plates exhibit good quality bonding. Scanning electron microscopy observations and energy-dispersive X-ray spectrometry (EDS) results revealed that bimetallic nano-grains of Ti/Al phase were forming in the interlayer. In the samples without aluminium interlayer the thickness of bimetallic Ti/Al interlayer was significantly smaller. However, near the joint one can see the grid in the grain boundaries rich in copper. These intermetallic inclusions were often accompanied by nano-cracks. The value of microhardness decreased when the distance from the interface increased.

104 Influence of thermal history on the hot ductility of Ti-Nb microalloyed steels

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The hot ductility of Ti-Nb microalloyed steel has been investigated to evaluate the sensitivity to surface crack formation during the continuous casting process. Tensile samples were subjected to different thermal treatments and were tested at deformation temperatures ranging from 650°C to 1000°C using a strain rate of $10^{-3}s^{-1}$. It has been found that the investigated steel evinced poor ductility over almost the whole testing temperature range characterized by marked grain boundary cracking, irrespective of which thermal cycle has been utilized or whether the samples have been melted or only reheated. Microstructural examinations and supplementary thermo-kinetic computer simulations revealed distinct Ti-Nb precipitation throughout the microstructure being responsible for the deteriorated materials hot ductility.

105 Innovative experimental approaches and physical measurement methods for fatigue monitoring and life assessment

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Reliable assessment of fatigue performance usually requires extensive experimental efforts. Therefore, short-term methods to estimate fatigue limit, HCF and LCF behavior are of great interest for component design and material development. As straight-forward approach, the fatigue limit could be roughly identified with the occurrence of first plastic deformation in load increase tests (LIT). This method lacks of two shortcomings: (i) Detection of transition from elastic behavior to cyclic plasticity in stress-strain hystereses is limited by inevitable signal noise. (ii) In case of pronounced cyclic hardening / softening, LIT don't resemble cyclic deformation at constant amplitude sufficiently precise.

Therefore, improved approaches were developed to overcome issue (i) by applying high precision electrical resistance and temperature measurement during LIT which proved to be way more sensitive to cyclic microplasticity than plastic strain amplitudes measured by extensometry, especially in case of high strength materials. Issue (ii) is tackled by re-calculating the cyclic deformation determined in LIT to constant amplitude tests and deriving the S/N curve from these data. In case of limited availability of test material, the cyclic hardening potential can be evaluated from instrumented cyclic indentation tests, which currently does not allow lifetime assessment, but has been successfully used to compare and rank fatigue properties of high strength and case hardened steel. The talk gives an overview of these methods and addresses innovative non-destructive approaches for monitoring the fatigue state of metals and hybrid welds using ultrasonics and measurement of magnetic properties which are currently under research at the authors' institute.

106 Novel concepts for the application of magnesium sheets and profiles in crash loaded vehicle areas

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As the lightest structural metal, magnesium alloys have been attractive to reduce vehicle weight and emissions by lightweight design in the automotive industry. Crashworthiness is not a physical property itself, but correlates with the material's ductility and structural design. Magnesium is known to be a material with lower failure strain than other metallic structural materials. Therefore the use of magnesium in crash-related areas is more challenging compared to steel and aluminum. In structures with a bending load, like e. g. the bumper or the sill, crash properties can be significantly improved by filling profiles with a stabilizing core. In order to evaluate the crashworthiness of this hybrid structure under bending loads, both empty and polyurethane foam-filled rectangular section beams were constructed and tested by using the quasi-static/dynamic three-point bending facilities at German Aerospace Centre (DLR) – Institute of Vehicle Concepts. For structures with axial crash loads the normal buckling mode will lead to a very early fracture of the magnesium part. In collaboration with researchers from the University of Windsor and the University of Waterloo, novel technologies for energy

absorption which are based on cutting or peeling mechanisms have been developed and investigated, which allow the use of magnesium in these challenging applications. Results of the joint research will be presented.

107 Influence of microalloying elements Ti and Nb on recrystallization during annealing of advanced high-strength steels

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To meet the need for weight reductions in the automotive industry, new steel grades combining high strength and high formability are being developed. Dual Phase steels (DP) achieve these requirements. DP steels are processed by intercritical annealing of cold-rolled low-carbon steels in order to produce ferrite-austenite mixtures. Subsequent cooling leads to a complex microstructure made of a hard martensitic phase dispersed in a soft ferritic matrix.

As tensile strength and ductility of DP steels are highly dependent on the microstructure obtained at the end of the intercritical annealing, microstructural evolution during this stage is of considerable interest. During the first stage of annealing, microstructural changes include precipitation of microalloying elements and recrystallization of the ferritic matrix. Ti and Nb contribute to the steel strength by precipitation hardening and grain refinement but they are also expected to have an influence on recrystallization kinetics. In order to study and quantify this influence, the mechanisms of precipitation and recrystallization are characterized depending on the heating rate for various cold-rolled low-carbon steel grades with the same initial bainite-martensite microstructure but different microalloying contents. Qualitative and quantitative data regarding these mechanisms are obtained by matrix dissolution techniques, SEM-FEG, TEM-EDX, and Atom Probe Tomography. A simple modelling approach is proposed to rationalize the effect of Ti and Nb depending on the processing parameters.

108 Regularities of microstructure evolution and strengthening mechanisms of austenitic stainless steels subjected to large strain cold working

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The deformation microstructures and their effects on mechanical properties of austenitic stainless steels processed by cold rolling at ambient temperature to various total strains were studied. The structural changes were associated with the development of deformation twinning owing to low stacking fault energy. Moreover, the austenite is meta-stable at room temperature. Therefore, the strain-induced martensitic transformation took place during cold rolling. Both the deformation twinning and strain-induced martensitic transformation promoted the grain refinement during cold rolling, leading to nanocrystalline structures consisting of a mixture of austenite and martensite grains with their transverse grain sizes of 50-150 nm containing high dislocation densities. The rolled samples experienced substantial strengthening resulted from high density of strain induced grain/phase boundaries and dislocations. The yield strength of

austenitic stainless steels could be increased to 2000 MPa after rolling to total strains of about 4. The contributions of structural and substructural strengthening of austenite and martensite to overall yield strength are discussed.

109 Effect of Ga additions on the glass formation and mechanical behavior of Ti₄₀Zr₁₀Cu_{36-x}Pd₁₄Ga_x (x = 2-10 at.%) bulk metallic glasses

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Ti-based bulk metallic glasses (BMGs) show good prospect in the field of bio-implant materials due to their low Young's modulus, high strength and good biocompatibility. However, the major problem facing the development of biomedical metallic glasses is the one of inducing amorphization without using any harmful alloying additions. In recent past Ti-Zr-Cu-Pd based BMGs show most promising results. However, Cu is considered as not biocompatible and it is observed that all Ti-based BMGs contain large amount (>35 at.%) of Cu. Therefore, it is necessary to develop new Ti-based BMGs by replacing Cu completely or partially without affecting the glass forming ability.

In the present work we report new high-strength Ni-free Ti-based BMGs belonging to the Ti-Zr-Cu-Pd-Ga system. The influence of Ga additions on glass forming ability, thermal stability and mechanical behavior of Ti₄₀Zr₁₀Cu_{36-x}Pd₁₄Ga_x (x=2, 4, 8 and 10 at.%) bulk glassy alloys was thoroughly studied by a combination of X-ray diffraction, differential scanning calorimetry, mechanical tests and ultrasonic measurements. It was found that Ga additions improve the glass forming ability as well as increases the thermal stability of the glassy structure. Furthermore, the alloys show higher compressive strengths than Ti₄₀Zr₁₀Cu₃₆Pd₁₄ BMG. However, additions of Ga by 8-10 at.% decrease the ductility. Ti₄₀Zr₁₀Cu_{36-x}Pd₁₄Ga₂ shows the best combination of compressive strength and ductility (~2150 MPa, 4.09% fracture strain) among all studied alloys. Funding from the EC (7. FP MC-ITN VitriMetTech, GA no. 607080) is gratefully acknowledged.

110 Solid state welding of different material – a comparison between steel/aluminum and copper/aluminum

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Thermal joining of hybrid structures is a challenge in a high number of applications nowadays. The combination of different mechanical properties, i.e. stiffness, strength, or of different electrical and thermic properties is only one of the purposes, which have to be reached via multi-material-mix. Automotive represents an high impact product and has been driver for the development of thermal joining procedures for material mixing and in the last years.

In this paper the two combinations steel-aluminum and copper-aluminum will be compared from the point of view of solid state welding. For both the elementary systems Fe-Al and Cu-Al a complete miscibility in the liquid phase is present, while during solidification IMC are

forming and growing, having a negative effect on deformability of the joint. Solid state joining allows it, to perform a welding below the liquidus-temperature, depending on the diffusion and interdiffusion of atoms from the one part into the other and viceversa. The two systems have very different properties, which will be shown in the talk and presented.

As results investigation regarding welding of Al/Cu and Steel/Al via resistance bonding, us-welding and friction welding will be reports. The different diffusion coefficients allow it, for example, the welding of Al and Cu at a temperature of approx. 550°C via an eutectic phase even for short times (<1-2 Sek.).

111 Functional materials deposition by magnetron sputtering

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Developing new functionalities is highly related to new materials. Nevertheless, prior to development of new devices, two major challenges have to be tackled. The former one is to obtain thin layers of active and functional materials. The second challenge is to recover the functional properties on thin layers. For several decades, magnetron sputtering is a widely used deposition technique in many applications domains and among them the earliest driving one is certainly microelectronics. Moreover, magnetron sputtering enables to deposit well-crystallized films of insulating or conducting materials, at low temperatures, over large areas, while controlling the film composition and microstructure. After a review of some recent developments, the basis of a well established know-how in deposition process and functional structures will be presented. In particular, studies on ternary and quaternary compounds will be described. Over the past decade, several deposition processes have been developed within our research team for various functional materials, including the use of home-made target by SPS. The presentation will highlight examples both by non-reactive magnetron sputtering in Ar or reactive processes in standard mixtures (O₂, N₂) or non-conventional.

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112 Preceramic polymer-derived sphene bioceramic coating on cpTi substrates for orthopaedic implants

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In order to improve the bioactivity of orthopaedic implants, their surface can be coated with a bioactive ceramic layer, such as hydroxyapatite (HAP) or bioglasses. The major drawback of the HAP coating is that it cannot maintain long-term stability, and in certain situations, delamination of the HAP coating from the alloy occurs due to its poor bond strength and insufficient chemical stability.

Sphene (CaTiSiO₅) ceramics have CTE ($6 \times 10^{-6} \text{ K}^{-1}$) similar to that of Ti, possess excellent chemical stability and have the ability to enhance the proliferation and differentiation of human

primary osteoblast-like cells (HOBs).

In this work, a commercially available preceramic polymer was used as source of SiO_2 for the synthesis of CaTiSiO_5 (spinel) bioceramic. Nano e micro sized powders of TiO_2 and CaCO_3 were used as source for Ti and Ca respectively. The powders suspension was sprayed on the surface of cpTi by a commercially available airbrush.

The deposited coatings were heat treated and the synthesized ceramic coatings were characterized in terms of composition, morphology and adhesion with the cpTi substrate.

113 Effect of microstructure, texture, and crack trajectory on small crack growth in Ti-6Al-4V subjected to dwell fatigue

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It is understood that fatigue lifetime can be severely reduced under dwell fatigue loading conditions. Influencing minimum lifetime is the accelerated growth rates of microstructurally small cracks at an equivalent driving force (ΔK) compared to long crack growth rates. Variability in small crack growth is strongly influenced by microstructure and localized preference towards “hard” oriented grains on or near (0001) planes. In this study, traditional roughness parameters were used to describe local microstructural interactions at the crack tip in Ti-6Al-4V as well as quantify crack trajectory and its deviation from pure mode I fracture. These factors along with the localized texture around each observed crack is correlated with their respective growth rates in an attempt to describe their influence on dwell fatigue crack growth.

114 Solid oxide fuel cell and stack development at Forschungszentrum Jülich

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Based on more than 20 years SOFC scientific development, JÜLICH has achieved remarkable improvements of cell and stack performance. Planar SOFC stacks with mixed conducting cathodes on anode substrate cells of the dimension $20 \times 20 \text{ cm}^2$ have been manufactured and tested in various stack sizes with power outputs of up to 5 kW, of which four were operated in a 20 kW system. In parallel to the cell development the stack design was improved aiming at enhanced thermo-mechanical robustness. Thereby a stack in the kW range could be cycled successfully 100 times between 200 and 700°C. To be able to integrate also cells from other manufacturers the design was modified towards a picture frame layout, incorporating currently four $10 \times 10 \text{ cm}^2$ cells in one layer. First successfully operated stacks showed an area normalized performance comparable to short stacks with $10 \times 10 \text{ cm}^2$ cells. The 20 kW system set into operation in 2012 was operated for about 7000 h in total proofing thereby the suitability of the module and system concept developed in JÜLICH. The long-term tests with short stacks operated at 700 °C and 0.5 Acm^{-2} have now reached operation times of 70,000 h (degradation

0.6%/kh) and 34,500 h (degradation 0.3%/kh, based on the implementation of an improved protective layer), respectively. The use of the SOFC as electrolyser (SOE) revealed very good operational behaviour and moderate degradation rates. Furthermore, the simultaneous electrolysis of water and CO₂ works very well.

115 Crystallography and self-accommodation of martensitic transformation in epitaxial Ni-Mn-Ga thin film

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Epitaxial Ni-Mn-Ga thin films are of potential candidates for sensors and actuators in micro-electro-mechanical systems [1]. Previous studies have demonstrated that the complicated microstructure are the major obstacles to achieve large magnetic field induced strain (MFIS) in epitaxial Ni-Mn-Ga thin films [2-3]. To further improve the MFIS in epitaxial Ni-Mn-Ga thin films, their microstructure has to be modified through the magneto-mechanical-thermal training [4]. The deep understanding of crystallography and transition sequence of martensitic transformation in epitaxial Ni-Mn-Ga thin films is helpful to the magneto-mechanical-thermal training. In the present work, scanning electron microscopy and electron backscatter diffraction technique reveal that the microstructure of epitaxial Ni₅₀at.-%-Mn₃₀at.-%-Ga₂₀at.-% thin films are composed of six different orientated variant colonies. In each martensite variant colony, there are four different orientated 7M martensite variants, which indicates that the crystallographic orientation relationship between austenite and 7M martensite during martensitic transformation are of Pitsch relationship, i.e. $(101)_A \parallel (1-2-1)_{7M}$, $[10-1]_A \parallel [-1-11]_{7M}$. The six 7M martensite variant colonies are transformed from six $(101)_A$ planes of austenite. In addition, the four 7M martensite variants in each martensite variant colony are of self-accommodated microstructure. The mechanism of self-accommodated microstructure in epitaxial Ni-Mn-Ga thin films was revealed by crystallographic calculation.

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116 Effect of microalloying elements on phase transformation, microstructure and mechanical properties in dual-phase steels

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Dual-phase steels are the most important AHSS grades for automotive application. Microalloying elements such as Ti, Nb or B are widely used to improve the strength of dual-phase steels. Thus, understanding the influence these elements on the microstructure and mechanical properties of dual-phase steel along the processing route is critical for the

development of new steel grades. In this work, different microalloying elements were investigated, separately or in combination. The influences of the different elements on the microstructure and mechanical properties of dual-phase steels in the hot-rolled condition and after annealing of cold-rolled material. Dilatometer measurements were performed to investigate phase transformation during typical continuous annealing treatment of dual-phase steel after cold rolling. It was shown that, for example, Ti has a strong influence on the mechanical properties of hot-rolled material while the influence in annealed materials after cold rolling was relatively small. Conversely, B had a strong influence on cold-rolled materials but an insignificant influence on hot-rolled materials.

117 First-principles modeling of copper impurity diffusion in TiN

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TiN is an important material used as a diffusion barrier in microelectronic devices to prevent copper from contacting silicon. There is however little known about the elementary atomistic processes underlying the excellent performance of TiN. The scarce experimental data presented in literature has a significant spread and the available theoretical works provide only qualitative estimates of the different atomistic mechanisms of copper diffusion. In this work, therefore, we quantitatively evaluate barrier properties of TiN against copper diffusion and estimate the effect of the off-stoichiometry of TiN using a first principles approach based on density functional theory. To this end we calculate concentrations of the intrinsic point defects in titanium nitride as a function of composition via the grand-canonical thermodynamic formalism based on the dilute solution model. We study the site preference of Cu impurity atoms on the TiN sub-lattices by calculating the corresponding defect formation energies, taking into account the effect of off-stoichiometry. Finally, we calculate the stoichiometry-dependent diffusion coefficients for three diffusion mechanisms: the interstitial mechanism and the vacancy-mediated mechanism on the Ti- and N-sublattices.

118 Effect of temperature and strain rate on the mechanical properties of 99.5 aluminium rods extruded by KOBO

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In the present study, aluminium rods cold extruded the process of direct extrusion by KOBO method in two variants: 1/ with varying (decreasing) frequency of die oscillations necessary to maintain a constant extrusion force, and 2/ with constant frequency of die oscillations, leading to a decrease in the extrusion force. The tensile test of rods was carried out in a temperature range of 20 - 200°C and at a strain rate from 8×10^{-5} to $8 \times 10^{-1} \text{ s}^{-1}$. Significant differences in the elongation of the tested rods were observed. It was found that rods extruded at variable die oscillations and stretched at room temperature had similar elongation, independent of the strain

rate. With the increase of temperature, the elongation of samples stretched at a low speed was growing from a value of about 8% at room temperature up to 40% at 200°C. At high strain rates, despite the increasing temperature, the elongation remained at the same level, i.e. 5-6%. In rods extruded at constant die oscillations, the elongation at a low strain rate was growing with the temperature from 10% at room temperature up to 29% at 200°C. At high strain rates, the elongation decreased from 28% at room temperature to 11% at 200°C. The results were interrelated with examinations of the structure of rods and fractures of tensile specimens. In the material extruded by KOBO method with constant die oscillations, the beginnings of the recrystallization process were observed, absent in the material extruded at variable die oscillations.

119 Miniaturized flow-through bioreactor for processing and testing in pharmacology

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Conventional Bioreactor systems for cultivating cells in Life Science have been widely used for decades. An in vitro cell culturing bioreactor should reliably and reproducibly mimic the in vivo microenvironment of the cultured cells. Mammalian cell cultures normally are performed out in conventional bioreactor devices such as culture flasks and culture-dishes. However, these tools have fundamental limitations in that they are not appropriate for high throughput screening and consume a considerable amount of resources and time [1]. Therefore, there is a trend towards miniaturization, disposables and even micro platforms that fulfill increasing demands strongly aiming for production and testing of novel pharmaceutical products. Here we present the development and manufacture of a disposable miniaturized flow-through bioreactor system that can be produced in large numbers at low costs. Nano-porous hollow fibers are located at the fluidic sources and drains of the miniaturized bioreactors and retain cells. The necessary mixture of oxygen and carbon dioxide is provided via diffusion through a semi-permeable membrane. Fluidic connections allow the continuous feeding of the cells adding nutrient solution at constant rates at the inlet of the micro bioreactor and removing the solution at the same rate at the outlet [3]. This medium can be collected and used for subsequent analysis. Different designs and concepts for such bioreactors were carried out with varying numbers of plates, and integrated or joined miniaturized reactor chambers. First tests show full technical and biological functionality, biological cells could successfully be cultivated at high viability rates for some days.

120 Torsional piezoelectric strain in monocrystalline paratellurite

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Crystals of the 422 symmetry class exhibit interesting piezoelectric behavior, as their piezoelectric tensor has only a single non-zero coefficient, d_{123} : such unique behavior has the

potential to enable novel gyroscopic sensors and high-precision torsional MEMS actuators. Although alpha-phase tellurium dioxide (paratellurite, $\alpha\text{-TeO}_2$) is one of the few materials belonging to this symmetry class, this material has been primarily studied for its interesting optical properties. Indeed, a large uncertainty in the piezoelectric coefficient of paratellurite exists, with d_{123} measurements on single crystals ranging from 8.13 pC/N to 14.58 pC/N; this large uncertainty results from the difficulty in using conventional piezoelectric characterization techniques on paratellurite, and impedes adoption of this extraordinary material. The present study characterizes the piezoelectric behavior of this interesting material using two independent techniques, (1) equivalent circuit analysis using electrochemical impedance spectroscopy (EIS), and (2) a three dimensional laser Doppler interferometer system. The experimental results are validated using analytical simulations for dynamic excitation conditions over a frequency range of 30 kHz to 1 MHz.

121 Evaluation of weld parameters on the mechanical properties of friction stir welded dissimilar Al alloy lap joints

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Friction stir welding (FSW) is an energy efficient method to produce strong joints in aluminum alloys that are otherwise difficult to join by conventional arc-welding processes. Due to rising fuel efficiency requirements, the utilization of AA7075 is increasingly migrating from aerospace to automotive applications. There is also an interest in the application of FSW to join dissimilar aluminum alloys, since hot cracking can be avoided. The geometry of the FSW tool is a parameter that plays a major role in both the quality of the weld and the speed at which it can be made. The present work compares three tool designs for FSW lap joints between AA7075-T6 and Al-Mg-Si thicknesses of 2mm and 1mm, respectively. Tool rotation speeds are kept constant at 1120 rpm, while travel speeds from 125 to 355 mm/min are compared in terms of joint mechanical properties and temperature profiles. Microstructures, hardness profiles, and tensile strengths, are examined to identify suitable parameters for production applications.

122 Nanotechnology for aluminum mechanical properties improvement

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The novel approach of influence on aluminum alloys structure formation by various nanomaterials affecting the solidification process will be discussed. Fine microstructure of the Al alloys has been obtained as the result of the direct influence of the nanomaterials addition to the metal solidification. It has been found that aluminum alloys mechanical properties improved after addition of relatively small amount of nanomaterials. The obtained results show unusual behavior, under which the elongation increases while metals strength remained unchanged. We found that grain-size strengthening mechanism is responsible in the process. A high resolution

transmission electron microscopy (HR-TEM) study indicated high concentration of dislocations near grain boundaries in the modified alloys which serve as obstacles to dislocation motion.

123 Plasma-based aerosol process for the production of single digit nanometer-sized particles from metal, oxide, semi-conductor and polymer

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This paper depicts a plasma process for the production of nanoparticles with tunable size and the same composition than the electrode in Dielectric Barrier Discharges (DBD). Such nanoparticles suspended in gases, also called aerosol with narrow size distribution are targeted for their size-dependent properties. However, classical ablation/vaporization processes by laser and spark (1-100 mJ per spot) usually leads to 10-100nm agglomerates of nanoparticles.

A compact, inexpensive and simple DBD design is presented for the production of such targeted single digit nano-sized particles. With smaller energy per filament in DBD (0.1-100μJ), it is first confirmed that the initial local vapor flux is produced from the spots of interaction between plasma filaments and different materials (Al₂O₃, Au, Ag, and Cu, Si, polymers). Then, as an example with a metal electrode, amorphous and crystalline pure primary metal nanoparticles are produced by physical nucleation in expanding vapors jets. Finally, small agglomerates with diameters still below 5 nm are formed by agglomeration of these primary particles at the end of the vapor jet expansion, as well as after the production during the transit between subsequent filaments in the DBD. The first local agglomeration step can be limited at reduced energy per filament by lowering the initial vapor flux, while the second growth step depends on the transit time in the DBD. This plasma process represents an alternative for nanotechnologies, since it is performed at atmospheric pressure and can be used to reach size-dependent properties of nanomaterial, without any precursor or solvent.

124 Thermal design of hard coatings

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Thermal transport inside of hard coatings is getting more and more attention due to ever increasing demands on coating durability. Hard coatings are applied on surfaces that face extreme thermal conditions such as on cutting tools, turbine blades and metal die casts. Recent developments in industrial scale coating fabrication and in thermal property measurement techniques allow to gain insight and customize the thermal properties of the coatings. Thermal conductivity can be varied accurately and over a wide range in typical transition metal oxynitride coatings such as Ti-O-N and Cr-O-N by adjusting deposition parameters. This can be used to fabricate graded and multilayered coatings that feature significant anisotropy of thermal conductivity even when employing industrial scale coating systems. The effects of common fabrication defects and thermal interface resistances were evaluated to be manageable or even beneficial. Coatings with high anisotropy of thermal conductivity were realized that have the potential to lower thermal stresses and lower spot temperatures at the interface between coating and substrate.

125 Role of plasticity during the microstructure evolution in metallic alloys

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Many materials used in every day life have a complex internal structure organized at different scales with a texture of grains, and a complex pattern of thermodynamic phases at lower scale. This microstructure strongly impacts the movement of crystalline defects in the materials and therefore controls the mechanical behavior. A detail understanding of the microstructure is therefore required for the development and optimization of new heterogeneous materials.

The phase field method has emerged as the most powerful method for tackling microstructure evolutions during phase transformations when elastic coherency stresses are generated. However, in many materials and especially at high temperature, the microstructure evolution is coupled with plasticity, and there is currently a great research effort to extend the phase field method to take this coupling into account.

I will first review several attempts to perform this coupling using a continuous description of plasticity. Then, I will describe a recent modeling framework in which a classical phase field model (for the description of the phase transformation) is coupled with a crystal plasticity model based on dislocation densities. The latter model uses a storage-recovery law for the dislocation density of each glide system and a hardening matrix to account for the short-range interactions between dislocations. The proposed model will be first applied to simple microstructures to illustrate the importance of the size effect and of the anisotropy of plasticity in heterogeneous materials. Finally, the coupled model will be applied to study rafting of ordered precipitates observed in Ni-based superalloys during creep.

126 Phases stability study of the shape memory alloy CuAl-X (X = Be, Zn, Ti, Ni, Ag and Au) by ab initio calculations

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The Shape Memory Alloys (SMA) have outstanding mechanical properties such as shape memory effect and superplasticity. Plastically deformed at a given temperature, these alloys can recover their initial shape by heat treatment (shape memory effect). The SMA also have different thermo-mechanical behavior from the other materials; their superplasticity is manifested by a reversible deformation of up to 3% to 10%. This particular behavior is related to the existence of a phase transformation called a martensitic transformation. The phase stability is very sensible to the composition (stoichiometric variation) and defects (point defects as vacancies, substitutions, doping...).

Many studies have been done on the Cu-Al based SMA specially on the beryllium addition effects. The sharp doped effects with the beryllium atom in the Cu-Al compound are not very known. Where are the beryllium atoms localized in the structure? What are the effects of the concentration on the phases stability? Are the ordered phases more stable than the disordered phases? We have determined, by Density Functional Theory method, the structural and the elastic properties, the stability of the different phases of pure Cu₃Al compound. The effect of

vacancies, the substitutions and the doping effect by a X element as well as the influence of these defects on the crystallography was studied. The atomic concentration of defects varies between 1,56% to 6,25%. For different phases, an order of stability had been established and following the nature of the X atom, the impact on the phases stability is different.

127 Mechanical properties of sol-gel hybrid coatings

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Surface coatings represent a flexible and economic mean to protect or modify the functional behaviour of glass component without having to modify the composition [1]. Sol-gel process is very attractive to prepare easily inorganic thin films with flexible thickness (by controlling the sols rheology) on small and various shaped materials by dip or spin-coating. Moreover, mild synthetic conditions provided by sol-gel chemistry allow to incorporate organic components into the inorganic network, producing hybrid organic-inorganic materials that may combine the qualities of both polymer and oxide materials. For example, mechanical properties are tunable between those of glasses and those of polymers by raising the initial composition of precursors. Instrumented nanoindentation has become widely used to test thin solid films and is applied to measure mechanical properties of sol-gel derived hybrid thin films. The nanoindentation technique allows investigating the elasto-plastic response of hybrid coatings as well as their brittle behavior and adhesion to the glass substrate. We shall discuss the indentation response of different complementary systems allowing for the control of organic-inorganic interfaces in terms of strength (class I and II) and morphology: PMMA-silica, PMMA-CNT, goethite-PHEMA [2]. [1] E. Le Bourhis, Glass mechanics and Technology, 2nd Ed., Wiley, Weinham, 2014. [2] F. Mammeri, J. Teyssandier, C. Darche-Dugaret, S. Debacker, E. Le Bourhis, M. M. Chehimi, J. of Colloid and Interface Sci., 433, 115 (2014)

128 Rapid, high-throughput mechanical properties measurements of additively manufactured metals

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Additive Manufacturing offers the opportunity to rapidly produce customized complex parts in hours rather than days to months associated with conventional manufacturing methods. This presents both a challenge and an opportunity for material qualification. To complement the rapid nature and geometrically complex capabilities of additive manufacture, a rapid high-throughput tensile test method has been developed and used to evaluate the properties of a PH17-4 alloy produced from two commercial vendors. The method permits ~100 tensile tests in a few hours of test time and for the same cost as a few conventional tensile tests, allowing for profound statistical insight into the extreme-value tails of property distributions. Using this high-throughput method, we were not only able to compare average material properties between vendors and to wrought/cast product, but also able to compare the distribution of properties. Moreover, the method revealed rare, statistically anomalous failures associated with sporadic manufacturing defects. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration

under contract DE-AC04-94AL85000.

129 Electrodeposition of sulfonated poly(phenylene oxide) as solid electrolyte in 3D microbatteries

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Micro-batteries are devices that can power a wide range of portable electronic appliances. The need of combining higher power and energy densities with long durability for such devices is still far from being fulfilled. Moreover, durability issues due to dendrites formation and electrolyte reactivity with electrodes are making this challenge even more difficult to be tackled; finding a good balance between performances and durability is of utmost importance. A significant performance improvement might be achieved if a 3-dimensional design of the electrode/electrolyte interfacial regions can be realized, while the presence of a suitable solid electrolyte would definitively address the durability issue. However, no practical realization of this concept has been reported yet. Our work is intended to develop such devices based on TiO₂ nanotubes with electrochemical preparation of a thin and conformal solid electrolyte separator based on aromatic polymers with high mechanical resistance and low solubility.

We report the achievement of the electrochemical deposition of a polymer electrolyte layer from various substituted sulfonated phenols. The precursors were synthesized by reaction of phenol with sulfuric acid under conditions corresponding to thermodynamic or kinetic control. The precursor was then electrochemically polymerized in various solvents by applying potentiostatic or galvanostatic conditions. EIS was used to calculate the conductivity of the polymer using equivalent circuit model fitting of the electrochemical cell. Analysis by FTIR and NMR spectroscopy finally confirmed the growth of a thin layer of a sulfonated polymer electrolyte onto the electrode surface.

130 Superelastic Ni-free alloys for biomedical applications processed by selective laser melting

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Combining the outstanding biomechanical and biochemical characteristics of Ni-free Ti-based superelastic alloys with manufacturing flexibility of the selective laser melting (SLM) makes it possible to create patient-specific load-bearing implants with extended service life. These Ni-free materials belong to the group of metastable beta-type titanium alloys, which closely mimic the plateau-like bone behavior by triggering, under stress, beta to alpha'' reversible martensitic transformation. Ti-(Nb, Zr) alloys of different compositions potentially leading to the highest recoverable strain were selected and produced in small quantities (50g-ingots) using vacuum arc- and vacuum-induction melting processes. The obtained ingots were subjected to thermomechanical treatments involving moderate cold rolling followed by post-deformation annealing at different temperatures. They were then characterized from the microstructural and macroscopic points of view to clarify the structure-properties interrelations and to select an alloy exhibiting the best combination of mechanical characteristics (low Young's modulus,

largest superelastic strain, low transformation and high dislocation yield stresses, high ductility). Finally, two 10kg-ingots of the selected alloy were skull-melted and atomized. The obtained powder feedstock was processed using a semi-industrial powder-bed SLM system. To determine the technologically-sound processing window (laser power, scanning speed and spot size), analytical model of semi-infinite solid with moving Gaussian heat source was used. Based on the results of thermomechanical processing of the precursor bulk material, the SLM-built specimens were subjected to the stress-relief and normalizing heat treatments intended to promote in the material stress-induced reversible martensitic transformation resulting in the superelastic behavior of the material at human body temperature.

131 Interfaces in metal-supported electrochemical energy converters

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Metal-Supported Solid Oxide Fuel Cells (MSCs) have long been explored as a promising 3rd generation solid oxide fuel cell (SOFC) technology, which allows the entry of ceramic cell technology to non-stationary applications and improve mass manufacturing. After years of research progress, MSCs have been demonstrated to be a valid technology. However, long-term stability remains a critical issue. Recently a Christian Doppler Laboratory has been established at Forschungszentrum Jülich GmbH in close cooperation with Technical University Vienna, Plansee SE and AVL List GmbH. Among those topics explored are the development of optimized electrode materials and microstructures that exhibit higher tolerance to sulfur contamination, allow for a simplified manufacturing route and show increased electrochemical performance. The dependence of the electrode performance to layer thickness and composition was identified and quantified with numerical calculations and electrochemical experiments. Furthermore, the protection of the metal substrate requires advanced oxidation protection and interdiffusion barrier layers for maximum lifetime of the cell. The challenges coupled with this issue are introduced in general.

132 Environmental protection of γ -TiAl alloys by coatings

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Gamma-TiAl based alloys are attractive light-weight materials for high temperature applications in automotive and aero engines. However, their oxidation resistance is poor at temperatures above 800°C. To improve the oxidation behaviour of TiAl components, the use of protective coatings is a suitable method. Furthermore, the application of thermal barrier coatings (TBCs) allows enhancing the operating temperatures of internally cooled components. In the present study, Ti-Al-Cr based coatings and TBCs of yttria partially stabilised zirconia (YSZ) were deposited on gamma-TiAl alloys using magnetron sputtering and electron-beam physical vapour deposition, respectively. The oxidation behaviour of the protective layers and the lifetime of the TBC systems were determined in the temperature range between 900 and 1000°C performing thermal cycling tests in laboratory air. Intermetallic Ti-Al-Cr based coatings with additions of yttrium and zirconium possessed high oxidation resistance at temperatures up to 1000°C. A continuous alumina scale formed on top of these coatings

stabilised by the $\text{Ti}(\text{Cr},\text{Al})_2$ Laves phase and the Z-phase ($\text{Ti}_5\text{Al}_3\text{O}_2$) evolved in their microstructure. During long-term exposure at elevated temperatures, the coatings depleted in chromium diffusing into the substrate and degraded due to dissolution of the Laves phase and decomposition of the Z-phase. Thermal barrier coatings were successfully applied on the bond coated titanium aluminides. The YSZ topcoat exhibited good adherence to the thermally grown oxide scales. Lifetimes exceeding 1000 cycles of 1h dwell time at high temperatures up to 1000°C were determined, depending upon the oxidation protection capability of the Ti-Al-Cr based bond coats used.

133 Microstructure tailoring by selective laser melting pulse optimization

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The concept that microstructures yielding improved properties can be obtained with AM technologies was demonstrated in few examples. However, the capability to tailor these properties is limited by the numerous parametric constraints present in the commercial SLM unit. This presentation will describe how a pulse-based SLM unit can be operated to fully capitalize on adjusting the microstructure to selectively minimize grain size, texture, residual stresses, etc. The case studies to be presented were based on Al alloys and stainless steels.

134 In-situ neutron diffraction measurements during loading and annealing of additively manufactured materials

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Additive manufacturing (AM) is a manufacturing philosophy where parts and components are built to near net shape by consolidating powders, contrary to the traditional removal techniques based on machining from wrought blanks. Several techniques for AM have been developed over the last few decades. Common for all the AM techniques is that the achieved microstructure within the final product is significantly different than that of the traditional wrought materials, which have been investigated and optimized for centuries. Hence it is paramount to understand how the unique microstructure relates to the observed mechanical properties of the AM materials in order to certify their use in critical applications.

In the present work, we will present results of in-situ neutron diffraction measurements of AM built 304L stainless steel and U6Nb, so-called “stainless uranium” in which the observed macroscopic mechanical response is related directly to microstructural observations gleaned from the diffraction. The neutron diffraction measurements were performed using the SMARTS instrument at Los Alamos National Laboratory. In the stainless steel, it is apparent that the ferrite fraction and initial dislocation density are higher in the AM material than in analogous material manufactured with traditional wrought processing and this is related to the macroscopic response. Likewise, the initial microstructure of the AM U6Nb material is distinct from traditionally processed material, as is the subsequent deformation. The measured data is

directly applicable as verification of modeling of both the build process as well as the constitutive behavior of the as-built material.

135 Magnetic hardening of iron and FeCo alloys via severe plastic deformation and bulk metal forming

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The relevance of permanent magnets in technology of today's society is continuously rising, due to the steady growth in demand of wind turbines and electric vehicles. Since high performance permanent magnets typically rely on rare earth elements with limited accessibility, there is a trend to develop and exploit alternative approaches for magnetic hardening aside from alloying. One approach is to tailor the microstructure of magnetic materials using metal forming processes or Severe Plastic Deformation (SPD) which allow the variation of several parameters that affect the magnetic properties of the material such as grain size, aspect ratio, dislocation density or crystallographic texture.

The aim of the present work is to separate and quantify the effects from different microstructural parameters on the coercivity of pure iron and FeCo alloys. Equal Channel Angular Pressing (ECAP) and High Pressure Torsion (HPT) are used to increase the boundary density without inducing a strong anisotropy in terms of grain shape, whereas infeed rotary swaging is used to generate a strong fiber texture and elongated microstructure. The experimental results show the beneficial effect of the total boundary density and crystallographic texture for magnetic hardening as well as a significantly higher susceptibility to those parameters for FeCo alloys compared to pure iron. Furthermore, the effects of boundary density and texture can be combined to reach a high coercivity at a moderate processing effort. This demonstrates the potential of processing strategies that combine SPD and bulk metal forming processes for the production of permanent magnets.

136 Lightweight sandwich structures in innovative vehicle design under crash load cases

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In the department Lightweight and Hybrid Design Methods of the Institute of Vehicle Concepts in Stuttgart in collaboration with 3A Composite Core Materials, a method which allows to realize sandwich structures for automotive structural applications analytically and conceptually, is developed. The development method based on material and component testing and material values would be determined at different loads, for example in pressure and in-plane tests. These values are transmitted into the analytical determination of so called failure mode maps to derive appropriate sandwich structures. With novel sandwich structures the objectives of high structural stiffness and strength are tracked, as well as a high level of energy absorption potential. By function integrating the potential of lightweight construction, depending on the energy absorption per structural weight, can be further increased. Accompanying tests on generic structures are made to validate the failure behavior. Also the influence of core material

on the deformation behavior is examined. The results from the tests are transferred to a vehicle front structure of a planned lightweight vehicle of class L7E called "Safe Light Regional Vehicle" (SLRV). The behavior of the structure is examined in static and dynamic tests. The energy absorbing capacity can be further increased by geometric optimization and the use of different core materials. The research on sandwich materials is part of the research project Next Generation Car (NGC) of the DLR and represents in terms of the new vehicle concept SLRV in sandwich design a novel vehicle concept of this joint project.

137 Properties of stainless steel 316L alloys processed by selective laser melting: A numerical and experimental study

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As a rather new technology, metal layer based Additive Manufacturing (AM) is still a process to be improved and fully understood. The knowledge on how process parameters are influencing the part mechanical properties represents one of the paramount issues to be investigated. Indeed few data is available on how to relate these parameters to the microstructure and the final properties. In the present work, a combined computational and experimental investigation is presented on Selective laser melting of 316L stainless steel alloy. Both computational and experimental tests are performed using common sets of parameters. The numerical results are obtained from a 3D nonlinear Finite Element Model (FEM) able to predict the thermal field by solving the heat conduction equation through the part processing. The experimental activity involves collection of data from a Selective Laser Melting (SLM) device on simple samples that had been modeled. The variables are the laser power, trajectory and velocity. Simple geometries are used in order to limit the building complexity to a minimum. The melt pool geometry and the in-process temperature obtained from experiments and simulations are then compared for validation and deeper analysis. The objectives of the investigation can be listed as follows: first model validation against experimental data, then correlation of the simulated temperature fields and thermal gradients with the microstructure of the built part. The conclusions obtained allows to draw preliminary recommendations on how to set the studied parameters in order to optimize the part microstructure and thus its mechanical properties.

138 Phase constitution and martensitic transformation behavior of Au-51Ti-18Co biomedical shape memory alloy heat-treated at 1173K to 1373K

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In the present study, phase constitution and martensitic transformation were investigated for a Au-51Ti-18Co alloy heat treated at 1173K to 1373K for 3.6ks. The Au-51Ti-18Co alloy was fabricated by Ar arc-melting technique and subsequently by hot-forging at 1423K for

10.8ks. X-ray diffraction analysis revealed that B2 parent phase, B19 martensite phase and $L1_2$ (Au, Co) Ti_3 simultaneously appeared regardless of the heat treatment temperatures. By increasing the heat treatment temperature, the volume fraction of (Au, Co) Ti_3 was slightly reduced and the B2 parent phase was stabilized. Besides, the lattice transformation strain which was calculated from the precise lattice parameters was evaluated to be 7% in the Au-51Ti-18Co alloy in all the heat treated conditions. This value is comparable to that of NiTi practical alloys. From differential scanning calorimetry (DSC) analysis, reverse martensitic transformation temperature was slightly increased with the heat treatment temperature. From the lattice transformation strain point of views, the Au-51Ti-18Co has a large potential for novel biomedical shape memory alloy. This work was supported by Grant-in-Aid for Scientific Research Kiban S 26220907 and Challenging Exploratory Research 26630343 from Japan Society for the Promotion of Science (JSPS).

139 Simultaneous precipitation and recrystallization during hot deformation of Ti, Nb and V micro-alloyed steel

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Recrystallization is a major means for controlling the grain size of steel during hot deformation. Usually, small grain sizes deliver superior mechanical properties. To aid the grain size controlling effect of recrystallization, small precipitates of carbo-nitride particles can be utilized to hinder the movement of grain boundaries. Interestingly, these particles are not only effective during grain growth, but also during recrystallization. In the present talk, a recently developed state-parameter based model is introduced that is capable of describing both, the individual processes of static recrystallization, dynamic and static recovery and precipitation as well as the mutual interaction of these mechanisms in the course of elevated temperature processing. The evolution of state parameters within the model is discussed and the simulation results are compared to experimental information. Within our approach, a vast amount of experimental data for microalloyed steel is reproduced on basis of a single set of input parameters.

140 Recrystallization after cold plane strain compression in a commercial AA6082

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The production of aluminium plates generally comprises a succession of cold rolling passes followed by a solution heat treatment during which the microstructure recrystallizes. The modelling of the recrystallization kinetics requires the knowledge of the mobility of high angle grain boundaries and data to estimate the initial quantity of nuclei. An initially hot rolled Aluminum Alloy 6082 is cold deformed to a nominal strain of 1 by means of plane strain compression. Subsequent recrystallization treatments are carried out at 300°C and 400°C. EBSD measurements are used to determine the evolution of the recrystallization grade and the recrystallized grain size. Because the deformation is heterogeneous, the microstructure after heat treatment depends on the initial local strain, calculated by means of finite element

modelling. The stored energy of deformation is calculated with a previously designed flow stress model featuring two kinds of dislocation densities. The recrystallization kinetic is then modelled using the approach of Zurob et al-where the initial quantity of nuclei is calculated as a function of strain and holding temperature. The mobility of high angle grain boundaries is adjusted to represent both the recrystallization behavior and the subsequent grain growth observed experimentally.

141 Microstructure and mechanical properties of dissimilar FSW joints between HSLA and austenitic high Mn TRIP steels

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The aim of this study is to evaluate the microstructure and mechanical properties of dissimilar friction stir welded (FSW) joints between high-strength low alloyed (HSLA) XABO500 and austenitic high Mn TRIP steels with different welding parameters. Tool rotation and offset toward the TRIP steel were varied between 300–500 rpm and 1–2 mm respectively. Tool advancing speed amounted to 100 mm/min. The properties of dissimilar FSW butt-joints were evaluated by tensile tests and microhardness profiles. Optical (OM) and scanning electron microscopy (SEM) were used to investigate the microstructures of the welded joints. Maximum tension stress was observed for the butt-joint welded with 300 rpm and 2 mm offset. The lowest increase in hardness within the stirred zone also occurred for this FSW condition, indicating that this tool rotation is more promising for welding dissimilar joints of HSLA and high Mn TRIP steels. An austenitic microstructure with nearly equiaxial morphology is formed in the TRIP base metal, whereas polygonal ferritic grains along with perlite colonies are found in the HSLA base metal. The perlite colonies in the HSLA steel grade appear aligned along the rolling direction. The weld microstructure consisted mainly of a stirred zone, and no significant HAZ nor TMAZ are observed. However, two main lobular regions are observed, one at the bottom and another one at the top side of the welds. Besides, the HSLA develops a multiphase microstructure consisting of bainite, martensite and retained austenite phases, whereas no alpha/epsilon martensite is found in the stirred zone of the high-Mn austenitic TRIP steel.

142 Complex nano-scale structures for unprecedented properties in steels

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Processing bulk nanocrystalline materials for structural applications still poses a significant challenge, particularly in achieving an industrially viable process. In this context, recent work has proved that complex nano-scale steel structures can be formed by solid reaction at low temperatures. These nanocrystalline martensitic and bainitic steels present the highest strength/toughness combinations ever recorded, unprecedented ductility, fatigue on par with

commercial bearing steels and exceptional rolling-sliding wear performances. A description of the characteristics and significance of these remarkable structures in the context of the atomic mechanism of transformation is provided.

143 Thermal plasticity index of nanostructured N-based coatings on HSS 6-5-2 (1.3343) tool steel

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Nowadays, cutting tools, designed to be used for machining without lubricants, are developed to improve the high working speed capabilities. With this respect, quaternary Ti- and N-based coatings are able to significant increase hardness, wear resistance and high temperature oxidation resistance. One of the major drawbacks still consists on the limited thermal stability of such coatings, which is reported to be about 600°C. In the present study, thermal stability studies of a nanostructured multi-layered N-based ($\text{AlTiCr}_x\text{N}_{1-x}$) coating on a HSS 6-5-2 tool steel were carried out. Two quantities were calculated out of the hardness and elastic modulus of the coatings. One is the ratio H/E that represents the coating resistance to compression without failure; another one is H^3/E^2 , which provides information on the specific contact pressure limit without failure. It was found that, by using the less demanding thermal cycling mode, the coating ability to plastically deform without damage, is retained up to 800-1000°C. The highest, and more effective coating plasticity index was obtained by using the less demanding cycling mode, while, the other two modes induced a continuous index decrease with temperature.

144 Microstructural investigation of oxynitrocarburized components processed at different temperatures

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Ferritic gas nitrocarburizing combined with a post-oxidation treatment is an interesting industry process developed in order to improve wear and corrosion resistance of low alloyed steel components. The nitrocarburized surface has featured microstructures and properties that are directly related to the phase transformations occurring in the surface layers during process. Therefore, the structure and properties of the oxide film formed over the compound zone during the post-oxidation process are strictly related to the surface characteristics of the nitrocarburized layer. In the present work, the microstructure resulting by a thermochemical treatment, which comprises nitriding, nitrocarburizing and post-oxidation stages, applied to an industrial 16MnCr5 component has been studied. In particular, both the nitriding and nitrocarburizing temperatures were systematically changed in order to study the resulting phases in the compound layer. The depth of the compound layer have been measured by optical microscopy to evaluate the effects due to the variations in the process variables during the nitrocarburizing process. Moreover, the microstructure has been investigated by means of a scanning electron microscope equipped with a electron backscatter diffraction detector in order to assess the amount and the distribution of Fe-N-C phases.

145 Nanostructured SPD-processed Ti-based materials for load-bearing orthopedic applications

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Compared to respective micro-sized counterparts most nano-scale structured metallic implant materials have been shown to enhance bone cell responses and functions that include cellular adhesion, proliferation and calcium deposition.

In recent years, extensive investigations were performed to produce novel Ti-based alloys with improved bio-mechanical properties for load-bearing orthopedic implants. The objective of this study is to develop novel nanostructured titanium alloys of beta-type, comprising only non-toxic and non-allergic elements, in an effort to match high strength with low elastic modulus to further reduce the “stress shielding” effect.

In this work, the microstructure evolution, beta-phase stability and mechanical properties have been investigated for a series of beta-type Ti-Nb-based alloys (binary Ti-Nb and multicomponent Ti-Nb-Ta-Zr/In/Cr). These alloys were produced by cold crucible casting and further processed by severe plastic deformation - SPD (high pressure torsion, accumulative roll bonding). Microstructural evolution was studied upon compressive loading and SPD for selected beta-Ti alloys with different phase stability against $\beta \rightarrow \alpha''$ martensitic transformation. From the TEM and HRTEM analysis, it was revealed that with increasing the deformation degree, martensitic transformation and deformation twinning are initially operative followed by slip of dislocations for the less stabilized beta Ti-alloys, whereas slip is initially operative followed by nanoscale deformation twinning for the relatively stabilized alloys. The nanostructuring occurred via SPD processing significantly contributes to the increase of hardness and strength of beta-type alloys but it has a little effect on elastic modulus since the bindings between atoms stay the same, even if defects are present.

146 Optimal deformation hardening in lead base anodes for copper electrowinning for an appropriate working life

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The lead base anodes (Pb-0.07% Ca-1.3% Sn) of 1x1 meters of section and 6 mm thick have limited working life due to their loss of thickness and corrosion in the electrowinning process and due their low yield stress. To achieve the minimum standard mechanical quality of anodes, the necessary precipitation hardening is reached after at least 30 days of ageing, and the needed deformation hardening is obtained with a final cold rolling of the anodes with a 40% of area reduction.

The aim of this study is to optimize the deformation hardening of the anodes, so as to achieve the best combination of yield stress and corrosion resistance to increase their working life. To achieve this the aged anodes were cold rolled to different area reductions up to 75%.

To each one of these rolled anodes its was determined their yield stress, grain size and their corrosion rate by coulombimetric assays using an electrolyte concentration of sulfuric acid of 180 g/l at an oxidation current density of 300 A/m².

It was found that the maximum yield stress achieved by the anodes is 68 MPa, for the total cold reduction of 75%. Regarding the corrosion rate, the maximum and minimum values found were 0.35 mm/ year and 0.31 mm / year, ie with no significant differences between the different rolled anodes. Based on the above results it is concluded that an increase in the working life of the anodes is obtained simply by giving them greater cold rolling deformation up to 75%, of area reduction.

147 Non-viral vectors for gene delivery

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Gene delivery is the transfer of genetic material into recipient cells to alter some of their functions. Unfortunately, the spontaneous entry of naked nucleic acids into cells is very inefficient. Cationic lipids and polymers, known as non-viral delivery systems, have thus made their breakthrough in basic and medical research because capable of spontaneously self-organizing with polyanionic nucleic acids to give nanoparticles. Nevertheless, albeit these are promising alternatives to viral vectors, the inability of these gene carriers to fully satisfy the contrary requirements of adequate nucleic acid protection and efficient release is believed to be one of the major impeding factors behind their generally poor effectiveness and adverse cytotoxic side effects. To elicit the desired outcome, major strides forward have been made in the development of stimuli-responsive gene delivery vectors that actively respond to changes in the (micro)environment (e.g., cell enzymes, redox status and pH) by altering their properties and behavior. On this ground, stimuli-responsive transfectants are considered as the Holy Grail of gene delivery due to their ability to restrict the release of nucleic acids to intracellular compartments.

148 Numerical simulation of the effects of preheating on electron beam additive manufactured Ti-6Al-4V build plate

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Electron beam additive manufacturing processes based on building up net or near net-shaped components in successive layers offer an attractive alternative to the machining of large parts. It is particularly appealing for titanium components that now have a high manufacturing cost and a very low buy-to-fly ratio. However, the rapid heating and cooling cycles involved lead to significant thermal induced issues. In an early study, a 3-D thermomechanical coupled finite element model has been built and experimentally validated to investigate the residual stresses and distortions in electron beam additive manufactured Ti-6Al-4V build plates. A very good agreement between theory and experiment verified that the model captures the essential characteristics of the electron beam additive manufacturing process very well. In this study, an investigation using this robust and accurate model was focused on an efficient preheating method, in which the electron beam quickly scan across the substrate to preheat the build plate prior to the deposition. A preliminary study predicted that the residual stress and distortion decreases with more scanning times across the substrate in the single pass, 11-layer Ti-6Al-4V

build plate. Effects of variables involved in this method were thoroughly simulated to provide guidance in deciding appropriate preheating strategies. Various input energies, scan rates, scanning paths and cooling times were examined, and the maximum distortion along the centerline of the substrate and the maximum longitudinal residual stress along the normal direction on the middle cross-section of the build plate were quantitatively compared.

149 Development of Al-Mg-Si-(Cu) alloys for automotive body panels and the related ageing behaviours

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The increasing use of Al alloys in automotive body panel applications has attracted intensive investigation on the alloy development and property / microstructure characterisation in Al-Mg-Si-(Cu) alloys. In this work, our development of Al-Mg-Si-(Cu) alloys for automotive body panels will be overviewed and the related ageing behaviours will be discussed in details to help understand natural ageing and pre-ageing, as well as their influence on the subsequent paint-bake response. The clustering behavior of these Al-Mg-Si-Cu alloys in different ageing conditions was investigated by hardness / yield strength and electrical conductivity testing, along with nanostructural characterisation using 3-D atom probe (3DAP) analysis. The results show that the initial hardening behaviour of the alloy is influenced significantly by the degree of clustering of Mg, Si and Cu solute atoms. The paint bake response is greatest in samples subjected to short natural ageing times, and decreases very rapidly within the initial 24h of natural ageing and then continues to decrease to less than zero at longer natural ageing times of 7 days or more. An effective pre-ageing treatment can contribute to an enhanced age hardening response. The different paint bake response will be discussed in terms of the presence of b'' precipitates, various distributions of clusters and/or GP zones.

150 Laser deposition repair of AA7075 alloy components using Al-12Si powder

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Due to long lead-times for the manufacture of new components, there is a strong driving force to investigate the feasibility of repairing service-damaged components. In the aerospace industry, conventional repair techniques for metal components utilize arc welding processes. However, the high heat input can cause significant degradation in the microstructures and associated mechanical properties, as well as impart considerable residual stresses and distortion. In contrast, laser-base additive repair is a promising emerging technology due to (i) low and controllable heat input that produces a small heat-affected-zone, low residual stresses/distortion, and high dimensional accuracy; (ii) high energy density that leads to high deposition rates and productivity; (iii) good flexibility and reliability due to fiber optic delivery, non-contact, atmospheric pressure operation, easy automation, and excellent equipment reliability; and (iv) onsite repair possibilities. However, the laser additive repair technology, particularly for the AA7075 components, is especially challenging due to its solidification crack sensitivity. In this work, the feasibility of laser repairing AA7075 using Al-12Si alloy powder

was investigated through process development, characterization of the defects and microstructure, and evaluation of the mechanical properties (hardness and ring tensile testing).

151 Preparation and characterization of porous magnesium for scaffold fabrication

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Porous magnesium based materials have been intensively studied as possible candidates for temporary scaffolds. It is because of their biocompatibility, biodegradability, good mechanical properties and interconnected porosity, which can improve biocompatibility of these materials. Therefore, porous magnesium materials seem to be suitable for fabrication of biodegradable scaffolds. In this work, we prepared porous magnesium samples (porosity 24-31 vol.%) by powder metallurgy using ammonium bicarbonate as a spacer material. The influence of sintering conditions (sintering time and purity of argon atmosphere) on microstructure and mechanical behaviour was investigated. We found that after sintering for longer than 6 h, the purity of argon atmosphere plays a significant role in the material characteristics. Under argon of technical purity, with increasing sintering time porosity slightly increased and mechanical characteristics significantly decreased. This was attributed to extended oxidation on powder surfaces. Using the gettered argon had an opposite effect because of the oxidation prevention, which caused better diffusion connections between magnesium particles. Sintered samples possessed significantly coarser grains compared with the initial magnesium powder; although, the grain size was approximately the same for all sintered samples independently on the sintering conditions. We would like to thank to the Czech Science Foundation (project no. P108/12/G043) for the financial support.

152 Investigation on sPEEK-porphyrin interaction for polymer electrolyte membranes portable applications

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The polyaromatic membranes can be considered as an alternative to PFSA based membranes, for portable fuel cell applications. In this context, the sulphonated polyetheretherketone (sPEEK) due to its properties such as high thermo-oxidative stability, high solvent resistance, excellent mechanical properties, proton conductivity comparable to Nafion depending on the sulphonation degree and low cost (about 1/40 if compared to Nafion) is a good candidate. In order to guarantee a good proton conduction in operative conditions useful for portable applications (room temperature and low pressure) a sulphonation degree of about 65% was selected. To improve the characteristics of membranes based on bare sulphonated polymer, composite membranes based on sPEEK and porphyrins with different substituents in the periphery of the macrocycle were studied. A screening of different weight percentages (in the range 0-5) was carried out in order to identify the best composition as a function of the interactions between porphyrins and polymeric matrix.

With the aim to better understand the influence of the aggregation state of porphyrins embedded

into polymeric matrix, physico-chemical characterizations such as UV-Vis and Fluorescence emission, ion exchange capacity, water retention, dimensional variations and swelling, structural and morphological analyses were carried out.

Moreover, proton conductivity measurements at low temperatures were performed to understand the influence of the interactions on the proton transport mechanism. Finally, electrochemical tests in a PEFC fed by hydrogen and air were performed on the developed membranes to verify the performance in the selected operative conditions.

153 Hot deformation behavior and stability criteria of magnesium alloy WE54

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A precise description of the hot deformation behavior as well as determination of the stability conditions as influenced by temperature and strain rate is fundamental for the simulation of metal forming processes. In this work, a revision of various stability criteria of magnesium alloy WE54 is conducted. The study corresponds to own work and that of Lentz et al. and is based on compression tests at high temperature and high strain rates. Stability and processing maps were obtained using a variety of stability criteria. These criteria are all based on determination of the strain rate sensitivity exponent, m . This parameter is usually determined by fitting the curves strain rate, $\dot{\epsilon}$, versus stress, σ , by means of a potential equation named "power law" or by a polynomial of second or third degree, and calculating the slope of the logarithmic curve at each point using successive derivatives. This procedure is compared with one developed by us where all experimental points are fitted to a single hyperbolic sine equation of Garofalo type and then m is calculated for each $\dot{\epsilon}$ and T using this equation. The maps obtained by one or the other method differ considerably. The predictions of these maps were contrasted with microstructural observations and conclusions on the deformation behavior of the alloy are reached.

154 Coupling experiments and models to interpret degradation in polymer electrolyte fuel cell

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Degradation is one of the most critical issues limiting polymer electrolyte fuel cells commercialization. Several degradation mechanisms have been observed, occurring simultaneously and not uniformly in the different components. Such complexity makes difficult both the characterization of degradation mechanisms and the development of a reliable modeling tool able to predict fuel cell degradation in different operating conditions. Thus we developed a combined experimental and modeling approach to interpret and predict degradation at cell level, based on the two-way interaction between: - a dedicated experimental analysis designed to gather the necessary information for models validation, including diagnostics measurements during the whole degradation test and specific tests to highlight the evolution of the investigated phenomena; - physically based models including all the relevant phenomena regulating fuel cell operation, keeping a low computational cost, in order to permit extensive

validation on different types of measurement over a wide range of operating conditions. This methodology has been applied to polymer electrolyte fuel cell technology: the investigation has highlighted a severe impact on performance of cathode degradation mechanisms, reproducing such effects required the modeling of different aspects among which heterogeneity of ageing and mass transport limitations, as observed in polarization curves and impedance spectra; investigation of DMFC has led to the distinction of temporary degradation at both anode and cathode, respectively associated to mass transport limitation and platinum oxides formation, and permanent degradation, mainly caused by a loss of cathode catalyst active surface, which effect on performance is also strongly influenced by cathode flooding.

155 Measurement of stress field in deformed material at the micron scale: Combining Laue microdiffraction with digital image correlation, and related accuracy

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A better understanding of the effective mechanical behavior of polycrystalline materials requires an accurate knowledge of the behavior at a scale smaller than the grain size. The X-ray Laue microdiffraction technique available at beamline BM32 at the European Synchrotron Radiation Facility is ideally suited for probing elastic strains (and associated stresses) in deformed polycrystalline materials with a spatial resolution smaller than a micrometer. However, the standard technique used to evaluate local stresses from the distortion of Laue patterns can lack accuracy for many micromechanical applications, mostly due to (i) the fitting of Laue spots by analytical functions, and (ii) the necessary comparison of the measured pattern with the theoretical one from an unstrained reference specimen. A new method for the analysis of Laue images will be presented. A Digital Image Correlation (DIC) technique, which is essentially insensitive to the shape of Laue spots, is applied to measure the relative distortion of Laue patterns acquired at two different positions on the specimen. The accuracy of this new method has been studied using synthetic Laue images generated numerically accounting for a random image noise model that has been fitted to real images. The new method has also been tested on an in situ deformed Si single-crystal, for which the prescribed stress distribution has been calculated by finite-element analysis. It is shown that the new Laue-DIC method allows determination of local stresses with a strain resolution of the order of 10^{-5} .

156 The effect of fast annealing on the strength and microstructure of CMnAlSi TRIP steel

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Fast annealing experiments have been carried out on 50 and 75% cold rolled Fe-0.2C-1.6Mn-1Al-0.5Si steel with ferrite and pearlite microstructure. The thermal path consist of four steps (i) slow heating (10 °C/s) to 400 °C; (ii), holding at 400 °C for 30s, (iii) fast heating to 860 °C and hold for 1.5 s, and (iv) quench to room temperature. The heating rates on the third stage

were 10 and 400 °C/s. For mechanical tests, 3 sub-size tensile test samples were cut from each specimen parallel to the rolling direction. The gage length and the width of the tensile test samples are 3x1 mm², respectively. The microstructure evolution was followed by Optical and Scanning Electron Microscopy and Electron backscatter diffraction performed on FEI Quanta 450-FEG-SEM. Results have shown microstructure refinement and increase of the strength when the fast annealing treatment is applied to the samples with higher amount of cold reduction. Tensile test showed an increment of ~250 MPa in the UTS with the rise in the heating rate. Microstructural characterization revealed that the improvement in the strength is mainly due to the martensite fraction and the average ferritic grain size, both being noticeably affected by the heating rate. Orientation measurements showed that the cold rolling texture remains unchanged after the first and second stage of the thermal cycle. In addition, there is a transition in the overall orientation components from ND to RD fiber as the heating rate rises. Such transition is influenced by the specific nature of recrystallization process triggered by fast heating experiments.

157 Crystallization behavior of cold sprayed pure Ni coatings

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Cold spraying is a coating technology on the basis of aerodynamics and high-speed impact dynamics. In this process, spray particles (usually 1-50 µm in diameter) are accelerated to a high velocity (typically 300-1200 m/s) by a high-speed gas (pre-heated air, nitrogen, or helium) flow that is generated through a convergent-divergent de Laval type nozzle. A coating is formed through the intensive plastic deformation of particles impacting on a substrate at a temperature well below the melting point of the spray material. In the present paper the main processing parameters affecting the crystallization behavior of pure Ni cold spray deposits on IN718 alloy are described. Many experimental conditions have been analyzed in terms of particles dimensions, substrate temperature, gas temperature and pressure, nozzle properties. In the present paper, the effect and the relative weight of each processing parameters on the microstructural and mechanical behavior of cold spray deposits have been analyzed. In particular, they were studied those conditions leading to a strong grain refinement with an acceptable level of the deposits mechanical properties such as porosity, adhesion strength and hardness. The shift toward amorphous phases have been studied. A systematic analyses of microstructural evolution, performed through TEM observations, as a function of processing parameters is presented. Keywords: Cold spray; processing parameters; Ni; microstructural behavior, mechanical properties.

158 Fuel cell electrodes based on electrospun mats

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Despite vast improvements in the stability of Proton Exchange Membrane Fuel Cells (PEMFC) materials, further increase in electrode durability is still required. Effective utilisation of platinum electrocatalysts and the development of electrochemically stable supports are the core of our research.

Our approach is based on the use of the electrospinning technique to prepare nanofibre based electrodes with specific architectures and properties [1]. The nanometre size and one-dimensional morphology of the fibres, and the porous structure of the electrospun mats, are expected to bring associated advanced properties, in particular with regard to directional and mechanical properties, and mass transport, with beneficial effects on the performance and lifetime of the resulting electrodes. We are developing nanofibrous electrocatalyst supports with extended durability and high conductivity: among them carbon fibres, metal fibres, metal carbide and oxide nanofibres and nanotubes including Nb- TiO₂, Nb-SnO₂, Sb-SnO₂. The latter represent a very attractive alternative to conventional supports because of their chemical and electrochemical stability and promoting effects, leading to improvements on the catalytic activity and stability of PEMFC electrodes [2]. In parallel, we are developing Pt deposition techniques leading to extended metal surfaces onto the electrospun materials, including Ni and Cu galvanostatic displacement [3], self-terminated Pt electrodeposition and electrochemical atomic layer deposition. This association of deposition techniques opens up a broad range of opportunities for tuning materials composition and architecture.

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159 Ultrasound assisted hydrometallurgical process for Gold recovery from PCBs using thiosulphate as complexing agent

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Waste electrical and electronic equipment (WEEE) is the fastest growing advanced type of solid waste streams in the urban environment worldwide and contains interesting amounts of precious metals. In this work, the use of thiosulphate as a cyanides substitute in the recovery of gold from PCBs was studied. In fact, nowadays the aim of recycling is focused on the recovery of these metals from WEEE and hydrometallurgical technique seems to be very promising due to its lower energy consume and lower smelter emissions than conventional pyrometallurgical processes. Thiosulphate has been studied because it's a compound able to leach gold whilst being much less toxic than the cyanides typically used for this application. The treated material was the metals enriched fraction of the powder of circuit boards, obtained by mechanical preparation of out of use cell phones PCBs, after a pre-treatment with acid before the thiosulphate leaching. Among the different parameters, ultrasounds in particular has been investigated to assist the hydrometallurgical gold extraction, as their application in ores leaching showed a greater metals release in shorter time and the advantage of working at lower concentration of reagents and at lower temperature. In fact, in this study the use of ultrasound allowed a higher recovery of the precious metals than leaching without ultrasounds in all the conditions studied (different concentrations of reagents, temperature and etching time). Moreover, the studied process allowed also the recovery of the other metals present in the waste (Cu, Sn and Ag).

160 Fracture surface characterisation of friction stir processed magnesium alloy after mechanical tests

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A high pressure die cast Magnesium-9%Al-1%Zn alloy was friction stir processed at two high rotation rates and advancing speeds. Tensile tests were performed at room and at higher temperature to study the mechanical properties of the microstructure induced by the friction stir process. Fracture surfaces resulting from tensile tests, were observed by scanning electron microscopy and investigated by microanalysis. The fracture occurred in the tma_z and inside the stirring zone, depending on deformation conditions (temperature and strain rate). The morphology of the fracture surface varied from ductile to brittle in the same sample depending on phase type. Microhardness was measured on cross sections perpendicular to the advancing direction of the stirring pin, at three depth levels, before performing tensile tests to estimate the attitude of a single region to be deformed.

161 The interface structure between CIGS and Mo films

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The adhesion behavior between CIGS thin film and the back contact Mo layer is one of the critical factors for CIGS solar cell efficiency. To investigate the CIGS/Mo interface structure, the characterization methods of X-ray diffraction, scanning electron microscopy and transmission electron microscopy were employed. It was found that a bilayered structure formed between CIGS and Mo films. The first layer adjacent to Mo is smooth and thinner than 100 nm, while the second layer close to CIGS is rough and thicker than 500 nm. Scanning transmission electron microscopy experiment showed that the thinner layer is Se rich, and the rough layer is mixed by Na-rich second phase and some Cu-rich CIGS grains. Combining all the data, the formation mechanism of the layered structure at CIGS/Mo interface was discussed, as well as the effect of Na atoms from soda lime substrate.

162 Influence of long term ageing on deformation and fracture behaviors of Alloy 617

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Alloy 617 as a super alloy has been used in advanced high efficient power plants. The long term mechanical behaviors of this material at high temperatures are therefore concerned. In this study, Alloy 617 material has been aged at 650°C and 700°C for up to 30 000 hours. The precipitates in the aged material have been studied using electron microscopy and then compared with thermodynamic calculation. M₂₃C₆ and γ' phases are two main precipitates. They are both inter- and intra-granular particles. The size increases with increasing aging time. The influences of ageing on hardness, impact toughness and fracture toughness have been investigated. As expected the hardness increase with ageing time, but

increase relatively small after 3000 hours. However, Alloy 617 can still shows very high impact toughness and fracture toughness even with an ageing at high temperature for 30 000 hours. The mechanisms have been studied using electron backscatter detection and electron channeling contrast imaging. The microstructure investigation shows that twinning is one of main deformation mechanisms in these aged Alloy 617 material. At the crack front, plenty of micro or nano twins can be observed. The formation of these twins lead to a high toughness, i. e. twin induced toughening. This is a new observation or a new concept. The influence of strain rate on the twinning behavior has also been studied. Twinning in this material depends on the precipitates, hardness and strain rates.

163 Linking microstructural evolution and friction in metals

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The tribology community presently relies on phenomenological models to describe the various seemingly disjointed steady-state regimes of metal wear. Pure metals such as gold -- frequently used in electrical contacts -- exhibit high friction and wear. In contrast, nanocrystalline metals, such as hard gold, often show much lower friction and correspondingly low wear. The engineering community has generally used a phenomenological connection between hardness and friction/wear to explain this macroscale response, and thus to guide designs. We present a suite of recent simulations and experiments that demonstrate a general framework for connecting materials properties (i.e. microstructural evolution) to tribological response. We present evidence that the competition between grain refinement (from cold working), grain coarsening (from stress-induced grain growth), and wear (delamination and plowing) can be used to describe transient and steady state tribological behavior of metals, alloys and composites. We will present the results of large-scale molecular dynamics simulations and targeted experiments that explore the seemingly disjointed steady-state wear regimes of metals and alloys, with a goal of elucidating the structure-property relationships, allowing for the engineering of tribological materials and contacts based on the kinetics of grain boundary motion. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

164 Optical and mechanical properties of Al-based amorphous/nanocomposite films with and without thermal treatment

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The aluminum-based metallic glasses as well as aluminum-based nanocomposite films consisting of Al nanoparticles embedded in the glassy matrix, prepared by magnetron sputtering on silicon and glass substrates, exhibit high reflectivity properties. The relationship between

composition, atomic structure and reflectivity performance is established. Metallic glasses can form smooth surface without the influence of crystalline facets and grain boundaries, hence have lower surface roughness. In addition, the initial transformation reaction upon devitrification during thermal treatment for the aluminum-rich compositions in the aluminum-based metallic glass is a primary crystallization of the base component that yields a nanophase composite. This composite structure can increase largely the tensile strengths of the aluminum films. The above advantages in terms of optical and mechanical aspects would facilitate the optical applications of aluminum-based metallic glasses/composite structures. Compared with pure Al films, the Al-Ni-Y films have much lower surface roughness and much higher hardness, suitable for optical reflection applications. For composite Al-Ni-Y films, the reflectance varies within 80-95% for different wavelengths. The reflectance at 800 nm is even better than the Al films deposited in the sputtering system.

165 Improvement of mechanical and wear characteristics at the welded joint of rail by ultrasonic nanocrystal surface modification

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Welding is an inevitable process to join two rails, but the weakening of wear resistance and fatigue strength at welded zone is a remaining problem which should be solved. Furthermore, noise problem arisen by chattering at welded zone caused by severer wear rate than common rail is becoming a more serious problem at urban operation. The ultrasonic nanocrystal surface modification (UNSM) technology which could increase not only wear resistance but fatigue strength is applied to the welded specimens of two rails. Depth profile of hardness, wear rate and friction coefficient at welded material, heat affected zone and common rail before and after UNSM are compared and analyzed. Microstructural and mechanical analysis will be added to explain why wear resistance after UNSM is increased. Two kinds of welded specimens made by thermite welding and gas forged welding are prepared for this study.

166 Application of physical modelling for fine grain structure formation in Nb-microalloyed pipe steels during controlled rolling

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In this research an effect of structure formation during slabs reheating was studied. Physical models of secondary recrystallization during reheating and austenite recrystallization at roughing rolling of Nb-microalloyed pipeline steels were developed. Strategies of roughing rolling were created by the physical modelling. The strategies were compared in terms of forming a fine and uniform austenite structure before finishing rolling. The strategies have been applied on rolling mill 5000 at Vyksa Steel Works for difficult pipeline projects as the South Stream and the Bovanenkovo-Ukhta. Shown a relation of roughing rolling strategies application

with increasing of toughness and DWTT results of industrial plates for pipes.

167 Effect of the cooling rate on phase transformation kinetics of the Ti-5Al-5Mo-5V-3Cr-1Zr alloy using in-situ synchrotron high-energy X-ray diffraction

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The phase transformation kinetics of Ti-5Al-5Mo-5V-3Cr-1Zr alloy, in the as-cast condition was investigated by means of in-situ high-energy synchrotron X-ray diffraction (HXRDXRD) at the ESRF and complemented by microstructural analysis using SEM. Analysis at room temperature in the Ti-55531AR alloy in the as-cast condition showed an alpha globular + beta phase microstructure. The phase evolution was studied on cooling with rate of the 20°C/min and 5°C/min from the beta field at 900°C up to 225°C. During cooling at 20°C/min, a qualitative analysis of the evolution of the (hkl) reflections from 2theta vs Temperature plots permitted to observe a possible decomposition of the beta phase into the omega phase around 650°C, by the apparent presence of the (200) beta and (201) omega reflections. This transformation is accompanied by a decreasing in the intensity of the reflection (110) beta. The broadening of the (211) beta peak around 500°C up to 225°C, may be due to the presence of the (103) alpha, (102) and (300) omega reflections. During cooling at 5°C/min, the early beta to alpha transformation was observed around 750°C by the presence of the (100) and (101) reflections. In the subsequent cooling, around 550 °C, (102), (110) and (103) reflections of the alpha phase appear. Thus, it is clear that the beta-alpha transformation is favoured during slower cooling.

168 Electroless plating of copper on TaN barrier layers using seed-anchoring self-assembled monolayer

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It is well known that traditional sensitization-activation or displacement-activation catalyst process tends to form agglomerated cluster of sizes typically tens to hundreds of nm. These seeding processes thus fail to be used for the electroless plating of copper metallization with uniform layer thickness of tens of nm for the next generation integrated circuits. The purpose of this work is to develop a new seeding process that suits the Cu electroless metallization in the future. In this study, the ammonia-based alkaline solutions containing various concentrations of hydrogen peroxide (H₂O₂), as an oxidizer, were used to convert a tantalum nitride (TaN) barrier layer on porous low-k dielectric films (k = 2.35), into a hydroxyl-terminated surface. The water-wetted surface allowed the uniform growth of an octadecyltrichlorosilane self-assembled monolayer (OTS-SAM), which acted as a seed-anchoring layer for the binding of metallic (e.g., nickel) particles as catalysts of electroless plating of copper. Then, a sequence of processing steps, including a dual (N₂/H₂ vacuum plasma and chemical-solution) surface modification of the OTS-SAM and seed-anchoring treatment, were performed to immobilize Ni particles on the topmost aliphatic chains of SAM.

Transmission electron microscopy showed that the size of the metallic particles anchored was typically 3 nm, and thus were able to trigger the electroless plating of Cu of only tens of nm. We are currently using X-ray photoelectron spectroscopy (XPS) to characterize the surface-bonding alternation mechanism of the TaN barrier layers under the treatments of ammonia-based alkaline solutions with various hydrogen peroxide concentrations.

169 An investigation of recrystallization behavior of a high-manganese transformation-induced plasticity (TRIP) steel

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The microstructure of high-manganese steel with low stacking fault energy (SFE) is mainly composed of austenite and ϵ -martensite at room temperature. The mechanical properties of high-Mn steel can not only be improved by transformation induced plasticity (TRIP) effect of hexagonal close-packed ϵ -martensite, but possess high damping capacity as well. These steels are increasingly found applications in the fields of automobile, machinery and construction as a new generation of low cost materials. In this presentation, recrystallization behavior of a high-Mn TRIP steel (Fe-17Mn-0.05C) have been studied by compressive deformation test at elevated temperatures to explore the effect of deformation temperature, strain rate and deformation amount. The results show that the main softening mechanism of this steel during elevated temperature deformation changes from dynamic recovery to dynamic recrystallization with increasing temperature and decreasing strain rate. Moreover, static recrystallization is readily to take place at large amount of deformation and high strain rate. Activation energies for dynamic and static recrystallization were calculated to be 415 kJ/mol and 257 kJ/mol, respectively. When quenched after hot deformation, a little α' -martensite could be generated on some ϵ martensite plates within unrecrystallized austenite in the deformed austenite owing to a higher density of defect. Finally, the hot working equation and kinetic equation of static recrystallization were established on the basis of compressive flow stress curves.

170 The unexpected role of benzotriazole in mitigating magnesium alloy corrosion: A nucleating agent for crystalline nanostructured magnesium hydroxide film

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Benzotriazole (BTA), an effective corrosion inhibitor for Cu or Cu-containing Al alloys, was added to 0.1 M NaCl to study its potential role in suppressing corrosion of the commercial Mg alloy, AMLite. In particular, the impact of pH and BTA concentration was investigated, indicating that BTA was effective at restricting corrosion of AMLite in weakly alkaline NaCl solution, i.e. pH 10.0. The degree of the protection afforded was a function of BTA concentration. The best corrosion inhibition was found in the NaCl containing 15 g/L BTA, however, the mechanism of inhibition was not a classical function, nor related to the inhibition mechanism of BTA for Cu-bearing alloys. Instead it is posited that BTA⁻ anions act as a

nucleating agent to stimulate the formation of a dense and highly crystalline $\text{Mg}(\text{OH})_2$ surface film with a uniform nano-structure capable of passivating the Mg-alloy, in contrast to say, the insoluble Cu-BTA complex via Cu-N coordination bonds for Cu-alloys. The magnitude of this effect of BTA on Mg corrosion was not anticipated, but effective, with beneficial implications to utilization of Mg alloys as anode materials, where in such cases, when dissolution occurs, the anode can passivate with little change in its potential.

171 Sample size and orientation effects of LiAlO_2 single crystal in micro/nano scales

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The mechanical responses of c-plane (001) and a-plane (100) tetragonal LiAlO_2 single crystals are tested by using the microcompression and nanoindentation methods. By compressing the micropillars 2 mm in diameter and 4 mm in pillar height under the micro-scale regime, the measured micro-scaled modulus and yield stress values are 153 GPa and 5 GPa for the (001) plane pillar, and 111 GPa and 3 GPa for the (100) plane. The nano-scaled modulus, hardness and yield strength (the first strain burst) readings of LiAlO_2 single crystals extracted by nanoindentation are 167, 12 and 10 GPa of for the (001) plane surface and 132, 9 and 9 GPa for the (100) surface, respectively. For the sample size effect, it is apparent that the micro-scaled properties measured by microcompression are always much lower than those measured by the nano-scaled properties measured by nanoindentation. This is thought as a result of the small volume constraint effect, the nano-scaled values are higher. For the orientation effect, it is consistent to find that the modulus, hardness and yield strength data on the c-plane (001) samples are always higher than those data on the a-plane (100) samples, due to atomic packing reasons.

172 Characterization of interfacial segregation in magnesium alloys

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Precipitation hardening is the major way to improve the strength of magnesium alloys and aluminum alloys, but the strength level of magnesium alloys achieved so far is substantially lower than that obtained in aluminum alloys. Thus, it is necessary to deeply understand the microstructural factors that are important in controlling the precipitation of strengthening phases. The segregations of alloying or microalloying element atoms in the interface between precipitates and matrix in aluminum alloys have been reported by many researchers, and are believed to stabilize the interface structure and contribute to the strength improvement. But few researchers concern the interfacial segregation in magnesium alloys. In this presentation, we will show our recent findings in the study of interfacial segregation in the Mg-Al-Zn and Mg-Bi-Zn alloys using the high-angle annular dark-field scanning transmission electron microscopy and energy dispersive X-ray spectroscopy chemical mapping.

173 Effects of precipitated particles on microstructure evolution during thermo-mechanical processing of Al-Zn-Mg-Cu alloy

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Ultra-high strength Al-Zn-Mg-Cu alloys have been widely used in aerospace industries for structural components due to their high strength and fracture toughness, low density and remarkable resistance to stress corrosion cracking. Metallurgical features such as dispersoids, constituent particles and precipitates, the grain structure and grain boundary characteristics during thermo-mechanical processing can be controlled to achieve desirable microstructure-property combinations.

In the present work, two kinds of intermediate thermal-mechanical treatments (ITMT) were designed for investigation the influence of different scale precipitated particles on microstructure evolution during thermo-mechanical processing of Al-7.6Zn-1.5Mg-1.75Cu-0.12Zr alloy by means of hot compressive and heat treatment experiments and microstructure testing of OM, EBSD and TEM. The conclusions can be drawn as follows. For samples over-aged at 400°C/14h, the size and distribution of precipitated particles can meet the particle stimulated nucleation (PSN) conditions for recrystallization. Refined and uniform grains present in the sample after compression at $\ln Z=20$ up to 60%-80% reductions and subsequent solid solution treatment at 470°C/2h. For samples aged by "435°C/2h+200°C/12h", when hot compression was carried out at $\ln Z=25$ up to 60% reduction, original grains elongated along the deformation direction. After subsequent annealing and solid solution treatments of "350°C/0.5h+470°C/2h", refined sub-grains present evenly in the elongated grains. Mechanical property testing results of ultra-thick plate forgings indicate that both of the investigated thermo-mechanical processing processes can lead to higher strength, good plasticity and toughness.

174 Control of microstructure of high anisotropic FePt film through interface modification and doping

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The main challenge of the application of $L1_0$ FePt thin films as magnetic recording media is the simultaneous fabrication of FePt (001) thin films with high perpendicular anisotropy and small grain size. $L1_0$ FePt (001) granular films with grain size of 5-6 nm were achieved on MgO underlayer by doping C and Ag. The large opening-up of in-plane hysteresis loop was observed in the FePt film on MgO that would reduce the signal-to-noise ratio. The large opening-up of the in-plane hysteresis loop was caused by the smaller surface energy of MgO which resulted in a large contact angle between FePt grain and MgO and is not favorable for epitaxial growth. The situation became worse when polycrystalline MgO underlayer was used. Any deviation of texture of underlayer and roughness change will cause deviation of the crystal orientation of FePt from (001) orientation. We proposed to use TiN and TiON as underlayers to promote the (001) texture of FePt film and grain isolation. The highly (001) textured FePt-SiO₂-C films with high magnetocrystalline anisotropy and in-plane hysteresis loops with small opening-up and

well-isolated grain with size of 5.7 nm. With introducing new doping materials, we have developed columnar structured FePt-X (001) films with well-isolated small grains and large coercivity on TiON intermediate layer. The FePt grains with size of 5.6 nm showed very good columnar structure with aspect ratio of around 2.6. The out-of-plane coercivity of the film is as high as 23.2 kOe.

175 Effect of buffer layer on microstructure, crystallographic texture and magnetic properties of Co/Cu multilayers

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Metallic multilayers containing alternating ferromagnetic and nonmagnetic layers show excellent giant magneto-resistance (GMR) effect, which is of great interest for applications in the field of data storage and magnetic sensors. Co/Cu multilayers are expected to be one of the best candidates for magneto-electronic device applications. In the present work, we deposit Co/Cu multilayers on Si substrate with Ti buffer layer by magnetron sputtering method and investigate the microstructure, crystallographic texture and magnetic properties of the multilayers using electron microscopy, X-ray diffraction, electron back scatter diffraction and atomic force microscopy. The experimental results reveal that Ti buffer layer has a significant effect on the microstructure and crystallographic texture of the multilayers. We find that the sharp $\langle 111 \rangle$ fibre texture component exists in the multilayers and interfacial roughness of the multilayers decreases when Ti buffer layer is introduced. We also find that the columnar crystallites exist in the multilayers and the generation probability of twins induced by annealing could be decreased due to the existence of the Ti buffer layer. In addition, the magneto-resistance increased as the interfacial roughness decreased in the multilayers. Throughout we discuss the role of buffer layer and demonstrate that the resulting microstructure and crystallographic texture of the multilayers depends strongly on the buffer layer. These characteristics, in turn, control the resulting magnetic properties of the multilayers.

176 Enamel coatings for high temperature protection of superalloys

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High-temperature protective coatings for superalloys are generally divided into the following two types: aluminizing and NiCrAlY coatings. They face two serious problems: interdiffusion and Al fast diffusion. As an inert alternative, ceramic coating spalls easily along its weak interface with superalloy substrate. Recently, enamel coating shows great potential as a novel high-temperature protective coating for the sake that it combines advantages of both the metallic and ceramic coatings, i.e. provides high oxidation resistance, avoids interdiffusion, while forms strong chemical bonding with the superalloy substrate.

For chromia forming superalloys, e.g. K438 and K444, which are trademarks of superalloys

used in power system, their high-temperature oxidation resistance was improved for more than five times by the enamel coatings. The protection mechanism lies in that enamel coating altered the oxidation mode of these superalloys from chromia forming to alumina forming at coating/alloy interface. Under thermal shock conditions, cracks nucleate easily within enamel coating rather than at the coating/alloy interface, which propagates quickly and leads to the final fracture. However, its thermal shock resistance was enhanced by addition of micro-sized alumina particles and NiCrAlY platelets. Sputtering a nanocrystalline bond coating beneath the enamel coating improved further substantially the thermal shock resistance. It was demonstrated that the enamel coating even shows a higher thermal shock resistance than the metallic ones at 1050 °C. Since heat components of thermal power system suffer a lot from chemical corrosion and intermittent operation, the enamel coating gives a good choice for protection at this environment.

177 Microstructure, texture and mechanical properties of magnesium alloys under special processing conditions

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In this study, large strain hot rolling (LSHR) and cold rolling were performed in the Mg-Zn-Gd alloys and a newly proposed multi-directional impact forging (MDIF) was applied to commercial AZ61 alloy. Two Mg-Zn-Gd alloys with different Gd concentration were fabricated by LSHR. The dilute Gd containing alloy presented a fine recrystallized microstructure while the rich Gd containing alloy exhibited a heterogeneous microstructure consisting of high density of shear bands and numerous intersected twins. During the subsequent annealing process, only the rich Gd containing alloy obtained a weak non-basal texture and showed high ductility. During cold rolling, the initial typical TD-tilted texture in the hot-rolled material was replaced by a novel ellipse-like texture after the accumulated rolling strain of 45%. EBSD and TEM observation indicated that {10-12} extension twins and pyramidal <c+a> slip were responsible for the formation of the weak ellipse-like texture. For commercial AZ61 alloy, MDIF resulted in significantly enhanced mechanical properties (ultimate tensile strength of 310 MPa and elongation of 26%) due to the fully recrystallized microstructure and weakened double-peak texture. Consecutive twinning and dynamic recrystallization (DRX) were responsible for the highly efficient grain refinement and texture modification. During the early forging passes, twin-induced DRX associated with {10-11} contraction and {10-11}-{10-12} double twins, and the coalescence and intersection of {10-12} extension twins with various variants effectively refined the coarse original grains and randomized the texture. With continuous forging, continuous and discontinuous DRX subsequently took place and eventually led to the fully DRXed microstructure and double-peak texture.

178 Analysis of deformation and internal defect in flat-wedge cross-wedge rolling of GH4169 superalloy

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The flat-wedge cross-wedge rolling (CWR) deformation and interfacial slip of GH4169 superalloy were investigated experimentally and numerically using a coupled thermo-mechanical finite element analysis (FEA) model. The simulation and experimental flat-wedge CWR forces showed well agreements except near the end of the stretching zone. The simulation analysis showed that the temperature distribution in the work piece was non-uniform during the flat-wedge CWR. When the initial temperature of the work piece was relatively low, the work piece temperature increased, a heating effect of the plastic deformation, while relatively high initial work piece temperatures resulted in cooling the work piece, caused by the work piece contact with the tools. The experimentally measured tool-workpiece interfacial slip was in good agreement with that predicted by the FEA model. The critical area reduction and friction coefficient are compared with the failure conditions that occurred in the finite-element modeling and the prototype experiments.

179 Deformation induced lattice defects and their recovery in nanoscale carbon-rich ferrite lamellae of cold-drawn pearlitic steel wires

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Cold-drawn pearlitic steel wires belong to the most successful engineering materials. High density of lattice defects formed in ferrite lamellae during cold wire drawing is believed to play important roles in their outstanding mechanical properties. In this work, by a combination of X-ray line profile analysis and positron annihilation spectroscopy, densities of deformation-induced defects and their recovery in carbon-rich ferrite lamellae of cold-drawn pearlitic steel wires are characterized. It is shown that both the dislocation densities and the vacancy cluster concentrations increase continuously with increasing drawing strain; upon annealing treatment, the recovery of the lattice defects is dominated by the agglomeration and annihilation of vacancy-clusters at temperatures lower than 523 K, while the temperatures higher than 523 K the recovery process is controlled by the annihilation of dislocations. In the cold-drawn state, contributions of the defect hardening to the total tensile strength of the wires reach nearly 40%, which is mainly ascribed to the dislocation hardening. Upon annealing treatments, the strain aging hardening leads to a maximum strength at 473 K; above 523 K, the annihilation of vacancy clusters and dislocations in ferrite lamellae causes a continuous softening of the wires, where the decrease of dislocation density plays a major role.

180 Effects of laser power on track profile and structure formation during selective laser melting of CoCrMo alloy

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With the recent advance on metal additive manufacturing, there has been a strong effort on studying how selective laser melting (SLM) parameters affect the structure and properties of CoCrMo alloy for medical/dental applications. However, from an energy and heat transfer point of view, how SLM parameters affect the volume of melt and size and shape features of SLM tracks which subsequently affect the amount of lack of fusion (LOF) has not been sufficiently studied. LOF adversely affects the properties of SLM components. In this work, how laser power and thus energy, as other build rate related parameters are kept unchanged, affects the size and shape of SLM tracks and the formation of LOF during SLM of CoCrMo alloy has been studied. Using the recommended condition (180W), solidified tracks are not sufficiently regular in shape. Tracks with the average size have geometrically been demonstrated to insufficiently overlay and overlap for LOF free. Shape irregularity also aids the formation of LOF. Increasing power increases the size and improves the stability of the melt and the shape regularity of the tracks, thus reducing the amount of LOF. The increase in size as power increases is not linear and we relate this to melt boundary to volume ratio which in turn relates to melting efficiency. Microstructures of the SLM CoCrMo alloy samples have also been studied. The influences of increasing power on dendritic/columnar cell size and growth orientation will also be presented.

181 The observation of austenite to ferrite martensitic transformation in an Fe-Mn-Al austenitic steel after cooling from high temperature

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Fe-Mn-Al steels with low density have the potential to substitute for TRIP (transformation induced plasticity) steels. For the development of Fe-Mn-Al TRIP steels, phase transformations play an important role. Our methods of studying the phase transformations of the Fe-16.7 Mn-3.4 Al (wt%) austenitic steel include heating, cooling, and/or annealing. We have studied the martensitic transformation of the ternary Fe-Mn-Al alloy. Single austenite phase is the equilibrium phase at 1100°C, and dual phases of ferrite and austenite are stable at low temperatures. It is noteworthy that lath martensite forms in the prior austenite grains after cooling from 1100°C via quenching, air-cooling, and/or furnace-cooling. The crystal structure of the martensite belongs to body-centered cubic. The formation mechanism of the ferritic martensite is different from the traditional martensite in steels. Ferrite is the stable phase at low temperatures. In the TEM study, we have also found that ferritic twins exist in the martensitic grains. Keywords: Fe-Mn-Al steel, TRIP steels, austenite, ferrite, and ferritic martensite.

182 Cu-induced dielectric breakdown for porous low dielectric constant film under static and dynamic stress

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Cu-ion-migration-induced the porous low-k dielectric breakdown was studied in alternating polarity-bias conditions using a metal–insulator–metal (MIM) structure with Cu as a top electrode. The experimental results indicated that Cu ions migrate into a dielectric film under a positive polarity stress, leading to a shorter time to failure (TTF). Additionally, the TTF obtained in the alternating-polarity test increased with decreasing the stressing frequency, indicating that the backward migration of Cu ions during the reverse-bias stress. Meanwhile, this Cu backward migration effect is effective as the stressing time of a negative polarity stress is larger than 0.1 s. When the frequency is decreased to 10^{-2} Hz, the measured TTFs were higher as compared to a direct-current (DC) stress condition. Finally, the electric-field acceleration factor of porous low-k dielectric breakdown became larger under Cu-ion-migration case.

183 Microstructure and mechanical properties of powder-pack boronized Inconel 625 alloy

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Inconel alloy 625 based nickel has been used in heavy industrial plants because of its high strength and excellent corrosion resistance. However, Inconel alloy 625 has low hardness and poor wear resistance. In this study, powder-pack boronizing process was applied to Inconel 625 alloy for improvement of its hardness related to wear resistance. Boronizing was carried out a powder-pack process using Ekabor-NI powder at 900, 950 and 1000 °C for 2, 4 and 8 hours, respectively. Microstructure and phase formation of boronized samples were analysed by a field emission scanning electron microscopy equipped with energy dispersive spectroscopy, a glow discharge optical emission spectroscopy, X-rays diffraction. Hardness and tribological properties of boronized layer on samples were evaluated by a micro-vickers hardness tester and a ball-on-disk wear tester. The thickness of the boronized layer on the samples increased with the increase of temperature and time. The boronized nickel phases formed on the surface of Inconel 625 alloy were confirmed by X-ray diffraction analysis. The maximum hardness values was up to 1500 Hv_{0.01} with the sample and the friction coefficient and wear resistance of Inconel alloy 625 were improved by boronizing.

184 Orientation dependent nanoindentation response of single crystalline Mg

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The small-scale deformation behavior of single-crystalline Mg was investigated using nanoindentation combined with transmission electron microscopy observation. The nanoindentation on both basal and second-order prismatic planes activated the tensile twin system although their spatial locations are very different. Moreover, atomic force microscopy revealed material pile-up with six-fold symmetry in the former case and a sink-in phenomenon with two-fold symmetry in the latter case. The experimental observations are consistent with our detailed CPFEM simulations which strongly corroborate that the tensile twin formation was attributed to this unique indentation characteristics.

185 Analysis of microtexture development and deformation heterogeneity in the weld region of friction stir welded AZ31 Mg alloy

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Evolution of microtexture in an AZ31 Mg alloy during friction stir welding (FSW) was investigated by microtexture analysis and polycrystal modelling. Characterization of microtexture distribution in as-weld specimens was conducted using electron backscatter diffraction (EBSD) technique. EBSD results revealed the heterogeneous distribution of microtexture in the each welded zone (SZ: stir zone, TMAZ: thermo-mechanically affected zone) after FSW. Finite element analysis (FEA) using the commercial software (DEFORMTM-3D) was conducted to simulate the deformation behavior and deformation history in each welded zone during FSW. The deformation history calculated from the FEA was used as input data for visco-plastic self-consistent (VPSC) polycrystal model to simulate microtexture evolution during FSW. Furthermore, the effect of crystallographic orientation on the texture softening was theoretically investigated using crystal plasticity finite element method (CPFEM). Micro-testing machine and digital image correlation (DIC) technique were used to measure the stress-strain relationship of the weld region under uniaxial tension.

186 Mechanical analyses of welding in practical field

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In order to investigate the essential points of manufacturing process, experience with time should be based. And with the aid of computer, experimental evaluation time was reduced in many production fields. So now computational simulation has been used as complementary

methods of experiments in some practical fields.

And through co-work of experiments and computations, more efficient and new understanding of process are developed in manufacturing. And nice datum were accumulated in many fields of manufacturing. Based on the results of previous nice computational works in manufacturing analyses of each field, continuous analysis on complex field problems in field manufacturing could be approached. In this work, mechanical aspect of welding was investigated as continuous process of production. The examples are shown as three welding related problems.

1. Electric resistance welding 2. Thick pressure vessel welding 3. Non-ferrous welded production

187 Ultrafine-grained equiaxed and bimodal Ti-6Al-4V fabricated by thermomechanical processing

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In the present study, systematic thermomechanical processing and additional annealing in alpha+beta two-phase region were carried out on Ti-6Al-4V with a lamellar/martensite initial microstructure. The processes were carried out in Thermecmator-Z with deformation temperature below 850°C for obtaining ultrafine-grained (UFG) equiaxed microstructure. An UFG equiaxed microstructure with an average grain size of 510 nm could be successfully realized by a compression deformation at 700°C to a true strain of 0.8. In addition, the uniformity of the UFG microstructure was significantly improved by reducing the strain rate to 0.001 s⁻¹. Subsequent annealing at 930°C with different heating rates as well as holding times was conducted to transform the UFG equiaxed microstructure into UFG bimodal microstructures. It was found that the holding time should be as short as possible in order to avoid grain growth, while the effect of heating rate was little. An UFG bimodal microstructure with an average primary alpha grain size of 550 nm could be gotten by annealing at 930°C for 2 seconds. The room temperature strength of both equiaxed and bimodal microstructures followed Hall-Petch relationship. However, with reducing the grain/primary alpha size, the uniform elongation of equiaxed microstructure dropped drastically while it remained nearly unchanged in the bimodal microstructure. Also the uniform elongation of the bimodal microstructure was much larger than that of the equiaxed microstructure. The superior balance between strength and ductility in the bimodal microstructure was possibly attributed to the introduction of interfaces between UFG primary alpha and transformed beta area.

188 Advances in process qualification for powder-bed electron beam additive manufacturing by temperature simulation and measurement

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Powder-bed electron beam additive manufacturing (EBAM) has a potential to offer innovative solutions to many challenges faced in the industry. However, the complex process physics of EBAM hinders efficient process development, qualification and optimizations, etc. Thermal characteristics such as temperatures and melt pools in powder-bed EBAM offer crucial process

variable information that can be linked to the build part quality such as the accuracy and properties. To advance process qualification, a combined numerical-experimental approach was applied to research the process temperatures and melt pool sizes in EBAM using Ti-6Al-4V powder. The objective was to develop a comprehensive thermal model, using finite element analysis, for accurate temperature and melt pool predictions in EBAM. Simultaneously, a near infrared (NIR) thermal imager was attempted to acquire and analyze build surface temperatures in EBAM for model validations and thermal characteristic evaluations.

This presentation will detail the methodologies employed in thermal modeling and simulations, as well as temperature measurements and analyses in EBAM. Challenges in the NIR thermography for EBAM such as the resolution, transmission loss, and emissivity estimate, etc., will be addressed. In addition, the research results such as temperature contours/profiles, melt-pool sizes will be highlighted and compared including the process parameter effects and the influence of the powder-bed porosity.

189 Thermohydrogen treatment of beta titanium alloys

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Hydrogen is usually considered a detrimental alloying element for engineering materials, since it often causes (hydrogen) embrittlement. However, by means of purposeful and thorough application of hydrogen alloying, significant beneficial effects can be attained. It is shown in this contribution that hydrogen can be used as a temporary alloying element during heat treatment of beta titanium alloys, in order to improve the mechanical properties by means of microstructure modification. This process is often referred to as Thermohydrogen Treatment (THT) and allows for establishment of desired microstructures, which cannot be obtained by conventional heat treatment. Beta titanium alloys exhibit high hydrogen solubility and fast hydrogen diffusion. Therefore, these alloys are particularly suitable for THT. Hydrogen as temporary alloying element, which must be removed at the end of the THT process, is strongly stabilizing the beta (bcc) phase of titanium and its alloys. Hence, during heat treatment, the stability of phases and transformation kinetics are strongly affected. Moreover, at high hydrogen concentrations, new hydrogen-induced phases are formed. Two examples of successfully developed and implemented THT processes are introduced, both taking advantage of the volume change and corresponding local matrix deformation associated with hydride formation. In one example (Ti-10V-2Fe-3Al), this effect is used to cause hydrogen-induced recrystallization leading to a small beta grain size. In the second example (Ti-3Al-8V-6Cr-4Mo-4Zr), the temporary hydrides create the precursors for the alpha precipitates formed during aging and lead to a fine and homogeneous distribution of these strengthening particles.

190 Fluorinated copolymer membranes via initiated chemical vapor deposition

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Polymer electrolyte membrane fuel cells are promising candidates for efficient power generation with reduced pollutant emission. However, issues such as membrane degradation and costly membrane synthesis still prevent an extensive commercialization. Within this work,

copolymer proton exchange membranes of 1H,1H,2H,2H,-perfluorodecyl acrylate (PFDA) and methacrylic acid (MAA) are synthesized by initiated Chemical Vapor Deposition (iCVD). This solvent-free method allows for conformal coatings of tailored properties on a variety of substrates by radical polymerization from the vapor phase. The PFDA-MAA copolymers are characterized regarding their chemical composition, swellability, structure and thermal stability by infrared spectroscopy, ellipsometry and X-ray diffraction, respectively. Preliminary specular and grazing incidence X-ray experiments have shown the formation of a bilayer structure of perfluorinated pendant chains of PFDA. Among these bilayers, -COOH enriched regions form through the addition of MAA, resulting in proton conductivities up to 55 mS/cm.¹ The aim of the new research is to demonstrate the correlation between the bilayer structure and the proton conductivity and to study the stability of the bilayers with temperature. Temperature-dependent studies reveal a melting temperature of 81°C and a decrease of the paracrystalline fraction by thermal annealing. A strong correlation between the crystalline structure evolution with temperature and the proton conductivity is demonstrated by temperature-dependent electrochemical impedance spectroscopy. In-situ ellipsometry in aqueous environment confirms a reversible swelling of the membranes up to 12 %, which demonstrates water uptake as well as stability of the membranes, both necessary for the proton conduction mechanism.

¹ Ranacher et al., *Macromolecules*, 2015, 48 (17), 6177–6185

191 Atomically-resolved spectroscopy for emergent phenomena at oxide interfaces

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With the assistance of modern thin-film growth techniques, perovskite oxides with a three-dimensional crystal structure can now be grown in a layer-by-layer manner at atomic-level precision, opening up vast opportunities for unprecedented phenomena at the two-dimensional (2D) heterostructural interface. The emergence of a conductive interface between the two band insulators, LaAlO₃ (LAO) and SrTiO₃ (STO), represents the most celebrated exemplification in this framework. Up to the date, a plethora of oxide interfaces have been reported. It is, however, found that some of the interfaces remain insulating, whereas the same heterostructure-design concept as that of LAO/STO was exploited. To disentangle this puzzle, an atomic-scale spectroscopic characterization across the interfaces is indispensable and the quantitative chemical, electronic mapping by electron energy-loss spectroscopy (EELS) in conjunction with scanning transmission electron microscope (STEM) represents a powerful technique to this end. In this talk, I will explain the principles of atomically-resolved STEM-EELS and the unveiling of intriguing oxide-interfacial phenomena, ranging from the unexpected existence of a localized 2D electron density at the insulating (Nd,Sr)MnO₃/STO interface to the condensation of the 2D interfacial charges into one-dimensional electron chains by the misfit-dislocation strain field. The physics of the LAO/STO interface will be also scrutinized quantitatively at atomic accuracy.

192 Atomic structure and interface layers in thin films oxide heterostructures

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HRTEM and HAADF/STEM experiments were performed on two relevant oxide thin films heterostructures, TiO_2 anatase/ LaAlO_3 (LAO) and $(001)\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO)/ $(110)\text{MgO}$ thin films. Results obtained on TiO_2 /LAO provided evidence of a peculiar growth mode of anatase on LAO characterized by the formation of an epitaxial layer at the film/substrate interface. The film is split into two 20 nm-thick adjacent slabs hosting two different families of crystallographic shear (CS) superstructures, namely (103)- and (101)-oriented CS plane structures. By combining HRTEM results and relevant image simulations with DFT calculations, we determined the atomic structure of the CS planes which resemble the classical rutile-derived $\text{Ti}_n\text{O}_{2n-1}$ Magnéli phases [1,2].

HRTEM and nanodiffraction on superconducting $(001)\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (YBCO)/ $(110)\text{MgO}$ thin films confirmed the expected orthorhombic structure of YBCO and the total absence of twin domains. Moreover, the presence of a pronounced waving of the YBCO lattice planes along the two in-plane crystallographic directions, with a different wave-periodicity depending on the relative film/substrate crystallographic orientation was also observed. We associated the emergence of this feature to a inhomogenous strain induced by the bared $(110)\text{MgO}$ substrate in the two in-plane directions.

In conclusion, HRTEM is proven to be a unique tool characterize the structure at the atomic level and to determine the emergence of unusual properties in the film as a direct consequence of unusual strain mechanisms. [1]R.Ciancio et al., *Nanoscale*, 4, 9 (2012)

[2]R.Ciancio et al. *Phys. Rev. B*, 86, 104110 (2012)

193 The effect of final annealing heating rate to the abnormally growth grains in the Fe-3%Si steel

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The final annealing stage in the manufacturing process of silicon steels allows several grains, mostly $\{110\}<001>$ oriented grains, or Goss grains, to grow abnormally at the expense of other grains. In this study, the growth selectivity process of Goss was investigated through two different heating rate experiments. Several two-pass cold-rolled silicon steel samples were subjected to either rapid heating (RH) or slow heating (SH) ($5^\circ\text{C}/\text{min}$) rate during their final annealing at 900°C . The samples were annealed for 5 min, 10 min, 30 min and 60 min for the SH samples, while for the RH samples annealing was prolonged to 300 min. The texture information from all samples was acquired using electron back-scattered diffraction (EBSD). The orientations of the abnormally large grains (above $\sim 60\ \mu\text{m}$) was stereographically plotted. The constructed pole figures between the SH and RH samples present a different spread tendency around the ideal Goss texture plot. The spread in the RH samples tends to be equal both along the RD and TD, while in the SH samples, the data points tend to spread along the TD axis. In addition to this, the average abnormally large grain diameters exhibit a different size distribution. For up to 60 min annealing, the RH samples exhibit a more linear curve compared to the SH samples. This suggests that the grain boundary properties of the abnormally

growth grains are affected by the heating rate and this results in different growth kinetics between the two samples.

194 Characterization of void-dominated ductile failure in pure Ta

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Prediction of materials reliability requires a physical understanding of the failure process. In the case of void-dominated ductile failure, this includes an understanding of the processes and dominant microstructural variables that lead to void nucleation, growth, and coalescence.

Mechanistic descriptions of void-dominated ductile failure are largely based on studies from several decades ago that relied on optical microscopy techniques. This talk will discuss how modern techniques, such as scanning electron microscopy (SEM), electron backscattered diffraction (EBSD), site-specific focused ion beam (FIB) sample preparation, and transmission electron microscopy (TEM), can enable new insight into the ductile failure process---particularly at the nano to microscale.

As part of a collaborative effort to develop predictive computational models for failure of BCC metals, our experiments are focused on characterizing the void-dominated ductile failure process in pure Ta. Interrupted tensile specimens of 99.9% pure Ta were prepared for void analysis by polishing plastically-strained tensile bars to their mid-plane, where triaxial stresses are highest and thus voids likely to be present. Using EBSD analysis to characterize local misorientations in voided regions, we determined that voids in Ta are formed within grains (as opposed to at grain boundaries) along bands of high misorientation with respect to the tensile axis. We hypothesize that the dislocation processes giving rise to localized regions of high misorientation within grains are promoting the collection of vacancies, thus inducing void nucleation. This hypothesis is being further explored through site-specific FIB sample preparation and TEM analysis of dislocation structures in deformed Ta samples.

195 In-situ characterisation of gamma/alpha decomposition kinetics and interphase morphology and their influence upon interphase precipitation in V and V+Mo microalloyed HSLA steels

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Ever more stringent environmental regulations are a powerful driver for manufacturers within the automotive industry to strive for lightweight solutions in automotive body and chassis applications without compromising passenger safety. An attractive and promising solution is the extension of conventional High Strength Low Alloy (HSLA) steel grades utilising a combination of single-phase ferritic microstructures and optimised interphase precipitation providing significant strengthening through the Orrowan mechanism. The interphase precipitation reaction is a transient process lending itself strongly to take advantage of in-situ characterisation techniques. Through the use of dilatometrical characterisation of the bulk

transformation kinetics and in-situ imaging of the gamma and alpha microstructure and the gamma/alpha interphase morphology using high-temperature confocal laser scanning microscopy, the gamma/alpha interphase morphology and velocity is characterised to simulate and elucidate on processes taking place during isothermal holding at the coiling temperature after hot rolling. A posteriori characterisation of nano-precipitation is undertaken using a site specific FIB lift out and TEM to describe size and distribution of V containing precipitates. The role of isothermal transformation temperature (923, 973 and 1023 K) on the gamma/alpha transformation kinetics and precipitation morphology is analysed. The results of in-situ characterisation suggest that it is possible to optimise the thermal processing of microalloyed HSLA and to maximize strengthening potential of the precipitation processes.

196 Phase progression during reactive sintering of NiTi using in situ neutron diffraction

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The phase progression during reactive sintering of elemental Ni and Ti powders to produce the shape memory alloy NiTi was investigated using in situ differential scanning calorimetry, in situ neutron diffraction and microscopy. Phase fractions were determined using Rietveld analysis which has led to a better understanding of the sintering process. During sintering all three intermetallics in the system (Ti_2Ni , NiTi , and Ni_3Ti) form with Ni_3Ti being the only undesirable intermetallic to be fully removed. The remaining Ti_2Ni is typically labelled in literature to be either Ti_2Ni or a $\text{Ti}_4\text{Ni}_2\text{O}$ oxide phase. It is proposed that the two phases are in reality one phase: $\text{Ti}_2\text{Ni}(\text{O})$, an intermetallic phase with oxygen in solid solution. Ti_2Ni has a peritectic temperature of 1257 K, however when O is present Ti_2Ni has been found to be crystalline at temperatures up to 1473 K. Ti_2Ni acts as an oxygen getterer in the NiTi system. As the sample moves towards equilibrium the volume of $\text{Ti}_2\text{Ni}(\text{O})$ decreases, however the O remains in the Ti_2Ni enriching its oxygen content. This makes this phase difficult to remove and highlights the importance of purity with regards to O in the starting powders.

197 Analysis of pre-strain in a hybrid forming process including stretch and incremental sheet forming

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The incremental sheet forming process is being studied due to the rising demands on this manufacturing process. The incremental forming process can be applied in different areas, such as prototyping and the production of small batches of parts, aiming to reduce manufacturing costs. The conventional sheet metal forming processes, such as deep drawing and stretch forming, are used to produce large batches of parts while incurring higher initial manufacturing costs attributed to the use of a large amount of tooling. The application of conventional and incremental forming processes combined in the same metal sheet is called hybrid forming. This hybrid process is enacted by pre-forming the sheet through the stretch forming process followed by the final manufacture using the incremental process. The objective of this work is to analyze the influence of the pre-strain imposed during the conventional process on the DC04 metallic

alloy relative to the maximum strain obtained. A numerical simulation was used to define the parameters for the conventional process and to assess the experimental results. The higher major true strains are inversely proportional to the pre-strain in both experimental and simulated results.

198 Interfaces in functional materials: A pathway to design better properties

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Cu(In,Ga)Se₂ (CIGS), Cu₂ZnSnSe₄ (CZTSe), and multicrystalline Si (mc-Si) solar cells possess high efficiency, despite the polycrystalline structure of the absorber layer. One of the major factors controlling cell efficiency is the diffusion of impurities during the fabrication process into the absorber layer and to the p-n junction. However, the interaction between the defects and the impurities at the internal interfaces is not completely understood and often hard to track owing to the small concentrations at the internal interfaces at the nanoscale.

As a step towards a better understanding of the impurity redistribution at the internal interfaces, we have developed novel approaches of preparing site-specific atom probe specimens using combined focused ion beam (FIB), (scanning) transmission electron microscopy ((S)TEM) and electron backscattered diffraction (EBSD). These approaches allow selected GBs in polycrystalline CIGS, CZTSe, and mc-Si to be studied by atom probe tomography (APT).

Moreover, the electrical, structural, but also the chemical composition of the p-n junction in photovoltaic devices need to be controlled to achieve high-efficiencies. Joined APT-TEM data of the p-n junction of various solar cells will be equally presented. The aim is to understand the correlation between impurities and point defects at the internal interfaces of CIGS, CZTSe, and mc-Si solar cells and possible consequences for the cell efficiency.

199 Fatigue crack growth in forged and flow formed IN718

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Shear flow forming are evolving as cost effective process's for the manufacture of engineering components such as tubular shaft and cone structures. Whilst preserving high dimensional accuracy, production lead-times are reduced and material yields are improved through the reduced reliance on final machining and ambient temperature of the process operation. Clearly, subsequent mechanical properties are influenced by the degree of cold work during forming and any associated post process heat treatment.

Evaluating these mechanical properties in the laboratory setting can be difficult. Extracting conventional scale test specimens from the relatively thin and curved sections may not be possible. In addition, it may also be desirable to retain and sample the as flow formed surface to ascertain any control of the finishing process on properties, particularly fatigue crack initiation. Supplementary characterisation of microstructure / properties relationships is affected by the high degree of deformation imparted to the material, particularly when attempting to use electron back scattered diffraction (EBSD) to identify crystallographic

orientations and micro-textures. The present paper will describe the results of novel test techniques for the evaluation of fatigue crack growth behaviour in flow formed IN718 material. Measurements were obtained at room and elevated temperatures. Detailed microstructural characterisation, employing optical and scanning electron microscopy together with EBSD, will identify grain size and morphology, grain orientations and associated precipitation phases. A direct comparison to crack growth in similar materials manufactured via a conventional wrought process will be made.

200 Lattice strain measurement and simulation for non-proportional biaxial deformation

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High energy synchrotron X-ray diffraction data have been acquired in-situ during the deformation of a ferritic sheet steel for a number of proportional and non-proportional strain-paths. Specially designed cruciform specimens were deformed using a bespoke biaxial loading mechanism, permitting arbitrary strain-paths to be applied to metallic specimens. By collecting the full Debye-Scherrer diffraction rings, lattice strain as a function of azimuthal angle was obtained; corresponding to the in-plane lattice strains of the specimen. Crystal plasticity finite element modelling (CP-FEM) replicated these strain-paths with a starting texture obtained from electron backscatter diffraction measurement of the test material. Two hardening laws; self hardening and isotropic hardening were used to investigate the resulting lattice strain distributions. At increments of deformation comparable to the experiment, a methodology was developed that simulates diffraction patterns, from which lattice strains were calculated. Whilst it is only possible to model a small number of grains compared to those measured experimentally, it was necessary to relax the tolerance on the Bragg angle to improve the statistics of the simulated diffraction patterns. Good agreement was observed between the experimental and simulated lattice strains during proportional loading, demonstrating that relatively simple hardening laws are sufficient to describe this deformation response. However, deviations in lattice strain are observed between the experiment and model when the strain-path is changed. It is anticipated that these results will provide the basis for the future calibration of CP-FEM models, by updating hardening laws or otherwise, to accurately describe the deformation response of complex strain-paths.

201 Modeling of the microstructural evolution and yield strength of an innovative age-hardenable Al alloy for high temperature applications

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Age-hardenable Al-based cast alloys are promising materials in those industrial fields where high specific strength and resistance to high temperatures are needed. The mechanical properties of such alloys are strongly affected by their microstructure: the size and shape of precipitates, their homogeneous distribution and their coherency with the matrix are of primary importance for an effective strengthening of the alloys at elevated temperatures.

Physically-based models are powerful tools to analyze the influence of the mentioned parameters on the mechanical properties of the alloy after age hardening, and also to predict the effect of high temperature service conditions on microstructure evolution.

Among others, the Kampmann-Wagner-Numerical model (KWN) is thought to be one of the most physically sensible to describe nucleation, growth and coarsening of the precipitates during precipitation hardening and working conditions at high temperature.

Scope of this work is to apply the KWN model to an innovative Al-based cast alloy, to predict the microstructural evolution during high temperature exposure and the resulting alloy yield strength. The followed approach contributes to demonstrate if the precipitates have an improved resistance to coarsening and, consequently, if the alloy is a suitable candidate for applications at temperatures higher than the actual ones. The as-cast precipitates dimensions and type will be considered as a given parameter, neglecting, at least as a first approach, any influence of the casting cooling rates. Appropriate model calibrations and experimental validations are a fundamental requirement to obtain reliable theoretic results.

202 Formation and dissolution of hydride precipitates in zirconium alloys: Crystallographic orientation relationships and stability after temperature cycling

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Hydride precipitation due to the spontaneous and fast hydrogen diffusion is often pointed as causing embrittlement and rupture in zirconium alloys used in the nuclear industry. TEM, SEM-EBSD and XRD have been used to study the precipitation of hydride phases in zirconium alloys as a function of the hydrogen content. The orientation relationships observed between the hydride phase and the substrate were similar to those previously observed in Titanium hydrides grown on Titanium [1, 2, 3]. Dislocation emission from the hydride precipitates has been directly related to the relaxation of the misfit stresses appearing during the transformation. The stability of the hydride phases after several dissolution-reprecipitation cycles have been studied by DSC, TDS, TEM and XRD for different total hydrogen content in several alloys. The energy of precipitation observed is lower than that of the dissolution in each case studied. The temperature associated to these two processes slightly increase as a function of the cycle number, as a result of the homogenizing hydrogen distribution in the alloy bulk. The same hydrides phases present before cycling were also observed after 20 cycles. However, transition phases poorer in hydrogen than the dominant one may precipitate at the interface with the substrate. The evolution of these transitions phases with the temperature increasing will be investigated by TEM in-situ heating in the next future.

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[2] X. Feaugas, E. Conforto, *PlastOx 2007*, France, 2007, pp 161-178, EDP Sciences, 2009.

[3] Conforto, E., Caillard, D. *Solid State Phenomena* 172-174 (2011) 242-247

203 In-situ x-ray observations of the effect of ultrasound on liquid and semi-solid metal alloys

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Casting is a fundamental processing technology for metallic materials. Currently, there is considerable interest in using ultrasound during casting as a way of modifying and improving the solidification microstructure. In support of larger scale ultrasonic processing trials, we performed high speed X-ray imaging to observe the effect of applying ultrasound to small samples of metal alloys during solidification. X-ray imaging is necessary because metals are opaque to other techniques. By varying the experimental conditions, we aimed to establish the significant phenomena that occur when ultrasound is applied, and relate them to the parameters we can control like temperature and ultrasound power. The experiments were performed at Beamline I12 of the DLS Synchrotron, using ultrasonic processing apparatus developed at Hull University. The apparatus consists of a small furnace which is used to heat specially-designed quartz cells containing the metal alloys under investigation. The ultrasound generator has controllable output up to 100W at 30 kHz. The alloys studied were Sn-30wt%Cu and Bi-8wt%Zn. The majority of imaging was performed at 2000 frames/second. We observed extended cavitation and bubble cloud phenomena in the two alloys under known temperature and ultrasound power conditions, contributing to the knowledge of cavitation behaviour in opaque media. We also observed the formation of steady state vortices in semi-solid Sn-30wt%Cu at 575°C. Digital Image Correlation (DIC) was successfully used to determine the velocity field in the vortices. Modelling of the ultrasonically-induced flow phenomena based on experimental observations is ongoing. We plan further experiments on other alloy systems.

204 Understanding the mechanisms of blended powder sintering of Ti alloys using combined thermal analysis

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Blended elemental (BE) sintering, where commercially pure Ti powder is blended with pure Al and V powders to a bulk composition of Ti-6wt% Al-4 wt% V is an economical way to produce Ti6Al4V for aerospace and automotive applications. A related method is master alloy sintering, where commercially pure Ti powder is blended with a pre-alloyed powder containing 60 % Al and 40% V. During sintering the Al and V additives diffuse throughout the Ti powder compact and ideally form a homogeneous bulk alloy composition. In some cases these BE and MA additions can also act as sintering aids, resulting in improved densification.

The goal of this work was to develop combined high temperature DSC a dilatometry methods capable of determining the mechanisms of BE and MA sintering in the Ti-Al-V system. Included in the work was an identification of any sintering aid contributions to the rate of densification and in-situ alloying of the Al and V into the Ti matrix. Particular emphasis was placed on determining the relative contributions of Al and V to the sintering process.

205 Materials research at University of Nevada, Las Vegas

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High-pressure experiments on strongly correlated-electron systems allow the study of the relationship between structural, elastic, electronic, and magnetic properties of d- and f- band systems. The High Pressure Science and Engineering Center (HiPSEC) at UNLV has established a multi-disciplinary research program focusing on fundamental experimental and computational studies of material properties at extreme conditions. I will report on recent results on d- and f-electron materials at high pressures.

206 Phase transformations in nano-bainitic steels produced by direct-strip-casting

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Direct strip casting (DSC) is an innovative near-net-shape process for the formation of thin steel sheets which drastically simplifies the conventional process, allowing significant energy savings. In the current era of producing green technologies with reduced environmental footprint, DSC is the ideal candidate for steel sheet production and has thus been taken to the industrial stage in the past decades. The materials produced by DSC undergo extreme cooling rates, resulting in highly out-of-equilibrium conditions of solidifications and further phase transformations. The current challenge is to be able to predict the final microstructure under such processing conditions as a function of the steel composition.

Nano-bainitic steels have been increasingly studied in the past decades as they offer unprecedented mechanical properties but their upscaling to industrial applications has not been possible due to the high processing costs of these alloys using the conventional steel processing method. The aim of the current project is to explore the use of DSC to process nano-bainitic steels by understanding the phase transformations and the effect of minor alloying additions on the microstructure and resulting mechanical properties of DSC nano-bainitic steels. Five compositions were carefully selected to study the influence of chemistry (carbon, silicon and chromium content) on the microstructure development during DSC. The microstructure and precipitates are investigated using SEM, TEM, EDS and XRD. The impact of each element on microstructural development will be presented. The nano-bainitic steels are found to exhibit an excellent combination of strength (>1GPa) and ductility.

207 Nano-oxides derived from hydrotalcites as catalysts for dry methane reforming reaction – effect of [Ni(EDTA)]²⁻ adsorption time

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The hydrotalcite containing magnesium, aluminium and cerium were synthesized by the co-precipitation method at constant pH. The M^{2+}/M^{3+} molar ratio was equal to 3, and the amount of cerium was calculated so that resulting hydrotalcite would contain 5 wt.% of Ce. The obtained material was kept in contact with the solution of 5 wt.% [Ni(EDTA)]²⁻ complexes in the closed Erlenmeyer flask with stirring respectively for 4 and 24h, in order to introduce into the catalytic system nickel species. The resulting materials were calcined at 550°C for 4h in order to obtain nano-oxides with periclase-like structure. Subsequently, hydrotalcite-derived materials were characterized by XRD, FTIR, H₂-TPR, low temperature N₂ sorption and CO₂-TPD techniques. The characterization of the materials confirmed that synthesized samples were hydrotalcite-derived mesoporous materials and that through adsorption method the small amounts of nickel were introduced into the catalytic system. The contact time with the solution of [Ni(EDTA)]²⁻ complexes had an influence on the catalytic properties of obtained catalysts in the reaction of dry methane reforming – an important process with huge future potential as industrial method of syngas production. Catalytic tests and characterization of the materials showed that sample which underwent absorption of [Ni(EDTA)]²⁻ complexes for 24 h showed higher conversions of CO₂ and CH₄ at the level of respectively 20 and 14%.

208 Laser induced surface texturing of metal or organic substrates for structural adhesive bonding

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Laser-matter interaction is commonly described regarding three main factors: laser beam, materials and environment. Conversion of absorbed energy via collision process into heat is the most important effect that occurs during laser interaction. Short pulse laser beam induces fast transition from the overheated liquid to a mixture of vapor and drops which allows ablation of micro-metric layers. Specific patterns can then be achieved through the use of scanning and automation technology also called laser texturing.

New materials with specific properties such as endurance life and/or lower environmental impact attract emerging technologies such as thermal spraying. However, adhesive bond strengths have to be high enough and appear as a key feature of surface properties. A clean surface to enhance surface interlocking is a key element. Mechanical and physico-chemical bond strength for thick coatings elaborated by thermal spray process can then be developed using laser. The aim of this present article is to show the potential of such emerging treatments through new results using various spray processes (thermal spraying as well as cold spraying). Metal or organic materials were investigated implementing various powders.

209 Spark plasma sintering: A route for manufacturing TiAl blades?

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The aim of the present study is to evaluate the SPS as a possible route to produce TiAl blades for aircraft engines. This requires to obtain alloys with improved properties, satisfying the industrial requirements, and to be able to produce near-net shape components.

The first part of this talk will be devoted to the development of a performing alloy, with a good compromise between creep resistance at high temperature and ductility at room temperature. The microstructures were mastered through the optimization of chemical composition and the control of the parameters of the SPS cycle. To explain and improve the mechanical properties, post mortem analyses of samples deformed at room temperature and crept at service temperature were performed. They were supported by in situ straining experiments performed inside the transmission electron microscope.

In the second part, the method followed to sinter directly near-net shape blades by SPS will be described. The basic idea is to use the graphite assembly containing the powder to give the final shape to the piece. Finite Element Modelling is used as a predictive tool to determine the processing parameters and to control the temperature. The density and the microstructure of the products will be examined. The aim is to achieve materials with the optimized properties without applying subsequent thermal treatments.

As a conclusion, this work demonstrates that SPS may allow the production of blades, made of performing TiAl alloys.

210 Kinetic behavior of Fe-Ni-C martensitic steels during aging at room temperature

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The kinetic behavior of a Fe-24 Ni-0.4 C (weight percent) martensitic steel during aging at room temperature has been investigated by transmission electron microscopy. Electron diffraction, coupled with imaging techniques in a transmission electron microscope, including high resolution studies, have been used at various stages during the aging process, in order to characterize the microstructural features of the analyzed samples. Rapid cooling (quenching) in liquid nitrogen of the austenitic state of the above-mentioned material leads to the formation of a martensitic phase which, under normal conditions of temperature (293 K), begins to transform, through a process called spinodal decomposition. As a result of this process, a modulated structure is formed, in which carbon-rich regions (precipitates) occur in a periodic manner throughout the sample, leading to the presence of diffuse streaks or satellite spots around each fundamental (matrix) reflection on the electron diffraction patterns. This process is thus accompanied by a reduction of the tetragonality of the martensitic phase (bct), which evolves towards the formation of a cubic structure (bcc), corresponding to alpha-iron (ferrite). The microstructure of the carbon-rich phase is also analyzed by electron diffraction. The evolution of the tetragonality in time, translated as the c/a ratio, which is also function of the carbon composition, is studied. Moreover, the variation of the distance between the carbon-rich

precipitates (in fact, the periodicity of the modulated structure), along with the evolution of their width and length, are also observed.

211 Image based modeling of plasticity in polycrystals: From 2D to 3D

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Images of materials microstructures can easily be obtained today with the wide accessibility of techniques such as standard metallography, electron microscopy, EBSD in research labs and in the industry. Even 3D characterization becomes more and more popular with the emergence of serial sectioning in a SEM or computer assisted tomography for example. Moreover, fine constitutive models of the mechanical behavior of materials have been developed in the last decades and the increase of computational power tends to allow researchers to build models based on experimentally observed microstructures. Direct coupling between experimental observations of plasticity and modeling yields improvements in the understanding of the underlying mechanisms. The Mateis lab has developed a strong expertise in this field based on in-situ mechanical testing in a microscope or in a tomograph and on finite element modeling. A few examples of microstructure based models will be shown in this work with an emphasis on the procedures developed to build the models and on the effect of real microstructures (phase or grain morphology, local crystal orientations, ...) on the development of damage and plasticity during testing.

212 Comparative study of optimization in pultrusion with pre-heating and die-cooler temperature for improved cure

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Pultrusion is a composite manufacturing technique for processing continuous composite profiles with a constant cross section. During processing, the heat flux provided by the mold must be sufficient to promote the polymerization reaction of the thermosetting matrix (curing). Due to the exothermic character of the curing reaction, inside the composite exceed temperatures imposed by the cure cycle. This temperature rise can cause degradation of the final product. Thus, the process simulation is quite important for the prediction of pre-heating and die-cooler temperature. Some work in this area is presented by Carlone et al. (2006) that conducted simulations of the pultrusion process using the method of finite elements for an epoxy resin where was presented comparative results between two methods: Finite Difference and Finite Element. Joshi et al., (2003) discussed in his study the development and application of approximation using FE and volume control in three dimensions (3D). This work has the aim to compare the studies, present in literature, of optimization in pultrusion with pre-heating and die-cooler temperature to get the best optimization methodology. In the present study, a general-purpose FE software, Abaqus, is used in order to perform three dimensional (3D), conductive heat transfer analysis. The results will show the best simulation methodology. Thus quantify the uniformity of cure, by calculating the squared deviation of composite curing degree

of the values in the output section of the mold (metal) in relation to a degree of cure desired value.

213 Dynamic piezoelectric behavior of lithium niobate at high temperature

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High temperature piezoelectric materials have numerous potential applications, including high temperature ultrasound NDT, MEMS, sensors, or actuators. However, conventional piezoelectric materials are unsuitable for continuous operation above 400°C. Lithium niobate (LiNbO₃) is a promising candidate because of its very high Curie temperature (approximately 1210°C) and reasonable piezoelectric coefficients. However, the piezoelectric properties are not sufficiently understood, and long-term operation is problematic due to unidentified degradation mechanisms well below the Curie temperature, suspected to include phase transformations, oxygen loss, and excessive ionic conductivity. In order to better understand these physical mechanisms, electrochemical impedance spectroscopy (EIS) is used to characterize monocrystalline LiNbO₃ from room temperature to 900°C, with excitations from 20 Hz to 20 MHz. An equivalent circuit model analysis, including resonant frequencies, is employed to investigate the time and temperature dependence of the piezoelectric behavior, as well as the mechanical elasticity and damping. In addition, conventional quasi-static measurements are performed to validate this method. Numerical values extracted from dynamic experiments allow for numerical simulations to model device behavior. Most importantly, the improved understanding of degradation mechanisms obtained by this investigation is critical to attain the thermal stability of LiNbO₃, thereby enabling previously elusive high temperature piezoelectric applications.

214 On the development of specific heat treatments for TA6V parts produced by electron beam melting

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Electron Beam Melting (EBM) is an additive manufacturing process for metal parts. It can produce three-dimensional objects by melting successive layers of powder to the exact geometry defined by a digital model. As a result of the complex thermal history undergone by the Ti-6Al-4V alloy during the EBM process, characterized by a succession of melting, rapid cooling and partial remelting, a specific microstructure is generated. This fine lamellar microstructure differs notably from the equiaxed or duplex microstructures typically observed in conventionally processed Ti-6Al-4V. It is known that in order to obtain these microstructures, hot working in the two-phase region is required. However, in the case of near-net shape parts obtained by EBM, such a mechanical post-treatment cannot be considered. Therefore, in order to optimize the mechanical properties of electron beam melted parts, it is crucial to focus on the development of alternative thermal post-treatments. In this work, the effect of both subtransus and supertransus treatments was evaluated. Depending on the holding temperatures and cooling rates, these treatments generated a wide range of microstructures. The

resulting mechanical properties were discussed and compared to those of the as-build material. More specifically, the development of microstructures consisting of a mixture of primary alpha and martensite was deeply investigated. It is shown that such microstructures resulted in an original tensile behavior compared to the as-build material. Indeed they lead to a substantial increase in work-hardening rate combined with a much lower Young's modulus.

215 Microstructure behavior of pure titanium processed by gradation extrusion and its influence on the mechanical properties

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Severe Plastic Deformation (SPD) processes have been used for microstructural and mechanical improvements of commercially pure titanium. The microstructure provided by these techniques is consequence of the equally imposed high strain on the entire material, revealed by a constant grain refinement profile, which brings a homogeneous high strength. Due to the low ductility along the whole material, this uniform property does not make feasible subsequent forming steps that could be used for industrial applications, such as medical and dental implants manufacturing. The gradation extrusion method, which combines SPD with conventional impact extrusion, appears as a potential technique to generate a microstructural gradient with an increasing grain size in the radial direction of the extruded bar. This gradient distribution influences the material properties, providing a high mechanical strength near the surface due to the fine-grained structure and a core with higher ductility due to less refined grains. The nanostructured volume of the material is provided by several changes in deformation direction and dynamic recrystallization, in consequence of the integration of severe deformation into the process. Aiming to investigate and evaluate the pure titanium microstructure behavior during deformation, a numerical simulation was developed, and experimental forming tests were carried out using a gradation extrusion tool. EBSD analysis of the obtained microstructure showed the material texture and the gradient of grain refinement. A microhardness map through the longitudinal section revealed the influence of the localized strain levels distribution on the mechanical properties.

216 Mechanically driven martensite formation in ultra-strong pearlitic steel

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Pearlitic steel wires are amongst the strongest structural materials and can reach exceptional strength of as high as 7 GPa [1]. Such extreme properties can be achieved if the lamellar microstructure of cementite and ferrite is refined by severe plastic deformation and dislocation plasticity largely suppressed. Wire drawing of pearlitic steel leads to a strain-driven fragmentation and dissolution of the cementite phase finally rendering the material into a nanostructured composite. So far the underlying microstructure mechanisms are only partly understood. Especially, the accommodation of carbon in ferrite stemming from the cementite

dissolution process remained unclear. Here we discuss the mechanisms of the heavy deformation on the structural transformation of ferrite by using a combination of synchrotron X-ray diffraction, atom probe tomography and atomistic modelling. Based on our results we find that the carbon supersaturation of ferrite induces martensite formation. This is surprising, as martensite is believed to form only by rapid quenching of steel from the high temperature austenite field. Therefore, we propose that cold drawing can induce a martensitic transformation. [1] Li, Y.J.; Raabe, D.; Herbig, M.; Choi, P.; Goto, S.; Kostka, A.; Yarita, H.; Borchers, C.; Kirchheim, R.: Phys. Rev. Lett. 113 (2014) 106104

217 Metallic muscles: Nanoporous materials at work

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We will concentrate on electrical-to-mechanical energy conversion using nanoporous metal-polymer composite materials. Nanoporous metallic actuators constitute a new class of low-voltage actuators that feature a unique combination of relatively large strain amplitudes, low operating voltages, and high specific stiffness and strength. These so-called 'metallic muscles' consist of ligaments and pores in the nanometer regime giving rise to a very high internal surface area. The key obstacles to the integration of nanoporous metals into current fundamental concepts and technological applications (MEMS, NEMS) are (i) the presence of the aqueous electrolyte itself that is needed to inject electronic charge in the space-charge region at the metal/electrolyte interface. (ii) the rate of actuation due to the relatively low ionic conductivity of the electrolyte, and (iii), the magnitude of the actuating displacements. Here we discuss a novel approach to generate work from metallic muscles that overcome these hurdles. From an experimental viewpoint a new ultrafast, all-solid organometallic actuator has been designed, synthesized and tested. The tunable, semiconducting properties of conjugated polymers are exploited to inject charge into the metal. In addition, a new microstructural design based on a layered structure with enhanced actuation strokes has been developed. In the presentation also size effects of metallic muscles in particular will be discussed.

218 Relationship between the tensile strength-fracture toughness balance and the multiscale microstructure of a maraging stainless steel for aircraft applications

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The stainless maraging steels composition is based on the Fe-Ni-Cr-Mo-Ti-Al system and the recent development of new grades aims to achieve a higher tensile strength-fracture toughness balance, compared to other stainless grades. These research activities are driven by the needs of aircraft industry that requires weight reduction of structures in order to decrease consumption, noise levels and emissions of CO₂ and nitrogen oxide (NO_x).

However, the fracture toughness still needs to be optimized as this property still remains too scattered after heat treatment and in some unexpected cases, the fracture toughness does not reach the minimum value required for the specific applications.

The main objective of this work is to determine the microstructural elements that control the fracture toughness property in order to better understand the probable micro-mechanisms involved during the mechanical test and above all to suggest alternative heat treatments that can improve the toughness. Thus, the tempered martensitic structure was investigated at different scales for different conditions of the heat treatment. The quantity, morphology and distribution of precipitated phases in the martensitic laths were extensively analyzed by scanning electron microscopy observations as well as small-angle neutron scattering and x-ray diffraction (synchrotron source) experiments. Reverted austenite and two different intermetallic phases (η -Ni₃(Ti, Al), β -NiAl) were found to influence the balance of properties. Depending on the heat treatment, the volume fraction and the inter-particle distance of each phase are analyzed and the discussion is focused on the possibility of finding a suitable combination of those microstructural parameters.

219 Accelerated ageing and Portevin-Le Chatelier effect in Al 2024

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2024 aluminium alloys are used in aeronautics for various applications. However, the ageing treatment (T4) used to bring them to optimal mechanical properties, that is carried at ambient temperature, poses several problems. The first one is its length (5 days) which brings the need for storage of the parts. The second is the poor control of temperature during this ageing process that makes reproducibility of the treatment difficult.

The aims of this work was to develop an alternative treatment leading to optimized mechanical properties in a shorter time. For this, we investigated the kinetics of the ageing process and observed the apparition and disappearance of Portevin- Le Chatelier (PLC) effect in 2024 Aluminium alloy. The knowledge of the time at which PLC effect is observed is important in terms of process because shaping may be carried out before the end of the ageing process.

220 New route to develop multi-structured anti-CMAS coatings to protect thermal barriers

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Thermal barrier coatings are widely used to protect critical metallic parts of aircraft engines from oxidation and hot corrosion. It enables an extended lifetime and an increased efficiency by increasing operating temperature. A TBC system consists of a bond coat, a TGO (thermally grown oxide) and a ceramic top coat for thermal insulation, typically stabilized zirconia. Currently, the ceramic layer is industrially manufactured by EBPVD or by APS resulting in columnar or lamellar microstructures respectively. Innovative routes such as sol-gel processes are now developed to synthesize ceramic layers with non-directional porosity and offers benefits in terms of processing costs and flexibility. It is possible to repair damaged TBCs and to prepare either thin or thick conformal ceramic coatings. In working conditions, TBCs are subject to various kinds of degradation like CMAS (Calcium Magnesium Alumina Silicates) which

deteriorate the integrity and mechanical properties of TBCs by infiltrating the internal porosity of the TBCs. We propose to use sol-gel route to synthesize new functional coatings shaped by dip-coating technique. Several sol-gel formulations have been investigated to define the best compromise to elaborate TBCs with a lifetime similar or better than conventional systems. Multifunctional coatings with yttria and stabilized zirconia able to resist both aggressive environmental oxidation cycles and CMAS infiltration are proposed. The effect of key parameters such as the ratio of particles into the slurries, their morphology, microstructure and the impact of the temperature of heat treatment have been investigated.

221 Computational analysis of irregular rolling deformation in Nickel Aluminide single crystals

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Intermetallic Ni₃Al has some rolling deformability when brittle grain boundaries are removed from the microstructure. However, the rolling deformability strongly depends on the crystallographic orientation. Its single-crystalline sheet can be cold-rolled into a thin foil by a large reduction more than 90% when the initial rolling direction (RD) is parallel to $\langle 100 \rangle$. In case of the RD // $\langle 211 \rangle$, an irregular rolling deformation occurs, as follows: a widening along the transverse direction, bend, shear deformation in the rolled plane, tearing, and so on. It is likely that some slip systems necessary for regular rolling deformation were strongly hindered in this rolling direction. For understanding the mechanism that yielded the irregular rolling deformation, it is essential to determine the slip systems working through the actual rolling deformation in a quantitative manner. In this study, we analyzed the slip systems that worked in the irregular rolling deformation based on both changes in the outer shape and the crystallographic orientation of the rolled sheet by a computational method. We used an open-source crystal plasticity calculation code named DAMASK (Duesseldorf Advanced Material Simulation Kit). We set the orthogonal components of the gradient tensor from the change in the outer shape of the rolled sheet and then optimized the remaining shear components so as to that the calculated final orientation was as close to the experimentally measured one as possible. Based on the analyzed results, we will discuss a possible mechanism in terms of slip-slip interactions.

222 Process parameter optimization of fused deposition modeling for helical surfaces using grey relational analysis

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Fused Deposition Modeling (FDM) is a process for developing physical objects by depositing fused layers of materials according to numerically defined cross sectional geometry. One of the major drawbacks of FDM is the reduced part quality mainly in terms of dimensional accuracy, surface finish and mechanical characteristics. The major share of literature related to the field of FDM process parameter optimization focuses on flat and circular surfaces, while only a few studies are available on helical surfaces. This paper is based on a close study conducted to

understand how the process parameters of FDM affect the dimensional accuracy, tensile strength and surface finish of helical surfaces. The process parameters selected for study are layer thickness, print speed, raster width and support material density. The experiments to be run were designed by taking three levels of each process parameter selected. The optimum parameter setting for improving the responses individually was done using Taguchi method. Optimum parameter level for improving dimensional accuracy, tensile strength and surface finish simultaneously were done by Grey Relational analysis. The main effect plots were also analyzed. It is concluded that a minimum level of layer thickness is optimum for helical surfaces. This study attempts to provide the knowledge that contributes to the industrial businesses to produce helical end-parts that able to perform in real conditions. Keywords— FDM, Taguchi method, Grey Relational analysis, Optimization, Helical Surfaces.

223 Investigation of nanoscale interphase precipitates within Ti and Mo microalloyed steel

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Improved strength in microalloyed steels is reported to be imparted by alloy carbide particles precipitated in a fine-grained ferrite matrix. These particles are chiefly formed as interphase precipitates during austenite to ferrite transformation. Nanometre-sized carbides formed in Ti and Mo bearing steels are found to have high thermal stability, making these steels suitable for industrial production. The presence of Mo in these alloy carbides is assumed to be responsible for the sluggish growth kinetics of the particles, although its exact role is not clearly understood. This work attempts to study the nature of the carbide particles in a Ti-Mo microalloyed steel upon varying thermomechanical processing conditions. Atom probe tomography and transmission electron microscopy have been utilised for characterising the nanoscale particles in terms of chemical composition, size and volume fraction. Matrix atoms such as Fe and Mn were observed within the carbide particles composed of Ti and Mo, which have an average particle size within the nanometre range.

224 Finite element modelling of powder densification during spark plasma sintering process

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Spark Plasma Sintering (SPS) has the potential for rapid and efficient consolidation on a wide variety of powder materials. This technique allows both the enhancement of densification kinetics and the conservation of the material nanostructure thanks to the combination of high heating rate, dwell times of few minutes and an applied uniaxial pressure. However, the industrialization of this sintering method are limited by its low reproducibility and scalability. Therefore, there is a critical demand to increase the level of knowledge of the behavior of the powder during its densification stage through the development of reliable process models. In this study, a fully predictive finite element modelling of the process has been developed. To describe the nonlinear-viscous flow of the porous material, the micromechanical model suggested by Abouaf et al.[1] has been integrated. The use of such constitutive equations is

usually limited by the numerous experiments needed to identify the included mathematical parameters for a considered material. In the present work, analytical expressions of these porous parameters have been suggested thanks to an analogy conducted with the rheological approach of sintering developed by Olevsky [2]. The conducted methodology leads to a good correlation with experimental results. Finally, in a scale-up approach, the developed model is used to study the densification of a large piece and allows pointing out the effects of temperature and stress gradients on microstructural heterogeneities. References [1] M. Abouaf et al., J Theor Appl Mech. (1986) 121–140. [2] E.A. Olevsky, Mater. Sci. Eng. R Rep. 23 (1998) 41–100.

225 Microstructure and mechanical properties of Fe-Mn-Al-C low density steels

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In recent years, research work has been focused on high performance steels to reduce the thickness of structures of vehicles as to satisfy the need for weight saving. However, the requirement for the stiffness of structures cannot be guaranteed when the thickness of a structure is thinned to a certain extent. Therefore, reducing the density of steels by adding the light alloying elements has become one of the research trends in the development of high performance steels. In the present paper, the microstructures and mechanical behavior of Fe-Mn-Al-C steels with different Al, Mn and C concentrations were investigated. It was revealed that the variation in Al concentration could make a marked influence on stacking fault energy of the materials and in turns the mechanical behavior in Fe-26Mn-xAl-1C steels with Al changing from 3 to 11(wt%). The addition of C concentration increasing from 0.3 to 1.2(wt%) resulted in changes in the phase constituents and ordered phases in Fe-18Mn-10Al-xC steel system. This work demonstrated that the high strength and superior ductility can be achieved by choosing appropriate alloying elements and thermo-mechanical schedules. The detailed microstructure evolution has been characterized by OM, SEM and TEM and the correlation between the microstructures and mechanical behavior of different Fe-Mn-Al-C steels have been investigated. Key words: Fe-Mn-Al-C, low density, microstructure, mechanical property.

226 Pressure Tuned Insulator-Metal Transition in Mott systems

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Insulator-Metal transition is a fundamental topic in solid-state physics, but the underlying mechanism often remains unclear especially for Mott systems. Electron correlation effects can open up a gap in a partially filled band, giving rise to a Mott insulator. Its transition to a metal could be driven by either bandwidth control (BC) or filling control (FC). Recently, we have applied synchrotron x-ray spectroscopic methods (such as XAS, XRS, and RIXS) to study insulator-metal transition using pressure as a tuning parameter (BC) in four materials: a correlated metal V₂O₃, 3d Mott insulators PbCrO₃ and SmNiO₃, as well as a 5d spin-orbit induced Mott insulator Sr₃Ir₂O₇. Our results demonstrate that while pressure can be a clean, effective, and theoretically transparent method to change the ratio of electron interaction over

bandwidth U/W, its effects are still complicated owing to the interplay of charge, orbital, spin, and lattice degrees of freedom in Mott systems.

227 Microstructural banding in medium carbon steel

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Alternating bands of different microstructures aligned parallel to the rolling direction often observed in medium carbon steel, affect the directional homogeneity of the mechanical properties. We studied experimentally, the progressive rotation of primary and secondary dendrite arms as well as the associated inter-dendritic regions by hot-rolling in a laboratory-scale rolling mill in addition to modelling the rotation of primary and secondary dendrite arms and observing the microstructural band formation microscopically. Primary dendrite arms rotated progressively around the transverse axis during hot-rolling while the secondary dendrite arms, with their adjacent areas of inter-dendritic segregation, elongated progressively parallel to the rolling direction. At rolling reductions in excess of 80%, the primary and secondary dendrite arms, with their associated inter-dendritic regions, aligned parallel to the rolling direction, thereby forming layers of low and high concentration of alloying elements respectively. Ferrite/pearlite band formation could clearly be observed in-situ by the use of high-temperature laser-scanning confocal microscopy. A two-dimensional symmetric finite element model was developed to simulate the behaviour of the dendritic and inter-dendritic regions respectively during hot-rolling with a view to determining the microstructural changes leading to micro-chemical banding. Primary dendrite arms rotated about the rolling direction while secondary dendrite arms experienced larger elongations than the segregated regions within strain bands. Good agreement was found with experimental observations. The current study provided new insights with respect to the origin and alignment of bands of different chemical composition, which eventually results in the formation of microstructural banding.

228 Design of solution heat treatments for low-cost single crystal Ni superalloy

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Solution heat treatment is one of the most effective methods to eliminate coring and micro-segregation. The objective of the present research was to study varying heat treatments on micro segregation and creep properties of low-cost SX superalloys. In order to investigate the effect of heat treatment on microstructural uniformity, traditional stepwise heat treatment (SH) and continuous heating heat treatment (CH) were designed. Both Heat treatment conditions (temperature and holding time) were determined by DSC results. After heat treatment, eutectic γ/γ' regions dissolves into the matrix, and partition ratio values of Re, W and Ti were clearly decreased. Partition ratio value difference between SH treated specimen and CH treated specimen leads difference of creep life time. Due to uniformity of partition elements, a new solution heat treatment improves high temperature mechanical properties.

229 From single crystals to textured Mg alloys: Acoustic emission study

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In order to obtain a comprehensive set of acoustic emission (AE) data, magnesium single crystals with various crystallographic orientations were uniaxially compressed at room temperature (RT) and at a constant strain rate of 10^{-3} s^{-1} . Loading along the $\langle 11.2 \rangle$ axis led to a preferable activation of basal slip and it was accompanied by a low amplitude AE signal. Twinning was observed exclusively during compression along the $\langle 10.0 \rangle$ and $\langle 11.0 \rangle$ axis. The twin nucleation was associated with burst AE signals with high amplitudes. The results were applied in studies of twinning and dislocation processes in polycrystalline textured Mg alloys. The twinning activity was easily revealed by a strong AE response. A detailed insight into microstructural changes was provided by X-ray and electron back scattered diffraction (EBSD) techniques.

230 Scaffolds applicable as implants of a loss of palate fragments

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In the framework of research innovative porous biomimetic materials called scaffolds with the well-defined regular structure of open pores have been used. Virtual implant models have been designed by the use of the software such as CAMD (Computer Aided Materials Design). They have the geometrical dimensions corresponding to a fragment of a loss of a human palate. Losses of the type have been a result of a mechanical injury or a tumor. Designed components are to replace the missing bone fragments mainly a maxillofacial bone in an implant treatment process. Porous and regular structure with defined geometric dimensions and shape are designed in the form of the unit cell, which has then been subjected to the multiplication process. The task has been realised by the use of specialized 3D software, Marcarm Engineering AutoFab. A designed unit cell is of strict geometric dimensions and a shape. It is to ensure the appropriate physicochemical properties of the produced object. The designed virtual model of scaffolds have been produced in a process of selective laser melting (SLM). For their preparation titanium alloy powder - Ti6Al4V of suitable granulation and shape has been used. Thus obtained scaffolds have been observed in a scanning electron microscope. The structure of the pores is compatible with the shape of a designed unit cell. The outcarried EDS analysis has confirmed the chemical composition of the tested material.

231 Thermocyclic high temperature oxidation of intermetallic TiAl alloys and their protection by fluorine

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Intermetallic TiAl alloys suffer from accelerated attack by the environment at elevated temperatures. Their oxidation behaviour is characterized by non-protective oxide scale formation. These scales show severe spallation especially under thermocyclic conditions which can lead to a substantial material loss of the components. The improvement of the oxidation resistance is hence a critical factor before any high temperature application of TiAl alloys could be possible. Fluorine surface treatment with low amounts of fluorine i.e. micro alloying can improve the oxidation resistance of this class of intermetallics drastically, so called fluorine effect. Several fluorine treatments have been developed and can be applied according to the later use. Results of isothermal and thermocyclic high temperature exposure tests of several untreated and treated alloys will be presented and compared in this paper. Post experimental metallographic investigations reveal the formed oxide scales so that the effect can be rated. Finally the results will be discussed in view of a technical application of TiAl alloys.

232 Joining of aluminum alloy to galvanized and uncoated steels

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Dissimilar metal joint between aluminum alloy and steel has broad application prospects in automotive, shipbuilding and pressure vessel industries. However, it is still a great challenge to weld aluminum alloy to steel because of the large differences in thermo-physical properties between these two materials, e.g. melting point, thermal conductivity, linear expansion coefficient, and the easy formation of brittle Fe-Al intermetallic compound and large residual stresses after welding, which will significantly deteriorate the microstructure and mechanical properties of the resultant joint. Friction stir spot welding (FSSW), gas tungsten arc welding (GTAW), and gas metal arc welding (GMAW) techniques were carried out to join aluminum alloy to galvanized steel and uncoated steel. FSSW can attain spot weld between aluminum alloy and (galvanized and uncoated) steel with high strength over 3800N. GTAW technology can deliberately control the welding heat input and then suppress the thickness and distribution of intermetallic compounds and reduce the residual stress with post-weld heat treatment. Consequently, the microstructure and mechanical properties of the resultant joint was remarkably improved with failure occurring in heat-affected zone during tensile test. GMAW technology can enhance efficiency with higher welding speed than GTAW while welding aluminum alloy to galvanized steel. However, GMAW was restricted by the filler wire while welding aluminum alloy to uncoated steel. Detachment (spalling) of interfacial layer was detected in aluminum alloy/steel dissimilar metal joint both by GTAW and GMAW processes.

233 In vitro degradation behavior of AZ31 Mg alloy as biomaterial in Hank's solution

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Combined with immersion and hydrogen collection, the potentiodynamic and potentiostatic polarizations, electrochemical impedance spectroscopy (EIS), Scanning electron microscope (SEM), X-ray photoelectric spectroscopy (XPS) and X-ray diffraction techniques, the corrosion behavior of AZ31 alloy as a biomaterial in Hank's solution were deeply investigated. SEM observations and XPS analysis demonstrated that a layer of protective film could be easily formed on the sample surfaces, leading to a sharply decrease in corrosion rate at the initial stage. After it was maintained at a very slow level for about 20 hours, the film began to degenerate and the corrosion rate was accelerated. Moreover, the potentiostatic polarizations showed that hydrogen evolved in the cathodic polarization process while magnesium hydride could not simultaneously occur. It is ascribed to the cathodic reduction of glucose contained in Hank's solution. In the anodic potentiostatic polarization, the anodic dissolution of AZ31 magnesium was gradually increased while the negative difference effect (NDE) was gradually decreased with time. Besides, hydroxyapatite particles were found on the film layer, indicating that the alloy could have the good biocompatibility.

234 Mechanical behaviour of materials during creep with changing loads

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Metal components during service experience deformation conditions that change continuously with time. Since service conditions are usually quite difficult and expensive to reproduce in laboratory, the alloys mechanical behaviour during creep in service have to be extrapolated from creep tests at constant loads and temperatures. Empirical laws have been proposed to forecast the effects of complex service conditions, like the life fraction (LF) rule to foresee the rupture times, and the strain hardening (SH) rule to predict the accumulation of creep strain with changing stress and temperature [1]. Two directionally solidified (DS) nickel based super-alloys have been investigated with creep tests at constant loads and variable loads. These alloys are characterised by a negligible primary, a minimum of strain rate with no secondary, and a dominant tertiary caused by dislocation multiplication, called accelerating creep [2-4]. The damage mechanisms causing the final rupture appears only in the very last percentage of life. The reported simulation results show that the physical-sounded model used to describe the accelerating creep can predict successfully the times to rupture and the creep curves of the two DS alloys with changing loads, better than the empirical LF and SH rules.

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235 TEM observation of the evolution of the microstructure during aging of a betametastable titanium alloy

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The most recent aircraft programs such as the A350 and A320NEO reserve an increasing share in the use of titanium alloys for structural applications. This is particularly the case, but not only, at the pylon, for which the research of improved performance leads to the use of these alloys in more severe temperature conditions. This approach induces a sustainability assurance need of aeronautical Ti-made structures, in particular as regards to the mechanical characteristics of the alloys used. The presented work is focused on the TEM observation of the evolution of the microstructure of a beta-metastable Ti17 alloy as a function of temperature and time of aging. Samples have been aged at different temperatures and different aging time, then observed using conventional post-mortem TEM experiments. Emphasize is made on the morphological evolution of the alpha phase and the competitive process of length increasing and intersections of alpha-lamellae. The formation after long-time aging of omega-phase, which has the drawback of increasing brittleness, is also evidenced.

236 Simulation of the residual stress field in air-quenched turbine disks

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Inconel 718 is a nickel-based superalloy used in high temperature, high-strength applications like aero engines. The alloy has superior high temperature strength combined with a good corrosion resistance. Turbine disks from Inconel 718 are forged and heat treated to produce the desired geometry and material properties. Between the forging and the two-step heat treatment process the disks are air-cooled. Cooling generates a residual stress field in the disk, which has an influence on the lifetime of the disks and thus on the service intervals of the aero engines. The main aim of the present study is to simulate the residual stress field in air-cooled turbine disks from Inconel 718 and to validate the results with neutron diffraction measurements.

237 Low cycle fatigue behaviour of a high interstitial cast TRIP steel

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TRIP (TRansformation Induced Plasticity) steels exhibit an outstanding combination of strength and ductility due to the capability of forming alpha'-martensite under loading. These mechanical properties in addition to the excellent corrosion resistance make them the material of choice for a wide field of applications. Regarding the fatigue behaviour of TRIP steel, it is

generally agreed that a certain strain amplitude as well as a threshold of the cumulated plastic strain have to be exceeded to trigger the martensitic phase transformation.

This study presents the Low Cycle Fatigue behaviour of a high interstitial cast TRIP steel (18.7Cr-2.94Mn-4.22Ni-0.15C-0.18N) compared to a TRIP steel with low contents of nitrogen and carbon, including the mechanical properties, α' -martensite evolution, fatigue life estimation and investigations of the microstructure of selected fatigued specimens using a scanning electron microscope. After a heat treatment at 1150 °C for 30 minutes followed by water quenching, total strain controlled LCF tests with strain amplitudes in the range of 0,2 % to 1,2 % have been conducted at room temperature. Concomitant, the fatigue induced martensitic phase transformation was measured in situ by a ferrite sensor.

As expected, the steel exhibits high cyclic stresses and a martensitic phase transformation for all strain amplitudes equal or above 0.3 %. With increasing strain amplitudes the incubation period for the phase transformation gets shorter and the amount of α' -martensite increases essentially. Moreover, the cyclic stress strain curves show a secondary hardening whose onset and intensity can be correlated with the martensitic phase transformation.

238 Deformation behavior of extruded ZN11 magnesium plate

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Deformation behavior of ZN11 magnesium alloy in a form of extruded profile has been investigated with respect to different loading directions. The samples were compressed at room temperature with a constant strain rate of 10^{-3}s^{-1} along extrusion (ED), transversal (TD) and normal direction (ND). X-ray diffraction technique was employed to follow the development of texture during loading. The twinning activity was studied by the subsequent analysis of microstructure using scanning electron microscopy (BSD, EBSD). The deformation behavior of the extruded profile was also investigated by the acoustic emission (AE) technique, where the AE signal analysis correlates the microstructure and the stress-time curves to the active deformation mechanisms. Compression along the ND (i.e. compression perpendicular to the basal planes) is not favorable for twinning, while during compression along the ED and TD twinning activity is observed.

239 Quantitative transmission electron microscopy studies on deformation mechanisms in nanotwinned copper

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Materials with nanometer-scale twins have attracted intensive research interest, attributed to their potential for achieving simultaneous high strength and good ductility. The mechanical properties are associated with interactions between lattice dislocations and the coherent twin boundaries (TBs), as TBs serve as strong barriers for the motion of dislocations. Accordingly, the strength of nanotwinned metals supposedly increases with decreasing twin lamella spacing. Nevertheless, when twin lamella spacing decreases below a critical thickness, approximate 15 nm for nanotwinned copper, a softening behavior instead of strengthening prevails for the yield

strength. Molecular dynamics simulations suggested that the softening corresponds to the dominant activity of Soft mode dislocations below a critical twin lamella spacing, where the dislocations nucleate from the twin boundary/grain boundary intersections. Hence, quantitative electron microscopy investigations are desirable to resolve the actual deformation mechanisms in nanotwinned metals and their relationship with the mechanical properties. In this work, from the quantitative analysis of local stress, the statistics of dislocation activities by in situ TEM and the result from mechanical testing, a direct relationship can be established between dislocation activities and the strength of nanotwinned metals. The stress states measured from high-resolution TEM reveal that local stress concentrations vary significantly with the twin lamella spacing, thus a critical twin lamella spacing is determined for the transition from strengthening to softening of nanotwinned copper. The result agrees quantitatively with the experimentally measured critical spacing.

240 Microstructure and texture evolution in nickel during accumulative roll bonding

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Microstructure and texture evolution of commercially pure Ni processed by accumulative roll-bonding (ARB) up to eight cycles were studied using electron back scattered diffraction (EBSD). During ARB, the original, coarse equiaxed grains gradually evolved into lamellar grains along the rolling direction (RD) with refined sizes, with shear bands frequently forming after three cycles. The fraction of low angle grain boundaries (LAGB) increased dramatically after the first cycle as a result of orientation spreading in the original grains whose interior orientations were almost homogenous. However, their fraction decreased with the development of high angle grain boundaries (HAGB) during subsequent deformation, until saturation was reached after six cycles. Overall, the typical deformation texture components (S, Copper and Brass) were enhanced after six cycles of ARB; among them Copper was further strengthened thereafter. On the other hand, Cube component experienced significant and continuous decrease with increasing number of ARB cycles. In addition, the Goss component increased after the first cycle, followed by continuous decrease thereafter. No evidence of any variations in texture through the thickness was observed at lower ARB cycles. However, a higher concentration of Copper component was found near sample surface when compared to the quarter or middle thickness of the sample at higher cycles. It is proposed that the frictional shear introduced near the sample surface facilitated the formation of the Copper component.

241 An experimental investigation of the microforming process of high-purity thin metallic sheets

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Miniaturization of small metallic systems can lead to a softening of the mechanical behavior due to the reduction of scale. Size effects have been considerably studied recently for materials with various crystallographic structures loading in tension. Under tensile conditions, thin metals exhibits softer mechanical properties if the number of grains across thickness is lower than a

critical value. Previous works showed that this parameter depends on the temperature and on the stacking fault energy and that modification appears for a critical strain level. Stamping tests were performed on five hundred micrometers in thickness sheets of hexagonal closed-packed cobalt and face centered cubic copper and nickel. The influence of thickness t over grain size d ratio was studied for several proportional loadings linked to forming processes. Complex loadings were applied with 20 mm hemispherical punch and strain paths were checked with a 3D video extensometer. Hill criterion was systematically used to take into account the anisotropy of samples. Our results revealed that the critical strain level from which size effects appears is strongly sensitive to the stress triaxiality which in turn is closely dependent to the stress path. Complementary in-situ thickness reduction measurements were performed and results agree with the critical number of grains across thickness. The optimal conditions for microforming of thin sheets can be conveniently depicted thanks to original deformation plots taking into account the size effects.

242 The microstructural criterion for creep strength breakdown in a 10% Cr martensitic steel

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A 10%Cr martensitic steel with 3%Co and 0.008%B tempered at 770°C exhibits no creep strength breakdown at a temperature of 650°C up to an extremely long rupture time of approximately 40000 h under an applied stress of 120 MPa. The minimum creep rate was reached to $3 \times 10^{-11} \text{ s}^{-1}$. Microstructural characterization showed that superior creep resistance associated with a high stability of tempered martensite lath structure. Boundary $M_{23}(C \times B)_6$ phase is highly stable against coarsening under long-term aging and creep conditions. These particles remain their orientation relationship with ferritic matrix unchanged under creep at a temperature of 650°C. As a result, no migration of lath boundaries and their transformation to subboundaries diminishing the long-range elastic stress fields take place. The role of $M(C,N)$ carbonitrides in achieving extraordinary high creep strength is also considered.

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243 SPD processed materials mechanical properties determination with the use of miniature specimens

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The main reason why new technologies and treatment procedure are being developed is to attain special mechanical properties. However, these developments are nowadays done on a small material volume either using some laboratory simulators, applying severe plastic deformation procedures or chemical composition screening for multicomponent alloys development by laser or electron beam melting. In all these application a small volume of the material assessed is available and standard procedures for crucial mechanical properties determinations are not applicable. Thus small size techniques should be applied.

There has been extensively used small punch test technique (SPT) for those cases in recent years. This technique is mainly based on the evaluation using correlation between standard and

SPT tests for considered material. In cases when insufficient material volume is available, those correlations cannot be established and thus comparative evaluation only can be carried out. This kind of evaluation is insufficient for the contemporary purposes, when full material potential is to be utilized. Therefore, procedures providing results directly comparable with standard specimens are being developed. Fundamental properties are those determined from tensile tests. The current paper is presenting application of developed miniature tensile test specimen method to materials after SPD processes. Quasi static as well as dynamic properties determination is shown here for Titanium and Magnesium alloys for several SPD techniques. The results obtained from testing can be used not only for a direct material properties assessment and comparison, but also as input data for FEM codes, significantly increasing the materials considered application potential assessment.

244 Nanoindentation studies of inhomogeneities in high pressure torsion deformed bulk metallic glasses

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Bulk metallic glasses (BMG) have gained in interest because of their promising properties such as high strength and high elastic limit, useful for potential application as structural materials. Their huge drawback is their intrinsic brittleness. One promising treatment to improve their ductility is high pressure torsion (HPT) deformation. In this work a $\text{Cu}_{45}\text{Zr}_{45}\text{Al}_5\text{Ag}_5$ BMG alloy produced by copper mold suction casting was subjected to HPT deformation. Deformations were performed using different parameters and temperatures. On cross-sections nanoindentation measurements over the whole disc radius were performed, using a large number of indents to map local variations in hardness and young's modulus. As reference an undeformed BMG specimen was measured using the same indentation parameters. HPT deformed specimens showed a reduced average hardness and Young's modulus. Local analysis of the values obtained from the indents revealed a strongly inhomogeneous deformation. This shows up as strong gradient of both hardness and Young's modulus over the sample height and an increase in deformed volume with increasing distance from the center. The difference between these regions are found to be up to 20% in hardness and 10% in Young's modulus. To exclude effects by material pile-up, AFM measurements of various indents were performed. The pile-up area was found to be independent of the indent positions, therefore only changing the absolute hardness values and not the relative ones. The authors acknowledge financial support by the Austrian Science Fund FWF: [I1309]

245 Formation of new metastable phases and intermetallics in magnesium-based systems by high-pressure torsion

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Mg-based binary systems are interesting systems because of their high strength to weight ratio as well as their capability for reversible solid-state hydrogen storage. In this study, Mg powders were mixed with 21 different elements using high-pressure torsion (HPT) method and subsequently evaluated by different structural and microstructural analyses. In 70% of the selected systems, either nanostructured intermetallics, amorphous phases or new metastable phases were formed after HPT or post-HPT heat treatment. The current study shows the potential of HPT method in synthesizing Mg-based compounds, which their fabrication is generally difficult by melting because of low boiling temperature of Mg.

246 Potential for improved machinability in carbon steels via graphitisation

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Alloying with Si and Al has been found effective in reducing the annealing times required to exchange cementite for graphite in carbon steel, thereby identifying a potential commercially-feasible route to improved machinability and cold workability by reducing or eliminating the need for special alloying additions which can create processing or re-cycling difficulties. Metallographic techniques have revealed structural information on the formation of graphite nodules, on the accompanying dissolution of cementite phase and on lubrication by graphite during machining. Emphasis is placed upon the stability and dissolution of cementite during annealing, on which it is suspected that graphite phase can nucleate, and evidence can be provided to support this hypothesis. In consequence, the microstructure prior to annealing can influence the eventual graphite dispersion and hence the machinability characteristics which have been measured and compared with commercial free-cutting steel grades.

247 Exploring materials through time-resolved X-ray powder diffraction experiment during fast compression

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Pressure is a powerful variable to induced changes in the atomic arrangement and microstructure. These induced changes in the material are not only a function of the amount of pressure but also of the rate the pressure is applied. The experimental techniques covering the

two extremes of pressure generation are static compression with typical strain rates of less than 10^{-3} s^{-1} and shock compression with strain rates larger than 10^5 s^{-1} . The pressure temperature space between these two extremes is up to now largely unexplored. The recent developments in high-energy X-ray synchrotron sources, fast large-area X-ray detectors, and the advances in fast-pressure generation, allow now in situ X-ray diffraction experiments with a sufficient time resolution to follow the changes in the atomic and micro-structure in the strain rate regime between shock and static compression. We will present the results of fast compression experiments on a number of Earth materials.

248 Study of austenite grain growth of micro-alloyed steels by using metallography and EBSD analysis

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Knowing the evolution of the austenitic grain size distribution is decisive for the development and specific adjustment of steel properties. The austenite structure is passed on to the final microstructure and determines essentially the product characteristics. Therefore prior austenite grains were determined for different micro-alloyed steels, combining classical metallography and reconstruction based on EBSD data. Different microstructures were produced by varying annealing temperatures and times. After annealing the specimens were water-quenched. Generally, the determination of the prior austenitic structure, including the grain size distribution is a challenging task. Three different methods were applied: Chemical etching, thermal etching and EBSD measurements. The EBSD measurements were optimized for the subsequent reconstruction of the austenite grains. All methods were compared in respect to their applicability, reliability and accuracy.

249 Imprinting strain in nanostructured ferroelectric ceramics using spark plasma sintering: New strategy towards properties control

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A challenge in nanomaterials is to overcome the large density of chemical, structural and charged defects precluding the transfer of bulk properties to nanomaterials. Ferroelectric materials which display the highest dielectric permittivities cumulate these drawbacks because of the long scale correlation of their lattice features. Dense compacts are requested for most of the applications of these materials in electronics, telecoms, sensing, actuating... and the required high temperature densification step is enhancing the effect of interfacial defects on the overall ceramic properties. Ferroelectric ceramics such as $(\text{Ba,Sr})\text{TiO}_3$ are massively used in the capacitor industry but still suffer from limitations as, below a critical grain size, a decrease of the tetragonal distortion and thus a detrimental stabilization of the cubic phase is observed. This is described through the so-called “core-shell” model relating nanoscale ferroelectric particles built from a ferroelectric core surrounded by a non-ferroelectric shell

which is the disturbing unit. We emphasize here the advantages of combining supercritical fluid synthesis and Spark Plasma Sintering (SPS) to produce functional nanostructured ceramics (grain size < 50nm). The supercritical fluid synthesis offers a continuous, versatile and fast route towards well-crystallized oxide nanoparticles with an accurate control of size and chemistry. Using SPS process under air and applying uniaxial pressures of several hundred of MPa, we were able to imprint very large strains in dense nanostructured ceramics and to restore the bulk ferroelectric transition temperature. These results open new insights to explore the pressure effect during SPS process on the intrinsic properties of polycrystalline nanomaterials.

250 Physical simulation of thermo-mechanical processing of ferritic-bainitic dual phase (FBDP) steel

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The present work is dealing with a physical simulation of thermo-mechanical processing of ferritic-bainitic dual phase (FBDP) steel alloy containing 0.1% C, 0.3% Si, 0.9% Mn and 0.7% Cr. The microstructure changes and allotropic transformations during thermo-mechanical simulation are investigated. A series of heating – cooling cycles to detect the critical and allotropic transformation temperature by dilatation were carried out on the thermo-mechanical simulator (Gleeble 3500). On the other hand, five – consecutive hits were used during the physical simulation of hot rolling process. Two hits were representing the roughening stage followed by three ones representing finish rolling. Holding at 500°C for 5, 7, 10, 12 and 15 min. after last hit has been applied and then followed by air cooling. Dilation curves appear that $Ac_1 = 766\text{ }^{\circ}\text{C}$, while Ac_3 was detected as $883\text{ }^{\circ}\text{C}$. Bainitic allotropic transformation temperatures were clearly noticed as $618\text{ }^{\circ}\text{C}$ for Bs and $542\text{ }^{\circ}\text{C}$ for Bf. The recrystallization temperature was also detected as $1035\text{ }^{\circ}\text{C}$. Holding for 5-7 min. at $500\text{ }^{\circ}\text{C}$ was concluded as the optimum for creation a bainite volume fraction. Rough hot deformation a higher temperature above the recrystallization temperature is essential, where no strain hardening and possibility for achieving high strains without excessive loads. Finishing deformation at temperature lower than Tr would create fine bainitic structure. The flow curve of the steel ensures continuous strain hardening. The strain hardening rate (σ_f/ϵ) was directly proportional to temperature difference from pass to pass.

251 Evolution of microstructure, phase composition and hardness in 316L stainless steel processed by high-pressure torsion

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The 316L stainless steel (main components: 69% Fe, 17% Cr, 10% Ni) is widely used in many industrial applications due to its high corrosion resistance, strength and ductility. In the present study, the mechanical performance of 316L stainless steel was improved by severe plastic deformation (SPD). The samples were processed by High Pressure Torsion (HPT) operating under quasi-constrained conditions with an applied pressure of 6.0 GPa and numbers of turns

between $\frac{1}{4}$ and 10, at room temperature. The evolution of the microstructure and the phase composition during HPT were investigated by transmission electron microscopy (TEM), scanning electron microscopy (SEM) and electron backscatter diffraction (EBSD). The crystallite size, the dislocation density and the phase composition were examined by X-ray diffraction (XRD). The mechanical behavior of the specimens was studied by hardness measurements as a function of the distance from the center of the disks processed by HPT. It was found that during HPT simultaneous grain refinement and phase transformation occurred, yielding very high hardness in the SPD-processed 316L steel. A flow pattern was developed due to HPT which consists of regions with different phase compositions and grain sizes. The correlation between the microstructure and the hardness was investigated in detail.

252 Influence of cross section on the parameters for linear friction welding of high-strength chains

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In respect of weld quality, weld strength and the heat affected zone, linear friction welding has significant advantages compared to flash-butt and resistant welding. The production of chain links is realized by one c-shaped part in reciprocating motion being moved towards a stationary one under applied forces. The friction at the contact faces causes heat and thus, the material is plastically deformed. Due to relative movement excessive material is pressed out of the joint area leading to a characteristic weld burr. Relative motion is stopped once the defined burn-off is reached and by applying the forge force the chain is welded.

In this paper, welding parameters for three different cross sections are investigated. Based on successful welding trials on 30CrNiMo8 steel chains with 26 mm diameter, parameters are adapted for smaller cross sections. On 48 steel specimens with 7, 10 and 15 mm diameter systematic welding trials are performed. The influence of the welding parameters frequency, amplitude, forge force and burn-off on welding strength and welding time are investigated with and without application of post weld heat treatment. This test series was evaluated using DoE.

253 Texture evolution in multi-phase TNM sheet materials measured by means of high-energy X-ray diffraction

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As intermetallic gamma-TiAl based alloys, the beta-solidifying TNM alloys with a nominal composition of Ti-43.5Al-4Nb-1Mo-0.1B (in at.%) belong to a class of innovative lightweight high-temperature materials. Their outstanding characteristic is their remarkable processing capability, which is due to a high amount of disordered beta-phase present at hot working temperatures. Utilising this advantage, as well as the homogeneous and fine-grained microstructure originating from the solidification via the beta-phase, a TiAl sheet processing technology has been developed on lab scale, in which cast and hot-isostatically pressed feedstocks are hot-rolled without the need of a preceding ingot breakdown. Depending on the rolling temperature, which determines the phases present during hot deformation, different

microstructural features and textures can be created. In the present work, diffraction experiments using synchrotron radiation were performed to illuminate the understanding of the interplay of the various phases involved in the hot deformation, and, therefore, of the evolution of textural components resulting from hot rolling as well as during a subsequent heat treatment. High-energy synchrotron radiation was applied in transmission geometry to record the pole figures of all phases within the advanced multi-phase material with high accuracy and, concurrently, within a decent time range.

254 Determination of crystallographic orientation near a chill zone using ghost lines

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Many crystals nucleate on the mold surface when the molten alloy is poured in a mold cavity. Because the crystallographic orientations of these crystals are random, the solidified structure near the mold surface is very complex. The ghost lines, which are sometimes thick and the angle between them is not 90 degrees, are often observed in this region. However, if the crystallographic structure of this alloy is cubic, such as bcc or fcc, the ghost lines are very regular. In order to understand the geometry of ghost lines, Al-20 mass%Cu alloys were unidirectionally solidified with constant growth velocity. The solidified structures on the obliquely crossed section were observed. The ghost lines were quite regular and parallel to each other in a solidification grain. The angles and the ratio of the width of ghost lines were measured and crystallographic orientations were estimated using these parameters, based on the solid analytical geometry. EBSD analysis were also performed on the area, where the ghost lines were characterized, and the precise crystallographic orientations were decided. The comparison between both analytical values indicated that the differences between them are within 10 degrees and it can be safely concluded that the estimation for crystallographic orientation using ghost lines agreed well with the EBSD analysis.

255 Effect of the compositional variations on the early-stage precipitation hardening in Al-Mg-Si(-Cu) alloys

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Al-Mg-Si(-Cu) alloys are among the most widely used and currently investigated alloys for automotive industry's strive to achieve significant lightweighting in structural components of passenger cars and trucks. A great challenge is however to further advance the state of knowledge in order to open new opportunities for alloy and process development and optimization. Here, we report a new understanding on the complex early-stage precipitation hardening phenomena in these alloys through a multi-length scale experimental investigation. The effects of the Mg/Si ratio and the Cu content, and their relative significance, on the nucleation phenomena and the kinetics of precipitation hardening will be presented.

256 Numerical simulation of laser beam welded steel-aluminum joints

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Nowadays the reduction of metal structure weight has a crucial role. It can be achieved through the application of lightweight materials and their different combinations. Especially combinations of aluminum and steel are interesting for modern lightweight design concepts. Recent developments for laser beam welding between steel and aluminum structures are based on the partial mixing between both materials. Numerical simulations of those welded joints can help to control the mixing between different metals through the prediction of the penetration depth and thereby to increase mechanical properties.

Known microstructure transformation models focus so far on the thermo mechanical properties in a heat affected zone, however these models are not yet qualified to take into consideration complex processes taking place in a weld during dissimilar metal joining. In particular, the impact of the heat input on the weld geometry, the Al-Steel mixing and the thermo-mechanical properties is of interest. Thus cannot be taken into account in conventional simulation models. To expand these known approaches for temperature field calculation, structural modeling, distortion and residual stress estimation an appropriate numerical method and extensive material data are necessary.

In this study experimental information from welding experiments are used for the selection of appropriate heat source model and its calibration as well as for the defining of correct boundary conditions. By means of the validated FE model the precise welding penetration can be predicted as function of welding parameters such as power and joining speed.

257 Innovative route for elaborating metal/metal composite

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A new route for elaborating metal/metal composites with a given 3D arrangement is proposed. A combination of an additive manufacturing technique (Electron Beam Melting) and sintering (by Spark Plasma Sintering) is used to generate a microstructural composite made of TA6V. A lattice structure made of TA6V is first fabricated by additive layer manufacturing (EBM). The lattice structure is then filled with TA6V powder before spark plasma sintering. A bulk specimen is thus obtained with two different microstructures: one inherited from the solidification during the EBM process and the other one resulting from sintering. These microstructures are revealed by microscopy (optical and EBSD) and lead to a heterogeneous distribution of the mechanical properties. This innovative route definitely opens new avenues to achieve complex microstructural composites. Indeed, different microstructures between the lattice and the matrix can be generated by applying a pre-heat treatment on the lattice or by milling the powder before sintering. In the first case, a composite exhibiting a coarse acicular microstructure in the lattice and a fine one in the matrix is observed. Milling the powder leads to an acicular microstructure in the lattice combined with equiaxed structure in the matrix. This new process is very general and can be used for elaborating metal/metal composites made of

different materials.

258 Hydrogen-induced decomposition of Cu-Zr binary amorphous alloys

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Binary amorphous Cu-Zr alloys which are homogeneous in atomic scale decomposed into Cu-rich and Zr-rich clusters by the introduction of hydrogen into the structure due to the significant difference in the affinity to hydrogen. (i) On surface, by the involvement of oxygen, metallic Cu was precipitated in a relatively large scale, order of micron, as a consequence of Zr-oxide formation. (ii) On the other hand, the inside of the alloy was decomposed into Zr-hydride and metallic Cu in nanometer scale. Such precipitations on surface and inside having different scales showed the obvious bi-modal profile in SANS measurement; the first shoulder corresponds to micron-order decomposition on surface and the second shoulder to nanometer-sized decomposition inside. The phase separation occurring both inside and on surface ranging from nanometers to microns was confirmed by the combined characterization using XRD, SEM, TEM, and SANS analyses. Hydrogen induced phase separation inside of the alloys in a very uniform and regular manner that SANS showed a peak indicating the segregation is uniform and periodic. Specimen preparation: Binary amorphous ribbons of Cu-Zr system were prepared by a melt-spinning technique at Eco-FM Company (Incheon, Korea) with a width of 75 mm and a thickness of about 80 μm . Hydrogen charging was carried out inside a cylinder equipped with a pressure gauge by applying a hydrogen pressure of 8 bars at the temperatures of 473K, 573K and 673K for 24 hours. With those ribbons, various analyses were carried out to reveal the microstructural change induced by hydrogen absorption.

259 The interface character distribution and intergranular corrosion resistance of duplex stainless steel UNS S32304

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Abstract: The duplex stainless steels (UNS S32304) after solid solution annealing at two different temperature (1573K and 1323K) were subjected to the same cold rolling with $\epsilon=3$ and subsequent annealing at 1323 K for 30min. A fine-grained microstructure with the average grain size of less than 10 μm are introduced in both ferrite and austenite. The ICR of the two specimens as processed were investigated by means of double loop electrochemical potentiodynamic reactivation (DL-EPR) technique in a solution of 1 mol/L H_2SO_4 +1 mol/L HCl +0.2 mol/L NaCl at 30°C with a scanning rate of 1.667 mV/s after age-annealing at 650°C for 2 h. In addition, the morphologies and microstructures of the specimens after electrolytic etching in oxalic acid were characterized by SEM. In order to evaluate the corrosion susceptibility of various interfaces including grain boundaries and phase boundaries, the

corresponding ICD were determined by electron backscatter diffraction (EBSD). The results show that S3 twin boundaries, low angle grain boundaries (LABS) and phase boundaries (PB) having K-S orientation relationship (OR) between the neighboring d and g grains are mostly more resistant. And therefore, the specimen initially solid-solution annealed at 1573°C exhibited a higher resistance to IC due to the larger populations of LABs in ferrite and PBs with K-S OR. Key words: interface character distribution; intergranular corrosion; EBSD; duplex stainless steel.

260 Sequentially layer-by-layer growth of Cu film on patterned Ru/Si substrate

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This study investigates Cu film prepared layer-by-layer on a patterned Ru/Si substrate using an electrochemical atomic layer deposition (ECALD) method. The structure and property of the films were investigated to elucidate their capability as Cu interconnects for microelectronics. Previous researches mainly used a vapor-based atomic layer deposition to form a conformal Cu film. Herein, an entirely wet chemical process was used to fabricate the Cu interconnects through sequential, self-limiting surface reaction combining underpotential deposition (UPD) and surface limited redox replacement (SLRR). The experimental results indicated that Pb deposition potential affected the subsequent SLRR of Cu. The lowest resistivity obtained was 3.65 $\Omega/\text{sq.}$ for the Cu film grown on a 20 μm linewidth patterned underlayer. The self-limiting growth method proposed here offers a layer-by-layer, all-wet deposition capability to fabricate Cu interconnects.

261 Microstructure and mechanical properties of Mg-6Zn-1.4Y alloy prepared by rheo-squeeze casting process

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Mg-Zn-Y alloys containing a thermally stable icosahedral quasicrystal phase (I phase) will have wide application future on condition that the primary α -Mg and the I phase can be refined during the casting process. In this research, the microstructure and mechanical properties of the rheo-squeeze casting Mg-6Zn-1.4Y alloys have been investigated. The Mg alloy melt at temperature of 20~30 $^{\circ}\text{C}$ above the liquidus was exposed to ultrasonic vibration (USV) for 60 s with different power densities from 3W/ml to 12W/ml, and then the slurry was formed by squeeze casting. The effects of different power densities on microstructure and mechanical properties of the alloys have been analyzed. The results show that good semi-solid slurry with fine and spherical α -Mg particles could be obtained, and the primary α -Mg crystals are with average particle size of about 50 μm and average shape coefficient of 0.7 after USV. Meanwhile the original coarse W-Mg₃Zn₂Y₃ phase and I-Mg₃Zn₆Y phase get refined obviously and distributed uniformly. Compared with the conventional gravity casting samples, the tensile strength and elongation of the rheo-squeeze casting samples are significantly improved. The investigation

indicates that the USV is a good method to prepare semi-solid slurry of Mg alloy with fine and spherical primary particles and an effective approach to refine the RE-containing intermetallic compounds.

262 Introduction of a new class of creep resistant engine materials based on the Al-Si-Mn-Mo system: Creep properties and microstructure

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High temperature Al-Si cast alloys are on the way to becoming an appropriate replacement for cast iron in Diesel engines. However, the severe working conditions of the engine parts have put some limits on this trend. As a result of improvements in engine performance and demands for higher pressures and temperatures, the existing aluminum alloys have reached their strength limits. This has triggered efforts in alloy modification to enhance the elevated temperature performance of these alloys. One route towards improving the performance of such alloys is to introduce thermally stable particles, which impede the movement of dislocations at elevated temperatures where age hardening precipitates lose their strengthening effect. These particles enhance the strength and creep resistance with varying degrees of effectiveness depending on their size, distribution, coherency and orientation relationship with the Al matrix. In the present work, a new Al-Si cast alloy has been developed that exhibits significant improvements over the existing alloys used in Diesel applications. These improvements are attributed to the formation of nano-scale dispersoids during solution treatment in the grain interiors. The precipitation of these particles and their effect on mechanical properties at elevated temperatures are studied. The Al-Si-Mo-Mn system is introduced, which offers significant potential for development of elevated temperature cast alloys. This alloy system can be used for lightweighting in automotive industry and promote the wide use of Al-Si alloys in more severe operating conditions such as Diesel engine applications.

263 Microstructural evolution in a 9%Cr-3%Co-3%W-VNb steel during creep

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Microstructural evolution in the 9Cr-3Co-3W-0.2V-0.06Nb-0.05N-0.005B steel crept at $T=650^{\circ}\text{C}$ under an applied stress of 140 MPa up to strains of 1, 3, 5 and 12%, which represent primary, secondary and tertiary creep stages and rupture, respectively, was studied. The steel was initially a solution treated at 1050°C , normalized and tempered at 750°C . After tempering the boundaries of tempered martensite lath structure (TMLS) were decorated by M_{23}C_6 carbides, M_6C carbides and Laves phase particles. The 3% W additive provides the narrow size distribution of the boundary particles excepting M_6C carbides. The depletion of thermodynamically none-equilibrium content of W from the solid solution during creep leads to following events. (i) Continuous precipitation of small Laves phase particles occurs during all creep stages and results in the formation of bimodal size distribution. As a result, the average size of Laves phase particles remains unchanged during creep. (ii) Coarsening of M_{23}C_6

carbides starts to occur only at the onset of tertiary creep. (iii) Transformation of laths to subgrains followed by growth of subgrains is observed during tertiary creep only. The particle density at lath/subgrain boundaries decreases from $5.6 \mu\text{m}^{-1}$ to $2.6 \mu\text{m}^{-1}$ under creep up to rupture. However, no full transformation of TMLS into subgrain structure has been revealed. The reasons for such microstructural changes will be discussed. Acknowledgements
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264 Precipitation in the gradient nanostructured Al-Cu-Mg alloy

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Surface mechanical grinding treatment of a metal can introduce a surface gradient structure with grain sizes ranging from nanometer to micrometer scales. Such a structural diversity also means the diversified distribution of the defects such as dislocations and grain boundaries, which may significantly change the precipitation processes and features of strengthening phases and as a result affect the mechanical properties of the material. In this study, by using high resolution TEM and electron tomography, we investigate a gradient nanostructured Al-Cu-Mg alloy with a focus on crystallographic aspects of an S phase (Al_2CuMg) precipitated in various kinds of grain boundaries. The results will provide new insight into the understanding of relationships between microstructural features and mechanical properties in the precipitation strengthened materials with gradient structures.

265 Tunability of the domain structure of $\text{Pb}_x\text{Sr}_{1-x}\text{TiO}_3$ thin film capacitors and its effect on the dielectric response

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When a ferroelectric material is grown as a thin film, the depolarization field can lead to the formation of 180° domains, as observed for example in $\text{PbTiO}_3/\text{SrTiO}_3$ superlattices [1] as well as in PbTiO_3 thin films [2]. The shape and size of the domains depend on many parameters, such as the film, thickness and the type of electrodes.

In our previous work [3], we reported the effect of a thin SrTiO_3 spacer layer introduced between the electrode and the PbTiO_3 thin film. This spacer allowed the strength of the depolarization field to be controlled inducing a change in size and shape of the domains as well as a modification of the relaxation time of the switched polarization.

In this work we tune the composition of thin films of $\text{Pb}_x\text{Sr}_{1-x}\text{TiO}_3$ from $x=0.2$ to 0.8 . The films are grown by RF off-axis magnetron sputtering using alternating sequential deposition. By varying the composition, one can achieve a gradual tuning of the dielectric constant as well as of the polarization. Dielectric measurements at different temperatures using Nb:SrTiO_3 or LaNiO_3 as bottom electrodes are compared. The dielectric constant is strongly influenced by

the presence of domains and domain walls. X-ray diffraction allowed us to confirm the presence of these domains and piezoelectric force microscopy was used to study their size and shape.

[1] P. Zubko et al, PRL 104, 187601 (2010) [2] S.K. Streiffer, et al, PRL 89, 067601 (2002)

[3] C. Lichtensteiger et al, NanoLetters 14(8) 42025 (2014)

266 An overview on perlite-metal syntactic foam

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Perlite-metal syntactic foam is manufactured by combining expanded volcanic perlite with an aluminium matrix. The formed composite has a high porosity with typical values in the range of 60-70%. As a result the material exhibits the characteristic properties of metallic foam, i.e. it is an excellent energy absorber, exhibits a high specific stiffness and strength, has versatile thermal properties and is suitable for the damping of structural vibrations.

A major inhibitor for the widespread use of metallic foams has been their relatively high manufacturing cost and/or lack of reliable mechanical properties. Perlite-metal syntactic foams are cost-efficient because they use low cost raw materials in combination with a simple and versatile manufacturing procedure. Furthermore, the utilization of expanded perlite particles allows a close control of the geometry of the load-bearing aluminium phase. This manifests itself in a low scattering of important mechanical properties such as the effective Young's modulus or 0.2% offset yield stress.

This work provides an overview on the basic properties of perlite-metal syntactic foam and showcases its application on the example of advanced roadside barrier systems.

267 Technical challenges in narrow-gap root pass welding during hybrid laser-arc welding of 25-mm thick martensitic stainless steel plates

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Keywords: Hybrid laser-arc welding (HLAW); martensitic stainless steel; thick plate; narrow gap groove; microhardness. **Abstract.** As part of a collaborative program to develop advanced manufacturing processes for next-generation hydraulic turbine runners, this study investigated the technological challenges for joining 25-mm thick martensitic stainless steel plates using hybrid laser-arc welding (HLAW). Although candidate materials for the intended application typically include wrought AISI 415 and cast CA6NM, martensitic grade 410 stainless steel was especially selected in this study due to its greater sensitivity to cold cracking. A narrow gap groove was designed to minimize the amount of 410NiMo weld metal required to fill the groove using a multi-pass single-sided welding technique. All the welding trials were performed using a 5 kW fiber laser at its maximum power. The root-pass quality was characterized in terms of weld bead geometry, defects, microstructure and hardness. The main technical challenges for the root pass during HLAW were lack of penetration, lack of fusion, and centerline cracking. The main reasons for the appearance of these defects were investigated, as detailed in this paper. The microstructure and microindentation hardness in as-welded conditions are also presented.

268 Cinematographic observation of GTAW arc and weld pool surface phenomena in the presence of Marangoni convection

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Keywords: GTAW weld pool; metal vapor; Marangoni convection; surface tension active elements; slag; welding arc.

Abstract. Surface tension active elements, such as sulfur and oxygen, strongly influence the weld pool size during the GTA welding of stainless steel. The variation in concentration of these elements combined with the temperature gradient in the weld pool can generate a complex Marangoni flow pattern. At low arc power, this effect has been proven both experimentally and by numerical simulation to be the main factor behind the variation of the weld pool size and penetration. The current paper is based on a study, conducted in the research center of Hydro-Québec in the early 80s, involving a high speed cinematographic observation of these phenomena during autogenous GTA fusion tests of austenitic stainless steel tubes, plates and droplets. For the droplet test a unique molten split anode set-up was designed to analyze the current flux distribution between the two droplets. The present work compared these observations with recent numerical simulations of the GTAW arc and weld pool from the literature. This comparison shows that in order to explain in particular the observation of a quasiperiodic reversal of the Marangoni flow direction in a weld pool, a comprehensive understanding of the effect of slag formation and metal vaporization is required.

269 The influence of microstructure on low cycle fatigue behavior of steels containing retained austenite

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The influence of surrounding microstructure and pre-strain on low cycle fatigue behavior has been investigated in steels containing metastable retained austenite. Fully-reversed uniaxial and bending fatigue tests were performed on single phase austenitic (301 stainless steel) and multiphase microstructures including two transformation induced plasticity (TRIP) steels: a first generation advanced high strength TRIP steel and a 3rd generation TRIP7Mn steel. Additionally, notch fatigue experiments were conducted on select conditions to evaluate the effect of retained austenite on short crack growth. The retained austenite to martensite transformation rate is significantly different in fatigue in the multiphase and single phase austenitic microstructures. Retained austenite transformation occurs at the beginning of the fatigue life within the first 10-100 cycles in the multiphase microstructures, while the transformation occurs at the end of the fatigue life after several hundred or several thousand cycles in the single phase austenitic microstructures. Similar to monotonic deformation, the austenite grain size may play a role in its stability in fatigue. Pre-straining multiphase TRIP steel microstructures has little effect on retained austenite stability or fatigue life because the fatigue life is controlled by the softer ferritic phase; this effect of pre-strain is similar to single and multiphase steels that do not contain metastable austenite. The results are discussed in the context of the inherent austenite stability and other literature about the fatigue performance of

steels containing retained austenite.

270 Plasma polymerized allylamine - PPAAm - a cell adhesive finishing for implant surfaces

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Metallic implants are the state of the art in bone replacement surgery. The rapid cellular acceptance of the implant surface by the surrounding tissue is the crucial factor. Cell adhesion via integrins but also additional mechanisms via hyaluronan play a key role in the very first encounter with the biomaterial [1]. Gas-discharge plasma processes are successfully applied for surface modifications with bioactive coatings.

The finishing of titanium implant surfaces with a bioactive coating of PPAAm is especially beneficial. The thin film has valuable properties: homogenous, long time [2-4] and mechanical stable on air and in different aqueous solutions (cell culture media), well adherent, with a sufficient density of positively charged nitrogen functional groups for electrostatic attraction of ECM molecules. Tissue culture experiments with MG-63 cells demonstrate a considerable enhanced adhesion and spreading on PPAAm. In vivo investigations [5,6] show no inflammatory response and an enhanced bone-to-implant contact.

These films with its outstanding physico-chemical, in vitro and in vivo properties are particular favourable as thin cell adhesive coatings for titanium implant surfaces.

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271 Surface structuring of dental zirconia implants in terms of stable osseointegration

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Titanium is the material of choice in dental implantology due to its mechanical properties and its biocompatibility. However, in recent time the biocompatibility of titanium is doubted due to laboratory results, and some clinical observations of incompatibility are reported. An alternative to titanium is the application of zirconia, a tough ceramic, which is successfully used in total hip replacement. The strength of zirconia is based on a transformation toughening, based on a volume increase following the transition from tetragonal to monoclinic phase, which is triggered by tensile stress. Little evidence exists on the clinical performance of zirconia implants and insufficient experience is available on the requirements for stable osseointegration. For titanium implants it is known that a surface roughness of $R_a = 1.5\mu\text{m}$ improves the osseointegration. To achieve a similar roughness on zirconia implants sandblasting followed by acid etching has revealed to be an appropriate process. A subsequent

thermal treatment to recover the tetragonal phase seems to be reasonable. In cell cultures with human derived osteoblasts the thus generated surface demonstrated superior properties over machined or sandblasted surfaces in terms of cell morphology and improved mechanical interlocking of human primary osteoblasts into the porous microstructure of the acid etched and additionally heat treated YZ-surfaces. In a clinical two-center study with 70 implants the outcomes are promising. Except for one early failure all implants osseointegrated. The laboratory and clinical results suggest that the specified zirconia surface has the potential for stable osseointegration.

272 Integrated defect classification in manufacturing of carbon fibre reinforced thermoplastic polymer matrix composites

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Thermoplastic PMCs play a crucial role in automotive industry. This is mainly reasoned by their high potential in cost effective manufacturing. However, the manufacturing processes are challenging and require strong methods in quality control. Hence, a major goal is to evaluate methods for detecting and classifying defects in these materials under the requirement of short testing times. Active thermography could be classified as a promising NDT method for detecting defects. The underlying mechanism is an active loading of the composite part, e.g. with light pulses, such that a thermal wave is generated at the surface of the test object. The time behaviour of the transformed heat is detected by an infrared camera and hence an image of heterogeneities can be calculated from the measured data. Besides, external loading parameters and component configuration, results are strongly dependent on the material of the test object. In the past this method was used to detect e.g. impact defects in duroplastic polymer matrix composites and could be proved to be a valuable testing method. However, analyses of thermoplastic PMCs can only rarely be found. In this contribution, we show a concept for defect classification in the manufacturing line for thermoplastic PMCs. Strong focus is put on the optimization of active thermography for effective detection and classification of defects in carbon fibre reinforced thermoplastic PMC. Effects, having a high impact on defect detection in thermoplastic composites, e.g. heat conduction of fibres and matrix, interface and surface phenomena, are discussed in detail.

273 Size effects and plasticity of thin metallic materials: influence of the crystallographic structure and the stacking fault energy

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Due to size effects, the mechanical behavior of small parts of micro-systems and micro-components is different compared to massive ones, involving metal forming difficulties. The modification of the mechanical properties is directly linked to the thickness over grain size ratio (t over d ratio) of the parts. Three kinds of mechanical behavior have been recently highlighted:

polycrystalline, multicrystalline and quasi-single crystalline, separated by two critical t over d ratios. In order to study these size effects, tensile tests are performed on metals with different thicknesses and different grain sizes. The modifications of the mechanical behavior are noticeable in Hall-Petch plots: the linear relation is not respected anymore. The size effects also affect the work-hardening parameters like the threshold stresses between work-hardening stages. Previous studies carried out on two face-centered cubic metals (nickel and copper) have shown that the t over d ratio between the polycrystalline and the multicrystalline behaviors is directly linked to the stacking fault energy. This work focuses on high purity cobalt with an almost perfect close-packed hexagonal crystallographic structure. Whereas the deformation mechanism of copper and nickel is driven by gliding, the lack of slip systems in cobalt leads to a competition between gliding in the basal plane and twinning processes. The results clearly show that the low stacking fault energy of cobalt induces a high value of t over d ratio and the size effect appears at the very early stage of yielding.

274 Evolution of the dislocation structure of a cold worked high nitrogen steel during fatigue testing

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In this investigation an attempt was made to distinguish between the two alloying concepts for stainless austenitic steels, CrNi and CrMnN alloys, by comparing their behaviour under cyclic loading. It is well documented that CrMnN austenites have higher mechanical strength than the CrNi grades mainly attributed to the alloying element nitrogen, which occupies the interstitial sites in the crystal lattice and thus leads to a distortion of the lattice. Whereas the actual knowledge of the fatigue behaviour and the role of nitrogen is still limited and especially the evolution of the microstructure during dynamic loading is not well understood. To approach this issue fatigue tests were carried with one representative of each alloying group, each in solution annealed and cold worked state. Loading conditions where $R = 0,05$ under ambient atmosphere. The fatigue limit was chosen with 10^7 cycles. The changes in the microstructure during cyclic loading were documented by transmission electron microscope (TEM) investigations. A preferred formation of cellular dislocation substructures could be found in the CrNi samples. In contrast the CrMnN grade exhibited mainly planar dislocation glide. In the following pages possible explanations for this differing dislocation glide and its influence on the fatigue properties will be discussed.

275 Alloying effects on grain boundary motion and nanocrystal stability

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The evolution of the grain microstructure is a central issue in materials science. There is substantial current interest in the stabilization of nanocrystalline grain structures to maintain the enhanced properties of nanocrystalline metals. It is known that alloying can have a profound influence on microstructure evolution in general and for nanocrystals in

particular. However, there is ambiguity about the relative roles of various mechanisms including thermodynamic stabilization due to alloy segregation, impurity drag and precipitate-boundary interactions. In this talk, we will use a combination of atomistic simulations and phase field modeling to elucidate the relative importance of these various mechanisms. Atomistic simulations will be used to investigate the heats of segregation of alloying elements to grain boundaries, the influence of segregated species on grain boundary mobility and the variability of these factors with boundary type. Phase Field simulations will be used to study the influence of these factors, both individually and in concert, on the overall microstructure evolution. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

276 Effect of electrolyte solution concentration and composition on the transport properties of ion exchange membranes for applications in energy conversion systems

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Ion exchange membranes (IEMs) have consolidated applications in energy conversion and storage systems, like fuel cells and battery separators. Moreover, in the perspective to address the global need for non-carbon-based and renewable energies, salinity-gradient power (SGP) harvesting by IEM-based energy conversion processes, like reverse electrodialysis (RED), is attracting significant interest in recent years. RED requires membranes specifically designed with low ionic transport resistance and high permselectivity because of the significant role of these electrochemical properties on the system performance. However, these properties are not constant for a given membrane at a certain temperature, but they strongly depend from the concentration and composition of the contacting electrolyte solutions, due to the effect on membrane microstructure and ionic concentration profiles in the membrane and at its interface. The aim of this study was to investigate the effect of solution concentration, compositions, temperature and velocity, on the membrane and interface ionic resistance of IEMs (Fujifilm AEM-80050 and CEM-80045) by electrochemical impedance spectroscopy (EIS). Moreover, membrane permselectivity was measured by the membrane potential method. Pulsed gradient spin echo (PGSE)-NMR measurements gave additional important insights about the effect of external solution on the water self-diffusion coefficients in membrane. A good correlation between the membrane properties measured by ex-situ methods for each membrane type, with the maximum power density obtained in a 25 cell pairs RED stack, was observed. The findings assure one more step ahead in decision making for solution pretreatment and/or membrane design for energy conversion applications.

277 Modelling methods of magnetohydrodynamic phenomena occurring in a channel of the device used to wash out by a liquid metal of spent automotive catalyst on metallic substrate

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The recovery of precious metals is necessary for environmental and economic reasons. Spent catalysts from automotive industry containing precious metals are very attractive recyclable material because as the devices have to be periodically renovated and eventually replaced. Among automotive catalysts withdrawn from use, these with metallic carrier constitute quite a big group. Metallic carriers are usually obtained from steel FeCrAl, which is covered by a layer of PGM acting as a catalyst. World literature describes a number of pyro- or hydrometallurgical methods used for recovery of platinum from used automobile catalytic converters. However, all the methods, available in the literature, are used to recover platinum from ceramic carrier. This paper presents the new method of removing platinum from the spent catalytic converters applying lead as a collector metal in a device used to wash out by using magnetohydrodynamic pump. The article includes the description of the methods used for modelling of magnetohydrodynamic phenomena (coupled analysis of the electromagnetic, temperature and flow fields) occurring in this particular device for this kind of waste. The general phenomena and ways of coupling the various physical fields for this type of calculation were also described. The basic computational techniques with a discussion of their advantages and disadvantages are presented.

278 Biodegradability, cytotoxicity, mechanical and magnetic properties of newly- developed Fe-Mn-Si-Pd alloys during in-vitro immersion tests in simulated body fluid

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Two new, potentially biodegradable, Fe-based alloys, Fe-10Mn6Si1Pd and Fe-30Mn6Si1Pd, were fabricated by arc-melting and subsequent copper mold suction casting. The progressive changes in their microstructure, mechanical and magnetic properties during immersion tests in Hank's solution were investigated. The cytotoxicity of these alloys was also studied in view of their potential applications in the biological field. These alloys show relatively high nanoindentation hardness values (5.6 GPa and 4.2 GPa, for 10 and 30 wt% Mn, respectively), higher than the typical hardness of the 316L stainless steel, which constitutes one of the most common Fe-based reference materials for biomedical applications. Magnetic measurements evidenced ferromagnetic properties at room temperature for the alloy with 10 wt% of Mn whereas the alloy with 30 wt% Mn remained paramagnetic, even after long immersion times. Interestingly, structural and thermal analysis revealed that the sample with 30 wt% Mn is also

a shape memory alloy. The shape memory effect, together with the non-magnetic character of this alloy, make it very appealing for its applicability in a wide range of biomedical applications, including orthopedic implants or stents where non-magnetic alloys are required so as to be compatible with magnetic resonance imaging techniques. In terms of biocompatibility, the biodegradability rate was assessed by immersion tests in Hank's solution at 37°C for 7, 30, 60 and 120 days. Cell viability and morphology analysis using an osteoblastic cell line indicated that after 7 days, the alloy with 30 wt% Mn was non-cytotoxic and allowed better cell adhesion.

279 Multi-institutional collaboration of industry and university along the processing chain as a means to holistically optimize material characteristics of aluminum products

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Aluminum is preferred choice for a range of products, especially in the transportation industry. However, in the competition of materials, for aluminum there are still issues that need to be solved or properties that require improvement in order to increase market share. AMAG is a rather small producer of semi-finished aluminum products on a global scale, but is quality wise a worldwide well reputed rolling mill with a preeminent share of more than 57 % of specialty products. To maintain the strong pace for innovation of suitable products that meet the demanding requirements of its customers, AMAG relies on a close multi-institutional collaboration to holistically optimize material characteristics of its products. The investigated processing chain starts at the feedstock and ends with the processing of the semi-finished product at the customer. Increasing attention is paid to simulation and experimental verification of models including linking the different calculation tools. In this paper, the successful collaboration between industry and several university institutes is presented on the basis of an example for a 6xxx alloy for automotive applications which shows that each aspect has to be investigated to achieve a desired goal.

280 Polycrystalline gamma/gamma prime Co-base superalloys produced by casting and rolling

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In order to investigate the potential of Co-base superalloys as a replacement for polycrystalline Ni-base superalloys, a series of multinary gamma/gamma prime Co-base superalloys has been developed. This novel alloys are produced by casting and rolling and after a three-step heat-treatment they show a fine grained gamma/gamma prime microstructure with a multimodal gamma-prime distribution and a positive lattice misfit. The oxidation resistance of this alloys could be improved compared to the ternary Co-Al-W system and is now in the range of the commercially available polycrystalline Ni-base superalloys Waspaloy and Udimet720Li that are used as reference alloys. The alloys show a good high temperature strength that is

comparable to the reference alloys up to 800°C, but at 850°C it even overcomes the specific flow stress of both Ni-base superalloys. A reason for this could be a higher peak temperature of flow stress anomaly or different deformation mechanisms. In terms of creep resistance, the Co-base alloys show a higher creep strength than the compared Ni-base alloys. Differences in gamma prime content, grain size or deformation mechanisms could explain this behavior and are therefore investigated in further detail. Over all it could be shown that the alloys show good oxidation resistance and high temperature strength as well as excellent creep properties. The ability to produce this new alloy series via the cast-and-wrought route makes them attractive high temperature materials. Therefore they could be an alternative to some of the common polycrystalline Ni-base superalloys.

281 Quantum-mechanical study of clean and impurity-segregated grain boundaries in Ni₃Al and Fe₃Al

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Iron and nickel based intermetallics are considered as very promising high-temperature structural materials that may significantly improve the efficiency of future energy-conversion units. Their strength is to a large extent determined by the structure and properties of their internal structural defects. Grain boundaries represent an important class of two-dimensional extended defects and it is known that impurities segregating in ppm concentrations at grain boundaries can drastically change material performance and properties. After our previous study of segregation trends of sp elements in Ni [1,2], we extend our research to two industry-important binary intermetallics, Ni₃Al crystallizing in the L1₂ structure and Fe₃Al with the D0₃ phase. We employ quantum-mechanical methods to study the energetics, magnetism, segregation and strengthening/embrittling tendencies in the case of Σ5(210) grain boundaries. Using the VASP code, we simulate not only the clean Ni₃Al and Fe₃Al grain boundaries but also those with segregated Si. Specifically in case of Fe₃Al, a long-lasting problem with quantum-mechanical calculations is an incorrect prediction of the ground-state structure of Fe₃Al. Nearly all exchange-correlation functionals lead to the preference of the L1₂ structure instead of experimentally found D0₃ phase. Here we report on results of our comparative study of a number of different exchange-correlation functionals, and, importantly, we identify a single generalized gradient approximation parametrization and computational parameters setting that predict the ground state of Fe₃Al correctly. [1] M. Vsianska and M. Sob, Prog. Mat. Sci. 56 (2011) 817. [2] M. Vsianska and M. Sob, Phys. Rev. B 84 (2011) 014418.

282 Deformation behaviour of BCC metals investigated by small- and macro-scale testing techniques

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Investigation of size effects and deformation kinetics on small scale samples became very popular over the last years, since they allow to determine the temperature dependent mechanical behaviour of thin films or small components. Deformation mechanisms can be differentiated by the strain rate sensitivity (SRS) of the flow stress and the related activation volume.

Moreover, it is known that the deformation behaviour is influenced significantly by the present microstructure and the critical temperature of the respective material. In this work, we concentrate on the deformation behaviour of body centred cubic (bcc) metals, namely chromium and tungsten, which exhibit a strong contribution of the thermal stress component to the flow stress. Focused ion beam milling was used to fabricate single crystalline (sx) and ultrafine-grained (ufg) pillars in a size range between 200 nm and 4 μm . Subsequently, the pillars were compressed in-situ in a scanning electron microscope at different temperatures to extract SRS data from calculated stress-strain curves. This data was compared to results acquired by nanoindentation and macroscopic compression tests, carried out at different temperatures as well. Finally, the results were related to the corresponding critical temperatures of the examined sx and ufg bcc metals and their coarse-grained counterparts to investigate the deformation behaviour over a wide range of temperatures and sample volumes. We want to conclude, that small scale samples can be used to determine deformation behaviour as known in macro samples, as long as the sample size effect is considered.

283 Shear band and texture formation in intermetallic gamma titanium aluminides during severe hot-working

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The microstructural consolidation of the gamma titanium aluminides by hot-working is impeded by the formation of shear bands and textures. The shear bands and textures are particularly pronounced after severe hot-working necessary to achieve well-consolidated microstructures. Thus the underlying formation mechanisms and the resulting effects on the deformation behaviour could be studied thoroughly, which was done by scanning electron microscopic, transmission electron microscopic, high-energy X-ray diffraction, and mechanical testing techniques. It will be shown in detail that the plastic anisotropy of lamellar structure, which is the major microstructural constituent of the alloys in the as-cast and HIPed state, promotes the formation of shear bands. Once developed, the deformation within the shear bands is stabilized by recrystallization processes initiated at grain and twin boundaries leading to continuous softening of the shear zones. These difficulties can largely be prevented by extrusion, because it generates high hydrostatic stresses in addition to relatively small shear stresses, effectively suppressing the formation of shear bands. It will be demonstrated that very homogeneous microstructures can be achieved by this technique, when the material is extruded twice with the two extrusion directions being oriented perpendicular to each other. The microstructures show, however, morphologic and crystallographic textures. The textures can be ascribed to the plastic anisotropy of the constituent phases in accordance with the directional shear stresses induced by extrusion resulting in anisotropic deformation characteristics (U. Froebel and A. Stark: Metall. Mater. Trans. A, 2015, vol. 46A, pp. 439-55).

284 Ceramic coatings for protecting carbon/carbon composites against oxidation

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Carbon/carbon (C/C) composites are the leading candidates for high temperature structural components in aerospace fields. One key drawback of these composites for such an application is oxidation during their exposure to an oxygen-containing atmosphere and the resulting rapid recession. Therefore, the applications of C/C composites as the high-temperature structural materials depend on the development of protection technique against oxidation. Currently, multi-layer and multi-phase ceramic coatings are the most promising approach. From the late 1990s, silicon-based ceramic coatings had been investigated widely for anti-oxidation of C/C composites by forming a slow-growing and dense silica glass. After the discovery of cracking from the mismatch of thermal expansion between silicon-based ceramic coatings and C/C composites substrate, the focus of anti-oxidation coating research has been shifted towards the elimination of cracks in the ceramic coatings. Subsequently, some novel coating systems were developed, such as the multi-phase inlay ceramics, whisker-toughened ceramics and gradient ceramic coatings. Simultaneity, pre-oxidation was applied to obtain a transition layer to improve the thermal stress resistance of the coatings. This report will give a brief introduction of the history and the current status of oxidation resistant ceramic coatings for C/C composites in our university, and the protection and failure reasons of some coatings at high temperature will also be provided.

285 High-voltage scanning-transmission electron microscopic observation of labyrinth structure developed by cyclic deformation in a [001] copper single crystal

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Copper single crystals of a [001] multiple slip orientation were cyclically deformed under constant plastic-strain amplitudes of 6×10^{-4} and 6×10^{-3} at room temperature. Cyclic hardening curves showed cyclic hardening to saturation, and saturation stress depended on the plastic-strain amplitude. High-voltage scanning-transmission electron microscopic observation revealed that characteristic dislocation structure developed during cyclic deformation depending on the applied strain amplitude. Vein-like dislocation structure, having a parallelepiped shape with two longitudinal (100) and (001) sides, was periodically formed along the [010] direction at the strain amplitude of 6×10^{-4} . At the strain amplitude of 6×10^{-3} , on the other hand, typical labyrinth structure constituting of the (100) and (001) dislocation walls was evolved from the vein-like structure. The g•b analysis was also carried out to identify the Burgers vectors of dislocations in the walls.

286 Fatigue properties including fatigue free in bulk metallic glasses

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Fatigue tests were carried out on ultra-high strength Co- and Fe-based bulk metallic glasses (BMGs), the super-high strength Ni-based BMG and the high strength Ti- and Cu- and Zr-based BMGs. The test alloy rods with a diameter of 2 mm were prepared by the copper mold casting method. The test specimens were machined to hourglass shape type with a minimum diameter of 0.9 mm. The specimens were tested at a stress ratio of 0.1 and a frequency of 10 Hz. The fatigue limits in the Co- and Fe-based BMGs exceeded 2 GPa, and those in the Zr-, Ni- and Ti-based BMG surpassed about 1.5 GPa. Fatigue ratios in all BMGs were found to be about 0.5, exceptionally, the Ti- and Zr-based BMG showed the high ratio, about 0.8 and 0.9 respectively. These BMGs indicated the possibility that the BMG does not cause the fatigue phenomenon. In front of the fatigue crack initiation sites, fracture surfaces showed a radial ridges and valleys morphology as well as a striation like pattern. Only the Zr-based BMG did not show any striation like pattern. Unstable fracture surfaces consisted of facets with vein patterns. As the facet and vein pattern sizes increase, the strength and fatigue limit decreased, whereas the fracture toughness increased. Fracture surfaces in front of the fatigue cracks in strong BMGs showed the clear elastic wave traces.

287 Towards highly efficient wavelength-stable red light-emitting diodes using Eu-doped GaN

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After the groundbreaking invention of blue and green light-emitting diodes (LEDs) employing nitride semiconductors ($\text{In}_x\text{Ga}_{1-x}\text{N}/\text{GaN}$), there has been a strong demand to develop red LEDs using nitride semiconductors. We have focused on Eu ions that have been widely used as an activator for red phosphor, and have succeeded in developing the world's first red LED that operates at room temperature using Eu-doped GaN (GaN:Eu) as the active layer. Light output power of the LED has been growing steadily up to sub-milli watts in these three years. However, further increase in light output power by just one order is strongly desired for practical application. In this talk, we report on Eu photoluminescence (PL) properties in two series of GaN:Eu samples with artificial external perturbation; one was grown on AlGaIn/AlN superlattices (SLs) with different in-plane compressive strains, and the other was embedded into a microcavity consisting of an AlGaIn/GaN distributed Bragg reflector (DBR) and a Ag reflecting mirror, designed for Eu emission at ~ 620 nm. The compressive strain increased the fraction of an efficient Eu luminescent center and improved energy transfer efficiency of the center. Furthermore, by combining the effects of surface plasmon resonance (SPR) and increased optical mode density, the PL intensity was increased drastically as compared to typical GaN:Eu samples. These new findings will give a clue for the development of a practical electrically pumped GaN:Eu-based red LED.

288 Effect of IMC interlayer on mechanical property of dissimilar metal joint made by FSW

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Fabrication method for high performance dissimilar metal joint has been established in our research group by using friction stir welding. 100 % joint efficiency was given by the method in the welded joint between normal steel and Al alloy. Fe_2Al_5 intermetallic compound, IMC, layer with 300 nm thickness was observed at interface between both materials. It is believed that the thickness of the IMC layer at interface is a key factor to decide the mechanical property of the joint. However, any quantitative, reliable relationship between IMC layer thickness and the mechanical property of the joint has been verified up to today. This study aimed to evaluate the mechanical property of the dissimilar metal joints made by the friction stir welding systematically. The results obtained showed that the mechanical strength of the welded joint proportionally decreased with increase of the IMC layer thickness over 1 micron meter thickness range. Less than 1 micron meter thickness range, on the other hand, the strength of the joint had almost constant value. By heating the joint at 723 K, fraction of Fe_2Al_5 in IMC layer at interface seemed to increase with heating period. However, the clear relation between fraction of Fe_2Al_5 and mechanical strength of the joint was not verified. More precise observation should be carried out in the future study.

289 Aging property of AZ91D magnesium alloy screw thread-rolled at room temperature using extrusion-torsion simultaneous processing

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We have proposed a new extrusion process functionally combined with torsion. Extrusion-torsion simultaneous processing is a very attractive technique for fabricating a rod-shape material with high strength and excellent workability. To improve the hardness, the aging treatment was performed with AZ91D magnesium alloy screw thread-rolled at room temperature using extrusion-torsion simultaneous processing. Non-distribution of hardness from the tip to center in as thread-rolled screw was modified to uniform distribution by the isothermal aging treatment at 423K for 460.8ks.

290 Catalytic reaction with aunps/conjugated dibrock copolymers (iii)

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Gold nanoparticles (AuNPs) show excellent catalytic ability in a number of chemical reactions. In order to achieve high catalytic ability in such a state, aggregation of AuNPs should

be avoided by using appropriate dispersion reagents. Here, we used hydrophilic-hydrophobic diblock copolymers containing conjugated polythiophene derivatives for support materials of AuNPs. The effect of S_mHT_n on the catalytic ability and the structure of AuNPs were investigated. The diblock copolymers, poly(di[4-(2,2-dimethylpropoxysulfonyl) phenylene]-b-3-hexylthiophene), S-PDPrB-b-P3HT (S_mHT_n), were synthesized by the deprotection reaction of precursor diblock copolymers (NS_mHT_n) synthesized by the catalyst-transfer condensation polymerization method. AuNPs were supported with S_mHT_n (AuNPs/ S_mHT_n) in aqueous solution. STEM images of AuNPs/ S_mHT_n showed that AuNPs were well-dispersed in S_mHT_n . Alcohol oxidation reaction was carried out in aqueous solution with AuNPs/ S_mHT_n as the catalyst. X-ray photoelectron spectra exhibited that the characteristic peaks attributed to Au(0) for AuNPs/ S_mHT_n . While S_mHT_n has no catalytic ability, AuNPs/ S_mHT_n showed catalytic ability in the oxidation reactions of benzhydrol, and had two times of the AuNPs oxidative efficiency. Oxidation efficiency of AuNPs/ S_mHT_n increased as the hydrophilic ratio of S_mHT_n increased. These results suggest that S_mHT_n have the ability of supporting AuNPs and function to promote the catalytic ability of AuNPs.

291 Enthalpy recovery and aging dynamics measurements reveal a stick-slip mechanism of atomic motion during physical aging of a fragile metallic glass

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A key to understand the aging mechanisms of a glass on the microscopic level is to measure the time scale on which the system rearranges its internal structure. Here, a systematic study of the long term relaxation behaviour of the $Au_{49}Cu_{26.9}Si_{16.3}Ag_{5.5}Pd_{2.3}$ bulk metallic glass is presented. The work [1] was performed using conventional differential scanning calorimetry (DSC), nanocalorimetry (Flash DSC), and X-ray Photon Correlation Spectroscopy (XPCS).

Multiple plateaus in the enthalpy recovery and in the structural relaxation times during the physical aging of the glass are detected. Contrary to what it was observed on stronger glasses where smooth-sliding motion events affect the aging towards a fully relaxed liquid, like $Mg_{65}Cu_{25}Y_{10}$ [2,3], this more fragile system ages in a highly heterogeneous manner, consisting of periods of stationary dynamics interconnected with fast-motion popping events, typical of stick-slip dynamics. This complex stick-slip equilibration mechanism of the atomic rearrangements is connected to finite energy dissipations during slipping, typical for first-order thermal instabilities. [1] I. Gallino, R. Busch, D. Cangialosi, B. Ruta, S. Hechler, L.-Y. Schmitt, M. Stolpe, W. Hembree, and Z. Evenson, manuscript in preparation [2] R. Busch et al. *Acta Mater.* 46 (1998) [3] B. Ruta et al. *PRL* 109 (2012)

292 The mechanical and micro-structural characterisation of novel high strength, highly creep resistant maraging steels for shaft applications.

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This PhD project is in the final stages of understanding the mechanical behaviour of a new heat resistant steel alloy system for future aero engine generations. 5 novel maraging steels have been produced, each strengthened by the C14 Hexagonal Laves Phase, and the Cubic B2NiAl intermetallic phase. The major aims of this project have been to complete mechanical testing (tensile, fracture toughness LCF and creep stain) to optimise the heat treatment and down-select the leading alloy chemistry of the 5 variants. Post test fractography, microstructural characterisation using TEM and atom probe tomography (APT) has been crucial in helping to understand the role of precipitates during testing- in particular creep. To add to the understanding of precipitate evolution, a Small-Angle-Neutron-Scattering experiment (SANS) was performed in November 2014, quantifying the precipitate nucleation and coarsening during isothermal tempering. The microstructural measurement by APT has been linked to the mechanical response of the alloys, concluding that the size, shape and chemical composition of the precipitate phases is fundamental to their mechanical response. TEM analysis has been performed with Cambridge university linking microstructure and dislocation interaction in the creep test condition of the alloys. A key deliverable from this project has been the down-select of a composition and heat treatment displaying greater properties than that of the original alloy composition.

293 Irreversible thermodynamics applied to diffusional phase transformations

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Both the kinetics of phase transformations in the solid state and the kinetics of solid/liquid transformations can be visualized by high temperature laser scanning confocal microscopy. It had been confirmed by this technique that during partial cyclic phase transformations in the austenite/ferrite range of low alloyed steels two special stages may appear [1]. The phase becoming less stable at changing temperature grows during the stage of inverse transformation and transformation ceases completely for a certain time during stagnant stage. The thermodynamic system has to go through a series of off-equilibrium states and the underlying irreversible processes are quite well understood in case of the austenite-to-ferrite transformation in low alloyed steels. This work reports the existence of an analogous inverse transformation stage during solid/liquid transformation in ceramic systems (e.g. consisting of $\text{CaO} + \text{SiO}_2$). Unless the transformation proceeds without any change in composition (e.g. during congruent melting and solidification) – a stagnant stage is also detected during solid/liquid transformations. The irreversible processes resulting in an inverse transformation and a stagnant stage during solidification and melting are identified by numerical investigations. It is worth mentioning that the concept of partial cyclic phase transformations offers new possibilities for functionally oriented materials design. Reference [1] H. Chen, E. Gamsjäger, S. Schider, H. Khanbarez, S. van der Zwaag: Acta Mater. 61 (2013) 2414-2424.

294 Microstructure and residual stress in rotary friction welded dissimilar metals (Al7020-T6/316L)

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Rotary friction welding (RFW) is the first of the friction processes to be developed and used commercially [1]. No additional filler material is used and welding takes place in the solid phase during RFW, i.e. no macroscopic melting is observed. Since there is nearly no limitation to weld any metals the use of joints between dissimilar materials via RFW has considerably been increased, especially in the aerospace industry where conventional structures made of steel have been replaced by lightweight materials, such as Al, Ti, and even Mg alloys [2, 3].

In the current study the dissimilar metals Al7020-T6 aluminum alloy and 316L steel were joined using RFW. Neutron diffraction was used to investigate the texture gradient around the weld line and to map the residual stress over the whole specimen. The texture analysis showed a weak shear component near the bond line of Al7020-T6 side which indicated a plastic deformation of Al7020-T6 during welding. The shear bands were also observed in OM microstructures. Relatively high tensile residual stresses were observed near the bond line on the Al7020-T6 side, which were inhomogeneously distributed from the perimeter to the rod center, while high compressive residual stresses were found in the sample center at the bond line in the 316L steel. [1] P. T. Houldcroft, *Welding Process Technology*, Cambridge University, Cambridge 1977. [2] E.P. Alves, F. P. Neto, C. Y. An. *J. Aerosp. Technol. Manag.* 2 (2010) 301-306. [3] N. S. Kalsi, V. S. Sharma, *Int. J. Adv. Manuf. Technol.* 57 (2011) 957-967.

295 Modelling of diffusion limited growth in multicomponent systems

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Phase transformation involves diffusion-limited growth. For multicomponent systems, numerical solutions exist that provide sound and useful simulations. However, the use of analytical solutions remains pertinent in case of complex coupling phenomena between length scales or/and for achieving fast simulations. Analytical solutions for multicomponent systems presented in the literature are yet seldom and often based on crude approximations leading to unsatisfying results.

This contribution will present new one-dimensional analytical models for diffusion-limited growth considering multicomponent systems. Validations will systematically be considered by comparison with numerical solutions. As the analytical solutions are only valid for precipitate growth in an infinite matrix domain, the effect of the far field composition used by the analytical solution to compute the interface supersaturation will be discussed. Cross diffusion of species being accounted for by the analytical solution, its effect will also be demonstrated and discussed, evidencing the need to systematically take it into account. Coupling with thermodynamic and mobility databases will also be discussed.

296 Corrosion behavior of Al–7Si alloy processed by high-pressure torsion

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Due to the increasing demand for improving vehicle performance by using lightweight materials, aluminum alloys with silicon as a major alloying element are extensively used in automotive components. Among them, hypoeutectic Al–7Si alloys with ~4 wt.% Cu and ~3 wt.% Ni have predominantly been used for pistons. However, one of the drawbacks for these hypoeutectic Al–Si alloys is their low fracture toughness and strength. Severe plastic deformation processing is an effective method to achieve exceptional grain refinement and enhance strength of Al–Si alloys. In this study, high-pressure torsion was used to produce hypoeutectic Al–7Si alloy samples having a range of microstructures. The effect of the grain refinement on corrosion behaviour of Al–7Si alloy was subsequently investigated. Microstructure observation reveals that brittle coarse silicon particles and intermetallic phases are effectively broken into ultrafine-grained particles and redistributed homogeneously into the Al-rich matrix with increasing revolutions of HPT processing. Open-circuit potential and polarization curves results exhibit that corrosion resistance of the Al–7Si alloy in NaCl solution is significantly enhanced upon high torsion strains. Electrochemical impedance spectroscopy analysis combined with SEM observations indicates that the enhancement in corrosion performance of the Al–7Si alloy is due to the breakage of coarse silicon particles and intermetallic phases, the microstructure homogeneity and the increased HPT-induced active sites.

297 Residual stress measurements on IN718 fatigue specimens using X-ray diffraction techniques

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Residual stress measurements were successfully performed on the representative IN718 fatigue specimens by X-Ray Diffraction. All surface residual stresses were found to be compressive. A stress gradient normal to the surface was observed on all specimens. The residual stresses tended to become less compressive with increasing depth into the parts. Residual stress measurement is the special requirement for NADCAP CRITERIA AC 7101/7. Residual stress plays an important role in low cycle fatigue test. The low cycle fatigue test results were shown in this paper. Key words: residual stress; IN718; fatigue specimen; surface; stress gradient; compressive; low cycle fatigue test.

298 Plasticity analysis by synchrotron radiation diffraction in Mg-Y-Zn alloys

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Extrusion processing in magnesium alloys generates a strong fibre texture with the basal plane parallel to the extrusion direction. This texture causes the well-known tension-compression asymmetry in magnesium alloys in which material tested in the extrusion direction shows a lower yield stress in compression than in tension. However, such behaviour is not clear in Mg-Y-Zn alloys. The microstructure of these alloys after extrusion consists on of elongated fibres of LPSO phase embedded in a magnesium matrix. The magnesium matrix contains dynamically recrystallized (DRX) and coarse, worked unrecrystallized areas. The volume fraction of both grain regions depends on the LPSO volume fraction and the extrusion ratio. This article attempts to shed light on the tension-compression asymmetry in Mg-Y-Zn alloys using synchrotron radiation diffraction during in-situ tensile and compression testing to evaluate the deformation mechanism, basal slip or tensile twinning systems, controlling plasticity.

299 Deformation dilatometry to study the mechanical stability of austenite at different temperatures

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Studies of advanced high strength steels consisting of a martensitic or bainitic matrix coincide on the importance of retained austenite (RA) and its stability for their mechanical properties. However, the beneficial role of the metastable RA in the ductility and formability of these steels is still not fully understood. Several factors like RA morphology, size, carbon content and crystal orientation influence its mechanical stability. Surrounding phases also play an important role in the strain partitioning and they can delay or suppress martensitic transformation at larger strains. All these interacting factors complicate the study of the mechanical behaviour of single RA phase. This work aims to study mechanical properties and stability of RA in a 0.3C-1.5Si-3.5Mn steel by a novel approach, consisting of performing tensile tests at different temperatures (in the range between M_s and A_3) coupled with dilatometry measurements. The effect of grain size and temperature on the work hardening up to uniform elongation was studied. Thereafter, specimens were quenched and dilatometry curves combined with microscopy allowed to ascertain the fraction of austenite transformed during straining. Extrapolation of the results to finer austenite grain sizes and room temperature will allow a better understanding of the austenite mechanical behaviour during warm forming and also the determination of RA mechanical properties at room temperature without the influence of other phases.

300 Influence of long heat treatments on the microstructure and mechanical behaviour of HVOF sprayed WC-CoCr and Cr₃C₂-25NiCr coatings

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Thermally sprayed tungsten and chromium carbide-based coatings are widely applied in several industrial applications where wear resistance is required in relatively harsh environments such as in many components for oil & gas or petrochemical industries. The microstructural stability of the coatings is a key feature for improving the working life of components exposed at high temperature during service. While the two coatings are known to be suitable materials for low and high temperature, respectively, there is the need to know their behavior at intermediate temperature to make a proper selection in these cases.

In the present study two different coatings, WC-10Co-4Cr and Cr₃C₂-25NiCr, were deposited by HVOF and analyzed comparing the as received conditions with the aged states after isothermal treatments. The effect of aging at temperatures in the range 250-450°C was evaluated through microstructural evolution. In particular, the analysis was focused on the coating-substrate interactions and on the binder and reinforcement evolution. Furthermore, hardness, scratch tests and fracture toughness measurements were performed, before and after aging, for evaluating the contact response of the coatings under controlled loading conditions.

301 Components of a heart catheter system

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As late as fifteen years ago the intracardiac catheter was only used for diagnostic purposes. Since then it has also been established as a therapeutic method. The latest studies have shown that the reduction in convalescence that normally follows a catheter-interventional implantation of aortic valves by transcatheter aortic valve implantation is less significant in comparison to that of a cardiac surgery operation. It is expected that such minimal-invasive technologies will grow to a great extent, also helping to reduce socio-economic costs for the European health care system.

Patients of higher ages with acquired cardiac defects or children with congenital cardiac defects of heart valves, especially of the pulmonary valve, currently are the main target groups. We present an alternative and optimized mechanism for stent placement and similar therapeutical interventions. This project focused on the manipulation unit and tube system. It carries out a highly precise and repeatable linear motion. Ergonomic requirements are taken into account. Furthermore a possibility to support the linear motion by minimal strain is presented and discussed. The product is designed for disposable applications but is generally also suitable for long-term applications. Thermoplastic polymers have been used for the prototype. The design has been optimized considering the choice of materials and manufacturing technology.

302 Evaluation of hot pressing parameters on the electrochemical performance of MEAs based on Aquivion® PFSA membranes

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The membrane-electrodes assembly (MEA) is the core of a Polymer Electrolyte Fuel Cell (PEFC). It consists of membrane, catalytic and gas diffusion layers (GDL). In order to manufacture MEAs with suitable performance, a hot-pressing procedure is generally used. The relevant parameters are temperature, pressure and time of hot-pressing. Such variables need to be adjusted as a function of the type of ionomer used in the catalytic layer and the membrane. In this study, an evaluation of these parameters was carried out and their influence on MEA electrochemical performance was assessed. In particular, MEA preparation trials were carried out with reinforced experimental membranes based on Aquivion® short-side-chain PFSA (Solvay Specialty Polymers). The membranes were coupled to gas diffusion electrodes and MEAs were manufactured by using different temperatures for the hot-pressing. Electrodes were prepared by an in-house developed spray coating technique. The catalytic layer, based on a hydro-alcoholic ionomer solution D79-20BS (Solvay Specialty Polymers) and a 40%Pt/C (Johnson Matthey), was sprayed onto a commercial GDL Sigracet-25BC (SGL). A Pt loading of 0.2 mg/cm² for both anode and cathode was used. The influence of the hot pressing temperature on electrochemical performance was studied in the temperature range 80-110°C and low relative humidity for reactant gases. The electrochemical performance of the prepared MEAs was tested in a 25 cm² single cell in terms of polarization curves and cyclic voltammetry. This study was carried out in the framework of FP7/2011–2014 under Grant Agreement No. 303452–IMPACT.

303 Application of optical techniques to detect chemical and biological agents dangerous for human health

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Gaudio P., Malizia A., Gelfusa M., Murari A., Pizzoferrato R., Carestia M., Cenciarelli O., Ludovici G.M., Gabriele J., Gabbarini V. and Richetta M. Chemical or biological contamination, due to natural or man-made disasters represents a severe concern for safety and security of people and of the Environment. Chemical Agents (CAs) and Biological Agents (BAs) are commonly used for a number of civilian and military applications, and of course, can be used as a weapon for terroristic purposes. Therefore, it is necessary to develop specific systems aimed at preventing or reducing the consequences of the spread of these agents. To this end, the authors have developed an optical system to detect CAs, and are investigating the potentialities of optical techniques to detect and discriminate BAs; these studies will be presented in this work together with the analysis and discussion of the main results obtained so far. The authors are also investigating parallel developments of these approaches in the field of detection of contamination by undesired CAs and BAs in hospitals and medical related environments, as well as in pharmaceutical industries.

304 Continuous modeling of dislocations cores using a mechanical theory of dislocations fields

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The elasto-plastic model of dislocations fields[1] is used to simulate the relaxed configurations of dislocations cores having planar structure. A one dimensional model for edge and screw dislocations is developed. Initial narrow dislocations cores are shown to spread out by transport under their own internal stress fields, until they completely disappear, and no relaxed configurations is found. In order to stop infinite relaxation, a crystal misfit energy is introduced in the free energy density, leading to a restoring stress in the driving force for plastic deformation. When using the Peierls sinusoidal potential for the restoring stress, initial arbitrary dislocation core profiles relax and converge towards the Peierls-Nabarro analytical solution, corresponding to the lowest energy configuration. The model is then extended to use generalized planar stacking fault energies as an input. Dislocations in Hexagonal Close-Compact (HCP) materials are investigated. The gamma surface energies for the materials are calculated by ab-initio calculations. The model can be predicting partial dissociation of edge and screw dislocations. Motion, viscous deformation mechanisms of dislocations cores under loading is then predicted by the model. [1] A. Acharya, A. Roy, J. Mech. Phys. Solids 54, 1687(2006).

305 Granulation of bulk metallic glass forming alloys as a feedstock for thermoplastic forming and their compaction into bulk samples

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The various technologically important properties of metallic glasses are intimately connected to their amorphous structure that lacks the archetypical structural defects of polycrystalline metals and alloys, i.e. dislocations and grain boundaries. However, the amorphous structure also limits the application potential of this class of materials because of a macroscopically brittle behavior and size limitations. Consequently, with some exceptions, at least one dimension for technological products is limited to a few millimeters or even less. With the presented technological approach this drawback will be addressed. Our first results on several alloys show that with a dedicated instrumentation amorphous granulates can be successfully produced. By hot pressing in the supercooled liquid region, these granulates can be compacted into bulk shapes in the cm range. Further, due to the low viscosity of the supercooled liquid state, this technology disposes of a high formability. It is demonstrated that not only compact samples but also complex shapes in near net shape geometry can be produced. Results on the mechanical properties and microstructure will be discussed and related to important processing issues. Even though this technological approach does not directly address the second drawback of bulk metallic glasses, i.e. catastrophic failure due to highly localized shear bands, it is believed that this route offers possible pathways to improve this issue as well and, most important, to offer a technological route for implementing bulk metallic glasses into products of rather arbitrary

shape and larger size.

306 Modelling the local microstructure properties due to multi-pass welding

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Complex weld structures are mostly joined with overlapping or multi-pass welds. As a result of the repeated heat input, the local areas of the heat affected zone (HAZ) are subjected to multiple thermo-mechanical influences. This affects in a complex way the microstructure transformations and thereby the local thermo-mechanical properties. However, the lack of thermo-mechanical material properties for repeated heating and cooling is an obstacle for development and calibration of simulation models for multi-pass welding.

The paper presents an advanced approach which allows considering the influence of microstructure transformations on the change of the local thermo-mechanical properties during multiple temperature welding cycles. The approach describes the resulting material properties as a function of the local transient temperature history. Therefore the maximum temperature austenitisation cooling time model (abbr. from German: STAAZ) is extended taking into account the initial microstructure formed after the preliminary thermal cycle. The model is calibrated using experimental material properties obtained by repeated thermal load with different maximum temperatures and cooling times by means of Gleeble simulation.

An application example, realised by means of numerical finite element welding simulation is used to demonstrate the capabilities of the modelling approach.

As a result the model calculates the inhomogeneous distribution of the microstructure properties in the HAZ due to different initial microstructures and variable austenitisation conditions. The model for the complex microstructure transformations establish the basis for advanced thermo-mechanical simulation of the residual welding stresses and distortions of multi-pass welded components.

307 Properties of friction stir processed Al-alloy nanoparticle reinforced composites

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The use of friction stir processing has been developed to produce metal matrix composites with Al-alloy and different reinforcing particles including ex-situ Al₂O₃, SiC, and B₄C particles, as well as Al₃Ti in-situ nucleated particles formed by the addition of Ti powder. The development of processing parameters to fabricate composites with uniform particle dispersions is demonstrated. The microstructures and properties of both micro and nano-scale reinforcing particles are compared. Scanning electron microscopy and transmission electron microscopy is used to show that the particles are uniformly embedded within an ultra-fine grained matrix. The relative effectiveness of each particle type and different particle size distributions is compared versus volume fraction of reinforcement. It is shown that the yield strength can increase by 25% while maintaining comparable ductility to the Al alloy base material when nano-scale SiC particles are employed. In contrast, the formation of Al₃Ti particles increased

the yield strength by 63%, while tensile elongation decreased from 29 to 22%. It is also noted that refinement of the particles occurs during friction stir processing, however the refinement is only significant when the added particles are initially micron-sized.

308 Electron beam welding of the softmartensitic steel 1.4317 (CA6NM)

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The welding parameters for electron beam welding of heavy sections made of the softmartensitic stainless cast steel 1.4317 (CA6NM) were established. Welding was performed using a 40 kW electron beam welding generator. The aim of the investigation was to achieve a sound weld of parts showing a thickness of 93 mm and to assess the need of preheating and post weld heat treatment (PWHT). Welding was always done without using of a filler metal. The welding parameters were first identified for so-called blind seams (electron beam moving in bulk material) using single and multi-pass techniques. Subsequently butt seams were manufactured using the parameter sets obtained in the first project phase. During parameter optimizing the focus on interest was put on avoiding formation of pores and longitudinal holes. The testing program consisted of metallographical investigations as well as of hardness and Charpy impact measurements across the weld seams. Additionally non-destructive testing by means of x-ray was also performed. The preliminary tests showed that welding without preheating and PWHT results in a hardness of about 400 HV10. PWHT reduced the hardness to about 360 HV10 and improved the Charpy impact toughness slightly.

309 Microstructure evolution during high pressure torsion of W-20Cu bimetallic composite

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Severe plastic deformation techniques such as High Pressure Torsion (HPT), Equal Channel Angular Extrusion (ECAE), Accumulative Roll Bonding (ARB) have been successfully used for synthesizing ultra fine grain and nanocrystalline metals and alloys. During deformation the dislocations rearrange themselves in cellular and subgrain boundaries leading to fragmentation of initial microstructure. The understanding of deformation induced microstructure evolution is the key for developing materials with appropriate combination of properties such as strength and ductility. The fine grain microstructure is generally unstable at higher temperatures. However, introduction of second phase leads to significant improvement in thermal stability of nano composites. In this study tungsten-copper bimetallic composite (W-20 wt%Cu) is used as a model material for understanding the role of second phase in microstructure evolution. W20Cu composites are deformed by HPT method and the grain fragmentation process is followed at different strains. During deformation the initial W particles deform and fragment into smaller particles, while the copper grains deform between them. Electron back scatter diffraction (EBSD) is used to understand the crystallographic aspects of microstructure evolution. Further, comparison of microtexture and grain boundary character distribution in

W and Cu phases of the composite along with the HPT deformed pure Cu and W are conducted to get insight into the role of second phases on the process of grain refinement.

310 The quantification of galling in forming operations of hot dip galvanised sheet metal under laboratory conditions

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In the sheet metal forming industry, tools are subject to mechanical, thermal, chemical and tribological loads. One of the major problems in forming operations of hot-dip galvanised sheet metal is galling (build-up of zinc flakes on the tool).

This phenomenon develops gradually as an adhesion on the tool surface. The adhesive wear leads to high rejection and reworking costs for large car body forming tools. Due to economic aspects and the easy castability, the forming tools are made of grey cast iron. These materials tend to high adhesive wear.

The aim of this project is to find a three-dimensional surface parameter, which describes a tribologically advantageous surface of forming tools to reduce galling. An additional objective is to optimize tool materials, heat treatment and surface coating. The evaluation of galling under laboratory conditions is based on strip drawing tests. The characterization of tool materials was executed for lamellar grey cast iron (EN-GJL-200), globular grey cast iron (EN-GJS-700) and a cold work steel (Carmo). Investigations showed that the processing methods and test parameters like sliding speed and temperature have a significant influence on galling. Three-dimensional surface parameters have also shown an effect on galling.

311 An all-wet electroless-plating process for copper metallization of through-silicon vias involving amino self-assembled monolayers

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In this work, a process flow that involves grafting and functionalization of a 3-aminopropyltrimethoxysilane self-assembled monolayer (APTMS-SAM), seeding and electroless plating is developed that fabricates a Co-alloy barrier layer, Cu seed layer (or Cu line) onto through-silicon vias (TSVs) in an all-wet and in-line manner. First, ordering and quality of the grafted APTMS molecular layers as a function of reaction times are monitored by X-ray photoelectron spectroscopy, ultimately leading to the growth of a highly ordered APTMS-SAM with the content of primary amino groups ($-NH_2$) exceeding 95%. Subsequent treatment in SC-1 solution ($pH = 10.5$) causes deprotonation of the $-NH_2$ groups, creating negatively charged surface functionalities on the APTMS-SAM that are viable for the adsorption of metallic seeds (e.g., Co or Ni). Neither the combination of costly $PdCl_2$ and complex additives nor the demerits of the associated aqueous chemistry (e.g., agglomeration and sparseness of Pd seeds) are involved. The adsorbed Co (or Ni) particles act as seeds to trigger electroless plating of a Co-alloy barrier layer with high adhesion strength of greater than

70 MPa. By adjusting the plating solution, a Co-alloy barrier layer is uniformly deposited onto TSVs with high aspect ratios of 10. The Co-alloy barrier layer plated serves as a catalyst that triggers electroless filling of a Cu seed layer (or Cu line) into the TSVs.

312 Correlation between aging effects and high temperature mechanical properties of the unmodified A356 foundry aluminium alloy

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In this study, the effect of aging on the mechanical properties of unmodified A356 aluminium casting alloy with trace additions of Ni or V was investigated. Trace elements were added in concentrations of 600 and 1000 ppm of Ni and V, respectively. Samples from sand and permanent mould castings in as-cast and T6 heat treated conditions were tested. Tensile tests were performed at both room and high temperature (235 °C). Taking into account the results from both testing conditions, Vickers hardness was measured in order to endorse the hypothesis of artificial aging occurring during high temperature tensile tests. In order to study this effect, a series of specimens was aged at 235 °C for different aging times, and aging curves were plotted. The occurrence of static and dynamic aging was evaluated by comparing hardness values of tensile specimens and aged samples, particularly in the range of 15-20 min, as this range corresponds to the time necessary for pre-heating and testing of the tensile samples. A basic correlation between tensile strength and hardness is also given.

313 Control of physical properties of anodic coatings obtained by anodizing in aluminate solutions

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Titanium alloys have excellent physical and mechanical properties for a large number of applications. Some of these properties are due to a passive film which is formed spontaneously on the surface of those materials when they are exposed to environments containing oxygen. However, in some applications the passive film is not enough to protect the base material, particularly when corrosion and wear resistance are requested. Anodizing process allows the formation of an oxide layer which is denser and thicker than the passive film formed spontaneously. Moreover, such layer will improve corrosion and wear behavior of titanium alloys. It is well known that anodic layers obtained in alkaline solutions exhibit improved wear behavior; however, literature on the formation of thin anodic films with good mechanical and tribological behavior is very scarce. In this work, anodic films on Ti6Al4V were obtained in an aluminate containing solution with Na₂WO₄ addition. The coatings were obtained both in galvanostatic and potentiostatic mode. SEM, laser microscope, XRD, ICP, EIS and dry sliding friction tests were employed to study the morphology, thickness, chemical composition and performance of the coatings. Results showed that tungstate addition allows the formation of a

less rough anodic film and increases the dissolution of titanium in the anodizing solution during the process. The more suitable coatings for improving wear resistance are the anodic coatings formed at potentiostatic mode due their low porosity, despite the thickness is lower than in galvanostatic coatings, as demonstrated by the results of ball-on-disc wear tests.

314 Resistance upset welding of ODS steel fuel claddings - experimental and simulation approach

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Sodium Fast Reactors (SFR) are one of the most advanced concepts for Generation IV nuclear reactors. To ensure profitability of this concept, it is necessary to develop a high-performance fuel cladding. In this purpose, ODS steels (Oxide Dispersion Strengthened, reinforced by a nanometric distribution of Y and Ti oxides providing good high-temperature mechanical properties) are identified as candidate materials. In order to avoid oxide agglomeration in the melted bath during welding, a solid-state welding process is preferred to join the end plug to the cladding. However, under the effect of temperature and large strains imposed during the solid-state welding process, nano-oxides size and distribution are susceptible to change, which may affect the resistance to thermal creep and creep under irradiation. Microstructure also undergoes some modifications, and particularly highly strained zone located around the joint plane, which can be dynamically recrystallized depending on selected process parameters. In order to characterize and understand the effects of resistance upset welding thermo-mechanical cycles on ODS materials, an approach coupling welding experiments, numerical and physical simulation of the process is adopted. Finite element numerical simulation of welding is set up in order to assess the strains, strain rate and temperature locally imposed to the material around the join area during the process. In order to study the modification of nano-oxide and the recrystallization phenomenon as well as their conditions of occurrence, thermo-mechanical cycles in the range identified by numerical simulation of welding, are reproduced on compression and “hat-shaped” specimens on a Gleeble 3500 device.

315 Surface nitriding of beta-type titanium-based superelastic alloys for biomedical applications

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Superelastic and low modulus beta-type Ni-free Ti-based biomedical alloys were nitrided in surface in order to improve both the mechanical surface properties and the biocompatibility. The treated surfaces were characterized by X-ray diffraction, X-ray photoelectron spectroscopy, secondary ion mass spectroscopy, and the superficial mechanical properties were evaluated by nano-indentation and by ball-on-disk tribological tests. To investigate the biocompatibility, the corrosion resistance of the nitrided Ti alloys were evaluated in simulated body fluids (SBF) complemented by in-vitro cytocompatibility tests on human fetal osteoblasts. After treatment, surface analysis methods revealed the formation of a titanium-based nitride on the substrate surfaces. Consequently, an increase in superficial hardness and a significant

reduction of friction coefficient were observed compared to the non-treated samples. Also, a better corrosion resistance and a significant decrease in ion release rates have been obtained. Cell culture experiments indicated that the cytocompatibility of the nitrided Ti alloys was superior to that of the corresponding non treated samples. Thus, these new functional titanium-based superelastic alloys present the optimized properties that are required for various medical devices: superelasticity, high superficial mechanical properties, high corrosion resistance and excellent cytocompatibility.

316 Electron beam melting of Ti-6Al-4V: Effect of post-processing conditions on the microstructure and mechanical properties

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Electron Beam Melting (EBM) of Ti-6Al-4V alloy is a powerful additive manufacturing process that allows complex parts to be produced. There remains however many challenges if this process were to be used to build critical parts for aeronautic applications. In this work, the microstructures and mechanical properties of as-built parts were scrutinized. Moreover, the effect of thermal and mechanical post-treatments was extensively studied. In particular, the consequence of Hot Isostatic Pressing (HIP) was investigated. The as-built microstructure consisted of fine alpha lamellae at room temperature. Numerical reconstruction of the parent beta phase highlighted the columnar morphology of the grains, growing along the build direction upon solidification of the melt pool. Texture analysis of the reconstructed high temperature phase revealed a strong <001> pole in the build direction. The influence of surface finish on tensile properties was highlighted. Mechanical polishing induced an increase in ductility – due to the removal of critical surface defects – as well as a significant increase in yield strength – caused by the removal of a rough surface layer that can be considered as mechanically inefficient. Thermal post-treatments were also performed. Subtransus treatments induced moderate coarsening of the microstructure, whereas supertransus treatments led to the fast growth of the beta grains. HIPing of the samples below the transus led to coarsening of the microstructure and removal of porosities within the bulk of the samples. It is shown that HIPing must be followed by surface machining in order to reach large ductility and fracture strain levels.

317 Design of functional oxide nanomaterials: From nanoparticle synthesis to original densification route

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The design of functional nanomaterials may include a strategy either to synthesize nanoparticles or to elaborate densified nanostructured materials or both, depending on the targeted application. For example, the use of manganite perovskites for spintronic application in 0-3 type nanoarchitected composites needs.

(1) to master the chemical composition, the size of manganite nanoparticles : Aqueous self-combustion will be discussed as a powerful tool to elaborate such materials with an accurate

control of prevalent parameters. The molten salts synthesis will be presented as a suitable alternative. (2) to tune the coating of nanoparticles with an insulating shell : As obtained manganite nanoparticles will be coated with silica. (3) to consolidate 3D assemblies of as-obtained core@shell nanoparticles without any coalescence effect or reactivity at the interfaces : The hydrothermal sintering process was first developed by N. Yamasaki from Kochi University. Based on dissolution-precipitation reactions at the interfaces between solid and liquid, it is useful to densify non sinterable materials by ordinary sintering routes, such as hydrates, carbonates, hydroxides in a short time at a relatively low temperature. Because the chemical reactions are expected to be located at the surface of the grains, this process appears powerful to densify nanometric materials without any grain growth. The optimization of the process will be done on silica nanoparticles in order to transfer the know-how to core@shell particles.

318 Development of production of heavy TMCP plates up to 100 mm at NLMK DanSteel for construction, offshore and shipbuilding purposes

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At the end of 2012 a new rolling 4200mm mill at NLMK DanSteel was put into service. Since the company has focused on production of heavy plates for construction, offshore and shipbuilding purposes. The enterprise is consistently expanding assortment as regards sizes and grades of steel used for production of heavy plates. For example, at the end of 2014 the enterprise expanded the range of TMCP rolled heavy plates, namely structural steel of strength category S355ML, S420ML, S460ML up to 100 mm and received certificates. The same was done for TMCP plates for fixed offshore structures and shipbuilding.

The report presents slab quality requirements, features of production technology for specified quality and thickness categories as well as microstructure investigations, enhanced mechanical test results, including evaluation of the anisotropy of the hardness gradient, strain aging properties and toughness up to -80°C in different thickness layers, showing the actual level of cold resisting properties of produced steels. Investigations of welded joints with different heat inputs and for different chemical compositions of steel also will be presented.

319 Developing of the finite element model for temperature field and distortion during friction stir welding

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The purpose of the present research is to develop the finite element model for temperature field and distortion during friction stir welding (FSW). Experimental FSW of 2 mm sheets was provided for temperature and distortion determination. Aluminium alloy AW 6082 was chosen as a material for workpieces. Temperature was measured by thermocouples. The 3-D geometry of sheets before and after friction stir welding was measured by means of a precise robotic

system for distortion determination. An analytical heat source model developed before was improved by taking into account not only the friction at the contact surfaces or close to the contact surfaces under the tool shoulder and at the tool probe sides but also the influence of the plastic deformation in the weld nugget on the heating of the workpieces and asymmetry of the heat source. The temperature distribution and distortion was calculated by means of developed finite element model. The simulation model was compared with experimental results. The results of numerical simulation are in the good agreement with the experimental results.

320 MAX phases: New materials for high temperature applications

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MAX phases are a new family of materials with general formula of $M_{n+1}AX_n$, where M corresponds with an early transition metal, A is a A-group element, X is C and/or N, and n is typically equal to 1, 2 or 3. They are promising candidates for high temperature applications due to their unique combination of properties, bridging the gap between ceramics and metal materials. In general, MAX phases are lightweight, elastically stiff, good thermal conductors and resistant to oxidation and chemical attack at high temperatures as carbides and nitrides ceramics, while they are soft, readily machinable, thermal shock resistant and damage tolerant as metals. In this work, Cr_2AlC and Ti_2AlC are the selected MAX phases due to their excellent response at high temperature. Synthesis of these pure phases is the main challenge, since secondary phases alter the final response of the material. Synthesis, processing, densification, microstructural characterization and oxidation resistance of porous and dense structures of Cr_2AlC and Ti_2AlC MAX phases will be presented. Furthermore, a demonstration of a potential heat exchanger based on porous Ti_2AlC will be shown.

321 Role of microstructure on mechanical properties of ultrafine grained Cu processed by different ECAP pass-numbers

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Equal channel angular pressing (ECAP) is one of the most promising techniques for producing bulk ultrafine grained (UFG) materials with grain sizes ranging from ~100 to 1000 nm. The UFG microstructure processed by ECAP is sensitive to the parameters of the procedure, such as route, number of passes, deformation rate, shape and dimensions of the die and processing temperature. Therefore, these factors could have an effect on the mechanical properties. So far, a large number of data on mechanical properties, such as tensile strength and fatigue strength, of UFG copper have been disclosed by various laboratories. For a simple comparison of data between different laboratories, however, we were confronted with a significant difficulty due to differences in test conditions, such as the parameter of the ECAP procedure, type of fatigue test loading, shape of the specimen and trace differences in material purity. However, studies on the effect of the ECAP parameters on the mechanical properties of UFG copper under the

same testing condition have been relatively rare, and only a few reports can be found. In the present study, tensile and fatigue tests of oxygen-free copper (99.99 % Cu) processed by ECAP through 4, 8 and 12 passages were carried out under the same test conditions. The objective of this study was to investigate the effect of number of passes on the mechanical properties of UFG copper from the viewpoints of microstructural evolution during ECAP processing.

322 Influence of the microstructure on the shot peening of automotive springs

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Shot peening is a well-established method to increase the fatigue properties of springs. The effectiveness of the shot peening process is known to be influenced by process parameters such as shot size, velocity and coverage. In the present study, we examine the relationship between the peening process and the microstructure of the workpiece. Specimens with two different microstructures, tempered martensite and bainite of similar hardness have been produced and then subsequently peened. The microstructure characterization by means of electron backscatter diffraction shows a clear difference in the shot impact on the microstructure. Due to plastic deformation, bainite exhibits high and localized lattice rotation and high local misorientation values, while the tempered martensitic microstructure accommodates the plastic strain more homogeneously. Finite element modelling of the shot peening process reveals that shot peening of the two materials under the same peening conditions results in a different residual stress field. The modeling results are in good agreement with the results of the residual stress measurements by X-ray diffraction. It was found that the shot peening produces a deeper stress profile on the springs with bainitic microstructure than on the tempered martensite. On the other hand the tempered martensite shows higher maximum compressive stress after shot peening. The findings described above are expected to affect both the optimum peening conditions and the fatigue performance of the produced springs, to be established in relation with the microstructure features.

323 Lightweight titanium metal bipolar plates for PEM fuel cells

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Bipolar plates (BPPs) serve multiple roles in polymer electrolyte membrane fuel cells (PEMFCs). When assembled in a stack, they provide the structural backbone of the stack, plus serial electronic connections. They also provide gas (air and fuel) and coolant distribution pathways. Traditionally, bipolar plates have been made of carbon, but these are being replaced in favor of metal bipolar plates made of stamped foils. The Naval Research Laboratory has explored making titanium metal BPPs using 3D printing methods (direct metal laser sintering – DMLS) and superplastic forming, and then using a gold/TiO₂ surface layer for corrosion resistance. The 3D printed plates are made as one piece, with the coolant flow internal to the resulting 2-mm thick structure. Their surface roughness requires smoothing prior to coating to

increase their cell-to-cell conductivity. We found that 3D printed cells with 22 and 66 cm² active areas were slightly warped, preventing the robust sealing of the stacks. The formed plates are made in separate pieces and then joined. Despite the high temperatures required for superplastic forming, the resulting plates are thin and lightweight, making them highly attractive for lightweight compact PEMFC stacks.

324 Microstructure-property relationships in medium-Mn steels with metastable retained austenite

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Medium-manganese steels for the automotive industry have received much interest in recent years due to their beneficial balance between strength, ductility, formability and capability of energy absorption under dynamic deformation conditions. Their mechanical properties depend on relative volume fractions and mechanical features of individual structural constituents and a progress of strain-induced martensitic transformation of retained austenite embedded in carbide-free bainite. The study addresses relationships between the microstructure and mechanical properties of thermomechanically processed bainitic steels containing 3% and 5% Mn. The steels contain blocky-type and interlath metastable retained austenite embedded between bainitic ferrite laths. To monitor the transformation behaviour of retained austenite into strain-induced martensite interrupted tensile tests were applied. The identification of morphological features of retained austenite and strain-induced martensite was carried out using scanning electron microscopy (SEM) equipped with EBSD (Electron Backscatter Diffraction) and transmission electron microscopy (TEM). The amount of retained austenite was determined by XRD. It was found that manganese addition strongly affects mechanical stability of retained austenite resulting in a different degree of TRIP effect in the investigated alloys.

325 Novel Ti-25Ta-Zr alloys for biomedical applications

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Due to the excellent biocompatibility together the corrosion resistance of titanium alloys, their application in the area of orthopedic and dental implants have gained strength since 1970. However the Young's modulus of these alloys is still about 2 to 4 times higher than the human bone, and may cause stress shielding effect. The most widely used titanium alloy for biomedical applications is Ti-6Al-4V, however, previous studies showed that vanadium cause allergic reactions in human tissue and aluminum has been associated with neurological disorders. So, to solve this problem, new titanium alloys without the presence of these elements are being developed, with the addition of different elements, usually the β -stabilizers, which can change its microstructure and mechanical properties, and may make the titanium and its alloys, most promising for use as biomaterial. In this paper the development and characterization of Ti-25Ta-(10-40)Zr alloys, for biomedical applications are discussed. (Financial support: Capes, CNPq and FAPESP).

326 Fracture toughness investigations of a ferritic-austenitic steel deformed by high pressure torsion

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Duplex steels are two phase steels with nearly equal amounts of ferritic and austenitic phase. The application fields are constantly increasing because of their excellent combination of high strength and fracture toughness complemented with superior resistance to localized corrosion and stress corrosion. A further optimization of the material can be realized by nano-structuring as ultrafine-grained and nanocrystalline metals have shown improved mechanical and physical properties compared to their coarse grain counterparts. Therefore, a conventional duplex steel has been deformed by high pressure torsion. The evolution of the microstructure as a function of deformation has been investigated via Scanning Electron Microscopy and Transmission Electron Microscopy. The change in hardness was examined by micro-indentation hardness testing. Special focus has been on the variation of fracture toughness and ductility as a function of pre-strain induced by high pressure torsion. In order to take texture and grain shape changes during deformation into account, specimens with different orientations with respect to the principal shear deformation direction have been tested. The results will be compared to single phase austenitic and ferritic nano-structured materials exhibiting extreme differences in their fracture and deformation behavior.

327 Laser ultrasonic characterization of aluminium alloy coatings

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The non-destructive characterization of Al alloy coatings is a subject of constant importance in the fields of electronics, aircraft and automotive industries due to the many beneficial properties of aluminum e.g. high reflectivity, high conductivity, high corrosion resistance and low costs. In this work, a contactless laser ultrasonic (LUS) method is presented to determine the elastic properties of AlCu films of few micron thicknesses on silicon (100) substrates. To this purpose, nanosecond laser pulses were focused with a cylindrical lens onto the sample surface in order to excite plane, broadband surface acoustic waves (SAW). The excitation of broadband SAWs allows for the evaluation of the frequency dependent phase velocity, because low frequency SAWs propagate mainly in the substrate whereas higher frequency waves propagate mostly in the coating. Therefore, the phase velocity depends on the frequency as a result of the different SAW velocities of substrate and film [1]. An optical beam deflection method was applied to detect the SAWs, using a continuous wave laser, and the frequency dependent phase velocity was determined. The Young's modulus and the Poisson's ratio were derived by fitting the results of a theoretical model to the experimental evaluated dispersion curve. For validation, nano-indentation measurements on samples of the same wafer were performed. The nano-indentation outcome is comparable to the LUS results, identifying the presented non-contact approach as a tool of choice with high potential for in-line material characterization and reliability inspection of Al alloy coatings.

328 Mixed-valence vanadates at high pressures

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Many elements occur in more than one valence state in ionic compounds to form mixed-valence materials. The ability of an ion to change its valence state lies at the core of the functionalities important to materials sciences. Physical properties are closely linked to the variability of the valence state or to the redistribution and/or ordering of the charges. While mixed-valence materials containing Mn or Fe are very well investigated, data on mixed-valence vanadates are comparatively scarce, although they are useful for catalysis and energy applications. Their phase diagrams are not extensively investigated and relatively little is known with respect to the influence of pressure/temperature variation on their structures and physical properties.

The aim of this presentation is to demonstrate how diffraction techniques and synthesis in diamond anvil cells and large-volume presses could be used to study phase transitions and chemical reactions in vanadate systems in order to design and optimize subsequent materials synthesis at high pressures and high temperatures. It will discuss determination of not only crystal structures but also phase diagrams. The interplay between the effects of chemical substitutions and of external pressure on the stability of mixed-valence vanadates will be elucidated.

329 Combination of microstructural investigation and simulation during the heat treatment of a creep resistant 11% Cr-steel

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This work deals with the microstructural evolution of creep resistant martensitic/ferritic 12% Cr steel during thermomechanical treatment from an experimental as well as modelling point of view. The creep resistance of this material group is highly dependent on the precipitate status. The initial precipitate status is controlled by the chemical composition of the alloy as well as the heat treatment after casting or hot rolling. It is therefore of utmost interest to understand and model the precipitate kinetics during this process. Once the microstructural evolution has been modelled successfully, only minimum effort is required to computationally test variants in the composition or heat treatment in order to optimize the process. In this work, the material was hot rolled, austenitized and subsequently annealed. All heat treatments were performed during dilatometry tests in order to monitor phase transition temperatures. In order to investigate the microstructural evolution during the process, samples were extracted at definite stages of the treatment. The samples were then investigated applying various microscopical techniques in order to quantify the microstructural features (grain size, martensite lath width, precipitate data). The experimental data were then compared to thermodynamic simulations (MatCalc). General data such as nucleation sites for precipitates were taken from literature, grain size and martensite lath widths from the experimental data. Simulations include equilibrium calculations and precipitate kinetic simulations. In general, the simulations showed good agreement with the experimental findings, with minor room for improvements. The work thus lays a solid ground for future improvements of the heat treatment process.

330 A solid-state chemistry approach to design spinel cobalt oxides with high electronic conductivity for the positive electrode of asymmetric aqueous supercapacitors

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An increasing number of studies is devoted to the field of asymmetric supercapacitors, aiming at increasing both energy and power densities, in order to match specific applications of reversible energy storage in the fields of space, aircrafts, transportation. The MnO₂/carbon system is the most investigated system, due to its capability to work in aqueous medium at potentials up to 2 V, as well as to the low cost and environmental friendliness of manganese. Nevertheless, this system suffers from the poor electronic conductivity of manganese oxides and the limited ion diffusion/transport through the electrode, which limits the performances especially in terms of power. A solid state chemistry approach was used to design highly conductive nanometric cobalt oxides, with original structure (spinel with defects) and composition. The presence of Co⁴⁺ in the octahedral trivalent cobalt network entails electronic delocalization and very good electronic conductivity. Assembling our cobalt-based optimized positive electrodes with negative microporous active carbon in complete supercapacitors, leads to promising capacities in alkaline medium.

331 Assessment of creep tendencies in Cu-Al thin wires: correlation with pure Cu and Al behaviors

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Cu-Al bimetallic wires are good candidate materials for electrical industry since the cost increase of polycrystalline copper. Using aluminum as core instead of copper reduces the product price and decreases the overall weight structure. Moreover, the copper external layer gives excellent electrical conduction properties and good efficiency for high frequency applications. However, Cu-Al wires are subjected in automotive to severe conditions such as heat and stresses in the vicinity of the engine. This environment involves creep aging process that modifies the properties of the composite. The aim of this work is to assess the mechanical behavior of Cu-Al thin wires in comparison with corresponding pure copper and aluminum wires. Creep tests were carried out for 5 days at 423 K on 300  m diameter wires. Comparison of steady-state strain rate on raw and annealed Cu-Al wires shows a similar trend than aluminum for stress level typically below half of the yield stress. Annealed Cu-Al wires display this trend on the overall stress range of the study. However, raw wires strain rate approaches the one of copper for stress level above half of the yield stress. Microstructural observations coupled with X-Ray analysis in temperature were performed in order to correlate the mechanical behavior with the metallurgical evolution of the material. Results indicate that the creep rate of the Cu-Al wire is intimately related to the Cu and Al behavior rather than that of the intermetallic growing at the interface.

332 Microstructures and thermo-physical properties of thermal barrier coatings produced by PS-PVD

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Plasma spray physical vapor deposition (PS-PVD) is a newly developed technology, which reveals promising potential in processing advanced functional coatings such as thermal barrier coatings (TBCs) due to its capability in tailoring coating microstructures in a broad range. In this talk, the microstructures and thermo-physical properties of TBCs by PS-PVD were investigated. The coating revealed a novel quasi-columnar structure when deposited at spray distances between 1200 and 1400 mm. A microstructure evolution model along axial direction was built and associated mechanism for the formation of microstructure discussed. The thermal conductivities of the TBCs were in a range of 0.8~1.2 W/mk, which is relatively lower than those of the TBCs produced by electron beam physical vapor deposition (EB-PVD). Also, the PS-PVD TBCs exhibited several advantages in durability and mechanical properties.

333 Structural evolution of Cu-Fe alloys deformed by high pressure torsion

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Usually immiscible Cu-Fe system can be mechanically alloyed to form single phase, i.e. by means of high pressure torsion (HPT). Micrometer-sized Cu and Fe powders were directly used as starting materials to be mixed as (1-x) at.%Cu - x at.%Fe (x = 0, 5, 15, 25, 35) series of compositions. These Cu-Fe alloys with different atomic percentage of constituents were systematically investigated to get insights into the structural evolution of nanocomposites and to determine the solid solubility limit of Fe atoms in Cu matrix under HPT deformation. Finally, all investigated compositions of (1-x) at.%Cu - x at.%Fe with single face-centered cubic structure was successfully formed after two stages of deformation according to X-ray diffraction results. The exact limit of solid solubility for Fe atoms into Cu matrix was extended to 35 at.%Fe by HPT method. Grain size statistics from transmission electron microscope image shows that nanostructured Cu-Fe crystalline was obtained with the average size of 68 nm for 85 at.%Cu - 15 at.%Fe. Typical 'saturated softening' of micro-hardness was observed in Cu-Fe supersaturated area. And the possible mechanism of the dissolution process of Fe atoms into Cu matrix was discussed.

334 Modelling the transition from upper to lower bainite in multicomponent steels

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A model for estimating the upper to lower bainite transition has been developed for iron-carbon-manganese-chromium-silicon alloy system by comparing the time required to decarburise a supersaturated bainitic ferrite platelet and the time needed for precipitation of cementite inside it. The decarburisation time is calculated using published diffusion theory whereas the competitive kinetics of cementite precipitation inside bainitic ferrite is modelled using MatCalc. The time for forming a volume fraction of 0.01 of equilibrium amount of cementite is taken as the precipitation time. The model was calibrated using experimental data from iron-carbon system, and verified experimentally on a particular steel developed by Swiss Steel AG.

335 Enhanced mechanical properties of bulk graphene/aluminum composites with a bio-inspired nanolaminated structure

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Bulk graphene-reinforced Al matrix composites of various reinforcement concentrations were fabricated via a modified powder metallurgy approach. These composites possess a nanolaminated, brick-and-mortar architecture, where layers of ~200nm-thick pure Al platelets are stacked in a staggered arrangement, and are separated by graphene sheets, each containing 4-5 graphene monolayers. The composite containing 1.5 vol. % graphene were shown to have an uniaxial tensile strength of 302 ± 3 MPa, about 50% higher than that of unreinforced Al matrix prepared using the same fabrication route (201 ± 6 MPa). Moreover, the composite possess a uniform elongation of $3.4 \pm 0.2\%$, only slightly lower than that of the Al matrix ($4.3 \pm 0.4\%$), and have a significantly lower strain hardening capability. Combined with post-mortem and in situ transmission electron microscopic (TEM) analysis, our findings were interpreted in terms of the uniform distribution of graphene in the Al matrix, the effective load transfer between the graphene sheets and Al platelets, and the interaction between mobile dislocations and the graphene-Al interfaces.

336 High performance magnesium based composites containing nano-length scale/amorphous/hollow reinforcements

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Magnesium is the lightest weight metallic material that can be used in multiple engineering applications and biomedical sector as a structural material. It is abundantly available in earth's

crust and sea water and non-toxic in nature. Inherent to magnesium is its superior specific mechanical properties, high damping, electromagnetic shielding capability and ability to reduce carbon signature of the transportation sector. Magnesium is one of the widely available metal in earth crust and sea water. It is non-toxic and hence does not pose a health risk during recycling or waste dumping in natural water bodies. As a result, magnesium technology is sustainable and beneficial to planet earth and living organisms. Magnesium based materials are gradually being used in many applications and their performance and applications can further be stretched using the composite technology. Accordingly, the main scope of this presentation will be to highlight the enhancement of a number of properties of magnesium through the use of nano-length scale metal and ceramic reinforcements.

337 Damage generation process in cast Al-Cu alloys during in situ room temperature tensile tests

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Al-Cu cast alloys are potential candidates for cylinder head production. They show higher tensile and low cycle fatigue strength than the widespread Al-Si alloys, approaching even some grades of ductile iron. However, they have been rarely used for serial production owing to castability problems. In this work, the evolution of the microstructure of cast B206 (AlCu4.8Mn0.24Mg0.33), AlCu6 (AlCu6.5Mn) and AF52 (AlCu4.7Mn0.4Ti) alloys is investigated by two dimensional and three dimensional methods as a function of solution treatment (ST) time at 530 °C. Furthermore the damage accumulation process during RT tensile tests is investigated three dimensionally by means of synchrotron tomography.

Two factors determine the damage generation process during tensile loading at room temperature: i) ST time and ii) the different kind of aluminides present in the microstructure. The first 4 h of ST result in the partial dissolution of highly interconnected aluminides segregated during casting. Since cracks tend to initiate and propagate through the aluminides network, the ST reduces the crack nucleation sites and propagation paths. Two different damage accumulation mechanisms were identified depending on the volume fraction of aluminides. Damage mainly accumulates from cracks generated at large aluminides oriented perpendicularly to the loading direction. These cracks can propagate through aluminides networks with high interconnectivity and connectedness (AlCu6). On the other hand, porosity growth with further crack propagation and coalescence through the aluminides network represents the other damage accumulation mechanism, proper of alloys with less aluminides (B206-partially and AF52).

338 Effect of boron addition on microstructure and mechanical behavior of AZ84 Mg alloy

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Effect of B addition on the microstructure and mechanical properties of AZ84 Mg alloy was investigated in this study. Through calculation of phase equilibria of AZ84 Mg alloy, carried out by using FactSage® and FTLite database, solution treatment temperature was decided as

temperatures from 400 to 450°C, where supersaturated solid solution can be obtained. Solid solution treatment of AZ84 Mg alloy was successfully conducted at 420 °C and supersaturated microstructure with all beta phase resolved into matrix was obtained. After solution treatment, hot rolling was successfully conducted by reduction of 60%. Compression and tension tests were carried out at room temperature on the samples in as-cast, solution treated, hot-rolled and recrystallized after rolling. After solid solution treatment, each alloy was annealed at temperatures of 180 and 200 °C for time intervals from 1 min to 48 hrs and hardness of each condition was measured by micro-Vickers method. Peak aging conditions were deduced as at the temperature of 200 °C for 10 hrs. By addition of B by 0.2 weight percent, hardness and strength of AZ84 Mg alloy were enhanced.

339 Machining of titanium alloys with vibration assisted milling

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Tool wear and tool life time are currently the restricting factors in machining of titanium alloys. A styrian consortium with experts in materials, tools and machining is therefore formed to find sustainable solutions. The aim of the consortium is the extension of tool life in machining of titanium alloys for the aircraft industry.

A series of tests is set up to evaluate the roughing and finishing operations. A benchmark study of milling tools for rough machining is undertaken in the AEC machining laboratory.

For finishing operations a new technology, the vibration assisted milling, is introduced and the cutting parameters for titanium alloys are tried out. For this, the transfer of knowledge of previously unmachinable ceramics is objective. An ultrasonic five-axis machining center is equipped with an oscillating tool that achieves microscopic impacts for the material removal.

Force and power measurement together with optical tool wear detection are used to determine best process parameters.

340 Novel approaches for aluminium magnesium diffusion bonding by surface engineering

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Diffusion bonding is a well-known technology for a wide range of advanced applications, due to the possibility to join different materials within a defined temperature - time - surface pressure - regime in solid state. To improve the diffusion kinetics and thus to improve the joint properties of aluminium and magnesium joints at lower bonding temperatures, surface engineering is promising possibility. Mechanical, mechano-chemical and physical surface treatments were used to decrease the joining temperature and improve the joint properties, respectively. In this regard all surfaces were analysed by tactile surface measurement, contact angle measurement and XRD-analysis to characterise the surface before joining and to examine the influence of the surface conditions with regard to the joint properties. The results have shown that the formation of the intermetallic Al-Mg phases can be limited in width to a few microns by bonding temperatures at 260°C through adjusted joining surfaces and bonding

parameters. Thus, the critical hardness peak on the Al-rich side of Al_3Mg_2 phase could be avoided. Furthermore, SEM and EDXS-analysis were used to examine the role of micrometre and nanometre thin interlayers during diffusion bonding of aluminium and magnesium. Finally, the examinations have shown novel approaches to improve the joint properties of Al-Mg-joints by different surface treatments.

341 Unraveling the age hardening response in U-Nb alloys

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Uranium-niobium alloys are corrosion-resistant and ductile when quenched from the high-temperature gamma-BCC phase. Since these properties degrade upon thermal aging up to 647°C, predictive models of age hardening are desirable. In spite of dozens of studies over the past 70 years, a comprehensive and consistent picture of U-Nb aging remains elusive. This work covers experimental and modeling studies that unravel some complicating factors that have stymied understanding, including (1) variety of phase transformations microstructures, (2) residual niobium inhomogeneity in commonly studied pedigrees, (3) effects of machining damage on tensile properties, and (4) early-time transients of ductility increase. We focus on separating out time-temperature-composition domains of different aging phenomena, which involves key decisions at the data reduction stage. The entire body of hardness and tensile data (in alloys up to 10 wt.% Nb) has been modeled using an Avrami-Arrhenius approach. The apparent activation energies will be compared with plausible physical mechanisms.

342 Casting of an Mg alloy clad strip using a twin roll caster equipped with a scraper

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Casting of an Mg alloy clad strip was tried using a twin roll caster equipped with a scraper in the atmosphere. A base strip was AM60 and an overlay strip was AZ121. The AM60 strip was cast by a roll and an upper side of the strip was scribed by a scraper. The molten metal of the AZ121 was poured on the scribed AM60 strip. The scribed surface of the AM60 contacted to the molten metal of the AZ121 without contact to the atmospheres. Therefore, the scribed surface must not be oxidized. The most of the AZ121 strip was solidified by the other roll. The casting speed was 30 m/min. The roll load was 0.1 kN/mm. The roll speed was very high and the roll load was very small. However, the strips were bonded. The strips were not peeled by a bending until broken. The interface between the two strips was clear. This result shows that the AM60 strip was not melted by the heat from the molten metal of the AZ121. The AM60 molten metal did not leak from between the scraper and the solidification layer of the AM60.

343 High-temperature deformation behavior of (Mo_{0.85}Nb_{0.15})Si₂ crystals with C40/C11b lamellar microstructure

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Transition-metal disilicides have received much attention because they are promising candidates for such ultra-high-temperature structural materials. We have proposed to combined different silicides that have similar properties to increase the fracture toughness and high-temperature strength. We succeeded in producing a C40/C11_b duplex-phase crystal with an oriented lamellar microstructure. In this study, high-temperature deformation behavior, including the compressive deformation behavior, of the (Mo_{0.85}Nb_{0.15})Si₂ crystals with C40/C11_b oriented lamellar microstructure was examined over 1000-1400 °C. The deformation behavior of the crystals was found to vary greatly depending on the orientation of the loading axis with respect to the lamellar interfaces. On the creep deformation behavior, the creep strain rate when the loading orientation was parallel to the lamellar interfaces (0°-orientation) was approximately 2 orders of magnitude lower than that when the loading orientation was inclined by 45° (45°-orientation). The results suggest that the C40-phase effectively acts as a strengthening phase in the creep behavior of the C40/C11_b duplex-phase crystals. The variant-1-type C11_b phase grains, which have a loading orientation parallel to [001], were also found to act as an effective strengthening component. The effect of additional alloying elements such as Cr, Zr, Ir to the high-temperature deformation behavior of the C40/C11_b duplex-phase crystals was also examined. The results demonstrated that the addition of them all leads to the decrease in steady state creep rate at 0°-orientation. The influence of variation in microstructure to the deformation behavior is discussed.

344 Z-phase strengthened steels - the European Z-ultra project

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The creep and oxidation resistance of the best available martensitic 9%Cr steels for advanced steam power plants limits steam parameters to 300 bar and 600-620°C. Steels with 11-12%Cr and better creep strength are needed to provide oxidation resistance for increased steam parameters and higher thermal efficiency. Previous attempts to develop improved 11-12%Cr steels failed due to long-term microstructure instabilities caused by precipitation of coarse particles of Z-phase (Cr(V,Nb)N) and dissolution of fine (V,Nb)N nitrides. The European FP7 project Z-Ultra aims to develop new creep resistant martensitic high Cr steels based on alloy design to form fine Z-phase precipitates, which can provide precipitation hardening in the long term. Within the project several series of Z-phase alloy trial melts for mechanical testing and microstructure investigations were produced. Microstructure data provide input to development of materials models on several length scales. Trial melts were melted by induction melting, re-melted by vacuum-arc-remelting and forged in two steps to 40mm square bars. The final heat treatment included hardening at 1100°C and a two time tempering cycle. Superheater tubes were produced and installed in power plants to investigate creep and oxidation behavior. A large turbine rotor part forging with a weight of ca. 12 tons was manufactured to demonstrate

producibility of real size components.

345 Damage modelling in a gamma-TiAl alloy during hot deformation

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The hot deformation behaviour of a TiAl TNM alloy was analyzed by determining the relationship between thermomechanical processing parameters and microstructure. Flow curves were obtained by physical simulation using a Gleeble 3800® device. Metallographic analysis was performed by means of light optical and scanning electron microscopy with electron backscatter diffraction. Various approaches to model the hot deformation behavior were used to identify the optimum hot working conditions for γ -TiAl low-pressure turbine blade manufacturing. Evolving microstructure and the flow softening behaviour of the material were correlated with flow curves and strain rate sensitivity values. The prediction of flow localization was performed by existing flow localization parameter α_{SJ} , the strain rate sensitivity parameter m and continuum damage models (Cockcroft/Latham, Brozzo and Ayada) coupled to finite element simulations. Microstructural investigations revealed that both, physical based as well as macro-mechanical damage models predict instabilities in the material at a high level of accuracy.

346 Effects of Ti addition on properties of Au-Nb-Ti alloys for MRI artifact-free biomedical applications

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INTRODUCTION Mismatch between the volume-magnetic-susceptibility (χ_v) of biomedical-devices and the surrounding human-tissue causes a serious artifact in MRI. The authors have developed Au-Nb alloys with χ_v values similar to that of human-tissue [approximately -9×10^{-6} , (-9 ppm)] and with excellent hardness of approximately 230HV (Vickers hardness). However, HV of the alloy is lower than those of typical strong alloys for biomedical applications. In this study, the effects of Ti addition on properties of Au-Nb-Ti alloy were investigated.

EXPERIMENTAL METHODS Au-xNb-yTi (x = 4, 8 and 12, y = 1, 1.5 and 2) alloy ingots were fabricated, and then rolled at 573 K. The total rolling-reduction was 50-70%. The rolled alloy plates were homogenized at 1273 K. Aging-treatments were performed at 873 K and 1073 K. The χ_v values were measured using a magnetic susceptibility balance; and the phase constitution was analysed using XRD. **RESULTS AND DISCUSSION** Only Au-12Nb-1.5Ti alloy in the 9 alloys exhibited excellent properties; the χ_v values after aging-treatment were close to -9 ppm, and the HV values were higher than 400. The phase constitution of the alloy after homogenization was single phase, and it showed sufficient rollability. Both Au₂Nb intermetallic compound (IMC) and unknown IMC were precipitated after aging treatment.

CONCLUSION Addition of Ti to Au-Nb alloy helps achieve a MRI artifact-free alloy with high hardness. **ACKNOWLEDGMENTS** A part of this work was financially supported by JSPS KAKENHI, Japan Science and Technology Agency, Tanaka Kikinzoku Research Fund and Terumo Foundation for Life Sciences and Arts.

347 Analysis of mechanical properties in nitrogen-added duplex stainless steels by nano-indentation and in-situ neutron diffraction

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For duplex stainless steels, which are composed of ferrite and austenite, nano-indentation tests were performed to measure elasto-plastic transition loads at a constant loading rate. To obtain intrinsic mechanical properties of individual ferrite and austenite grains without grain boundary effect, the combination method with EBSD and SPM were used during nano-indentation. To correlate the small-scale nano-indentation behavior to the macro-scale tensile behavior, an angular-dispersive in-situ neutron diffraction test was carried out using a residual stress analysis diffractometer equipped with a deformation device enabling tensile deformation. Lattice strains of various lattice planes in austenite and ferrite were determined as a function of the applied tensile stress. As a first result, the tendency of plastic yielding in macro-scale could be well described by the nano-indentation data for each phase. To compare the initial yield stress, we used a maximum shear stress underneath indenter tip when first pop-in occurs is considered as elasto-plastic transition by dislocation nucleation. In the early stage of neutron diffraction data and nanoindentation data are related to the elastic deformation corresponding to single crystal elastic constants of each phase. Here, we obtained the anisotropic elastic constants by neutron diffraction data and self-consistent elasticity considering ODF. And then, the nanoindentation modulus for each grain can be predicted by using the obtained anisotropic elastic constants. In addition, the evaluation of stacking fault energy (SFE) based on the nano-indentation was tried and compared with the measurement data from neutron diffraction.

348 Fabrication of copper pattern with high adhesion via nano-structuring of PET substrate

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Copper patterns on flexible polymer substrates have been widely used for wearable electronics and display panels. To date, a lot of efforts have been focused on enhancing the adhesion between copper layer and polymer substrate. Among them, surface modification of polymer by plasma and inserting of tie layer between copper and polymer have been generally used. In this work, to increase the adhesion strength between copper layer and polymer substrate without any tie layers, nano-structuring of the polymer surface was designed via varying the oxygen plasma treatment time. PET (polyethylene terephthalate) was used for a polymer substrate, and copper layer was coated on PET using electroless plating. To elucidate the adhesion properties between copper and PET, peel strength and bending fatigue property were evaluated and effects of morphology of the surface nano-structure on the peel strength and bending fatigue property were examined through microstructure analyses using SEM and EDS.

349 Development of Zr-Mo alloy with low magnetic susceptibility for spinal instruments to decrease MRI artifact

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In this study, we have attempted large-amount melting of Zr-1mass%Mo alloy and investigated mechanical properties, crystal phase, and magnetic susceptibility, for the commercialization of the alloy. Finally, MRI artifact of spinal instruments consisting of the alloy and implanted in sheep spine was observed. The large amount melting was succeeded because molybdenum in the alloy existed in a range of 1.06-1.14mass% in all region of the rod. XRD revealed that the phase was almost a. Mass magnetic susceptibility was $0.98 \times 10^{-6} \text{ cm}^3\text{g}^{-1}$ as low as the previous study. Elongation to fracture of the alloy was 23% and much larger than that of Ti-6Al-4V. Crystal structure of the alloy was identified as follows. Regions with a width of 3 mm where dislocation density was low were surrounded by a network. The former consisted of a phase contributing to plasticity and the latter consisted of b and w phases contributing to strength. Artifact less than 2 mm was observed on 3T MRI image of the spinal instruments and the vertebral canal appeared. Zr-1Mo alloy after large-amount melting showed large strength and elongation and low magnetic susceptibility. 3T MRI artifact of spinal instruments consisting of the alloy and implanted into sheep spine occupied in 2 mm and the vertebral canal appeared. Therefore, this alloy is a candidate of MRI compatible alloy.

350 Effect of severe plastic deformation behaviour of aluminium alloys on friction surfacing process characteristics

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Friction Surfacing (FS) is a solid state joining process, in which a rotating stud is pressed onto a substrate, leading to plastification of the stud material through heat and shear stresses. Adding a translational movement, the plastified material is deposited as coating layer. The relative motion between stud and substrate is accommodated by deformation within the plastified stud material, which adheres to the substrate. This material therefore undergoes severe plastic deformation (SPD) at high temperatures ($0.8T_{\text{melt}}$), leading to complete dynamic recrystallization. In this study, Aluminium alloys 5083 and 6082 were processed by FS, and operable process parameters, coating dimensions, process forces, process temperatures and the resulting microstructure were compared. For 6082 a wide operable parameter window exists. For 5083 lower rotational and translational speeds are needed to avoid discontinuities in the deposition. To achieve a 3 mm thick coating, for 5083 500°C are required, and for 6082 440°C are sufficient at a 10% higher torque. Plastic shear in 5083 proceeds more localized due to thermal and strain softening, resulting in lower shear stresses, higher temperatures and thinner coatings. Deformation rates are limited for 5083, due to flow instabilities. The higher content of alloying elements in 5083 slows down dynamic recovery processes, which are the mechanisms allowing for the high degree of strain involved in FS. This study shows that FS

can be used to investigate material behaviour under high-temperature, high-strain rate thermomechanical processing, providing a technically feasible process for the generation of SPD materials in the shape of coatings.

351 Effect of friction stir processing on the damage resistance of 6xxx series aluminium alloys

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Damage evolution in ductile metals is characterized by the nucleation, growth and coalescence of small internal voids. In aluminium alloys, the void population generally nucleates by the decohesion or fracture of the iron rich intermetallic particles.

Previous studies have shown that the nucleation stress increases when the size of the intermetallic particles decreases retarding the final fracture of the material in tension. Furthermore, initial porosities and particle clustering reduce the final fracture strain as they favor earlier void coalescence by internal void necking.

Hence, friction stir processing (FSP) has been applied to a 6xxx series aluminium alloy in order to assess the ability of the process to eliminate initial porosity, fragment the intermetallic particles and distribute them more homogeneously to improve the fracture strain of the material. Detailed microstructural analysis of the intermetallics distribution has been carried out including 3D X-ray tomography and SEM micrographs. The mechanical properties have been investigated by tensile testing under various heat treatment conditions. In addition, a statistical study of void nucleation has been performed on 2D metallographic sections of interrupted tensile tests, as well as on 3D microtomography scan of fractured tensile samples.

This analysis confirmed that the size of intermetallic particles is reduced by FSP and that void nucleation is delayed. Furthermore, elimination of initial porosity and homogenization of the intermetallics distribution has been observed and quantified. Tensile tests have confirmed that these microstructural changes result in improved fracture strain.

352 Pre-hardened engineering and tool steel

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Pre-hardened engineering and tool steel. Per Hansson, Magnus Andersson. SSAB Special Steels. During the last decades has a rapid development in hard-machining taken place which enable steel manufacturers to develop a range modern pre-hardened engineering & tool steels which have leaner chemical compositions when compared with conventional steel grades commonly used in comparable applications such as moulds/dies. Use of pre-hardened grades enables faster, and also cheaper, component/tool manufacturing thanks to the elimination of heat treatment (Q&T) during component/tool manufacturing. Furthermore, the possible elimination of heat treatment during component/tool manufacturing also means less impact on environment due to elimination of transport to/from the heat treatment shop. Today's modern pre-hardened engineering & tool steels are delivered at nominal hardness ranging from 300 HBW up to 450 HBW (45 HRC) i.e. having Approx. yield strength ranging from 850 to 1300 MPa. The design of such pre-hardened steels, chemical compositions and processing routes, give steel having excellent machining properties combined with very high dimensional

stabilities when subject to machining. Most of the machining can actually be made in only one (1) set-up. Thanks to an extremely advanced steel making and slab casting can very high steel cleanliness in combination with low segregation levels be achieved, comparable with ESR-processed high cleanliness tool steel. Tools made from such steel can be polished to very high demands, which is of paramount interest for moulding applications. When necessary can attractive surface properties of a component/tool be tailor-made via use of surface engineering methods like Nitriding, PVD-coating etc.

353 X-ray and neutron scattering studies of the 9% Ni cryogenic steel and its weld joint

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The growing needs for energy lead to the increase of the demand for liquefied natural gas (LNG). Cryogenic materials used for the manufacture of the inner tanks for LNG storage are composed of 9% Ni steel plates with excellent properties for a high safety level. To guarantee the reliability and safe operation of large-scale metallic structures, it is important to evaluate structural properties of the base metal as well as the welded zone. Spatially-resolved high-energy synchrotron diffraction, pulsed neutron diffraction and small-angle neutron scattering have been used to study short, intermediate and mesoscopic range order in 9% Ni steel as well as the structural changes during welding. Pristine 9% Ni steel appears to be a metastable Fe-Ni alloy with main bcc martensitic and minority fcc austenitic phases. The fraction of austenitic domains changes between 1 and 6 % depending on thermal history and mechanical treatment of the samples. The austenitic phase forms mesoscopic domains with a radius of 600 Å and is enriched with nickel. Welding of 9% Ni steel using SMAW technique has reveal the existence of Heat Affected Zone (HAZ) of typical length 5 ± 1 mm. The HAZ are characterised by enhanced Ni concentration having a maximum at the welding interface and decreasing inside the welded sample. The Ni concentration gradient is related to fast intergranular nickel diffusion at high temperatures during welding and originated from high nickel content in the SMAW welding alloy. This processe increase the ductility of the HAZ and is beneficial for 9% Ni steel welding.

354 Interface storage and diffusion of sodium ions in titania-based Na-ion battery anodes

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Ordered, nanostructured transition metal oxides are currently in the spotlight of battery research. To exploit, however, the full potential of self-assembled titania nanotube layers in Na-ion battery technology we need to close the gap in knowledge about the underlying storage mechanisms as well as ion dynamics. For this purpose, amorphous TiO₂ nanotubes (70-130 nm in diameter, 4.5 – 17 µm in length) were fabricated by anodization, i.e., electrochemical oxidation. The electrochemical behaviour with respect to sodium insertion was studied by cyclic voltammetry and galvanostatic cycling with potential limitation. It turned out that the nanotubes are very resilient to cycling, some being able to withstand more than 300 charge-

discharge cycles without significant loss of capacity. Compared to classical insertion reactions, the mechanism of reversible electrochemical storage of Na^+ in the nanotubes seems to differ significantly. There is strong evidence that a large amount of the Na^+ ions is stored on the surface or in the surface-influenced regions while only a smaller fraction is inserted in the walls of the tubes. The interfacial Na^+ storage is governed by a faradaic adsorption phenomena, which is essentially different from the (pseudo-)capacitive model hitherto used in the literature. The finding that, in the case of sodium, interfacial storage is more important than ion insertion into the crystal lattice is supported by the very low Na^+ diffusion coefficients determined at various electrode potentials (in the order of $4 \cdot 10^{-20} - 10^{-21} \text{ cm}^2/\text{s}$).

355 Ultrafine-grained multifunctional titanium alloys

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Recently significant effort has been made in designing multifunctional titanium alloys with high strength and low modulus for biomedical application. The most promising of these alloys, called Ti2448 from its composition of Ti-24Nb-4Zr-8Sn in weight percent, has been developed. This alloy possesses good bio-mechanical compatibility of high strength and low elastic modulus closing to cortical bone as well as good bio-chemical compatibility of containing non-toxic and non-allergic alloying elements. Bone plates and spinal fixations made of the alloy have been finished clinical trials and applied for product licenses to State Food and Drug Administration of China. To further improve its bio-compatibility, a warm processing technique was proposed to refine the grains. Here gives a brief review of the warm process and the corresponding microstructure evolution and then focuses on its advantages to improve superelasticity and strength under the condition of good ductility.

356 Vertical free-standing ferromagnetic MnAs/semiconducting InAs heterojunction nanowires

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Magneto-electronic or spintronic nanowire devices are of great interest, since they offer a broader range of functionalities in the current nanowire devices. The author has demonstrated the bottom-up formation of ferromagnetic MnAs nanoclusters by selective-area metal-organic vapor phase epitaxy on partially- SiO_2 -covered GaAs (111)B substrates [1] and hybrid nanowires between ferromagnetic MnAs nanoclusters and vertical free-standing III-V compound semiconductor nanowires [2] to create novel spintronic nanowire devices, such as spin-nanowire-MOSFETs and LEDs. This invited paper focuses mainly on recent results of vertical free-standing MnAs/InAs heterojunction nanowires. It is possible to control the crystallographic orientations and the number of nanoclusters in and/or on InAs nanowires by the temperatures for endotaxial MnAs nanoclustering and/or the nanowire period. The $\langle 0001 \rangle$ direction of hexagonal NiAs-type MnAs nanoclusters grown at 580°C is parallel to the $\langle 111 \rangle_{\text{B}}$ direction of zinc-blende-type InAs nanowires, which are concluded from the characterization results using transmission electron and magnetic force microscopes. MnAs nanoclusters are not formed only in the middle of InAs nanowires, but also on the top $\{111\}_{\text{B}}$ facets of InAs

nanowires. MnAs penetrates through the InAs nanowires to form nanoclusters in the middle of nanowires. The surface migration length of manganese ad-atoms on nanowires, which is estimated to be approximately 600 nm at 580 °C, is a key to controlling the number of nanoclusters in InAs nanowires and successfully creating the vertical MnAs/InAs heterojunction nanowires with atomically-abrupt heterointerfaces. [1] Adv. Mater., 26, 8079 (2014); [2] Jpn. J. Appl. Phys., 51, 11PE01 (2012).

357 Surface modification of magnesium alloy by shot lining and laser heating

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Magnesium alloys are widely used in the automotive and aircraft fields. They have excellent physical and mechanical properties, such as low density and high strength-to-weight ratio. However, their poor corrosion resistance has been the main impediment in their applications. Surface modification is the main methods to control the degradation rate of magnesium alloys. This problem has been studied by many researchers. The authors have proposed a lining process of metals with thin aluminium foils using shot peening. In this method, the foil can be bonded to the workpiece surface bringing about large plastic deformation. The pressure generated by the hit of many shots is utilized for the bonding. In the present study, to improve the surface characteristics of magnesium alloy, the formation of an Fe-Al intermetallic compound film on magnesium alloy by compound treatment combining shot lining method and heat treatment was investigated. Shot peening was performed with a centrifugal-type machine using cast steel ball. The lined sheet is aluminum foil with pure iron powders, and the workpiece was the commercial magnesium alloy AZ31. The lining experiment was performed at 573 K in air. The lined workpieces are heat treated by laser in air. The Vickers hardness test was performed with a microhardness tester. It was confirmed that the present method could be used for the formation of functional films on magnesium alloy.

358 Nonlinear optimization methods for the determination of heat source model parameters

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In the past decades the importance of welding simulation continually grew, since it shortens the development process by reducing the experimental efforts. In addition it provides insight into the process which only can be observed with additional costs. One major issue is the adaption of surrogate model parameters, i.e. the reconstruction of volumetric heat source models with respect to some reference data. In this paper several techniques of parameter identification are investigated to reconstruct the 3D transient temperature field for the simulation of laser beam welding. The reconstruction is based on volumetric heat source models and makes use of experimental data. The identification process leads to an inverse problem which cannot be solved exactly but in terms of an optimal alignment of the simulated and experimental data. To solve the inverse problem methods of nonlinear optimization are applied to minimize a problem dependent objective function which can be subjected to constraints.

In particular Response Surface Models (RSM) technique is used to generate a surrogate formulation of the objective function. Sampling points on the RSM are determined by means of Finite-Element-Analysis (FEA). The scope of this research paper is the evaluation and comparison of gradient based and stochastic optimization algorithms. The proposed identification process makes it possible to determine the heat source model parameters in an automated way. Firstly the methodology is applied on an example of homogenous welds and in a subsequent step is extended to dissimilar welds, e.g. aluminum and copper in overlap joint configuration.

359 Environment-assisted cracking of super-elastic TiNi alloy depending on solution pH and electrochemical potential

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The behavior of environment-assisted cracking (EAC) for super-elastic TiNi alloy was investigated as functions of solution pH and electrochemical potential. The specimen was immersed in sulfate solutions at various pHs, subjected to a slow strain rate tensile test and a giving potential was applied to the specimen during the test. As a result, the EAC map for the TiNi alloy as functions of the solution pH and the applied potential was successfully produced. Larger EAC susceptibility was observed in the region of lower potential and lower pH, which indicated the feature of hydrogen embrittlement. The feature was not similar to that of TiAl. The EAC susceptibility was classified by cathodic charge density irrespective to pH nor potential: The charge density of and larger than 0.025 MC m^{-2} induced maximum and the same EAC susceptibility.

360 New trends of materials synthesis and science under ultra-high pressures using diamond anvil cell

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Materials synthesis and science at high pressures over $\sim \text{GPa}$ is one of the future areas of materials researches. It is very attractive from the viewpoint of not only materials science but also crystal chemistry, solid state physics and geoscience. Diamond anvil cell (DAC) provides us an easy means to achieve high-temperature and high-pressure conditions over several GPa pressure range. It can be combined easily with an infra-laser heating system to get a higher temperature for a sample. Therefore, many researchers have recently applied the combination of DAC and laser heating (LASER-DAC) to synthesize novel compounds and crystals at high temperatures and high pressures. The LASER-DAC also easily supplies a supercritical fluid at high temperatures and high pressures which can act as one of reactive species for chemical reaction. This is an advantageous point of the LASER-DAC compared to the large press apparatus in which it is very difficult to compress gaseous species. In this talk our recent studies on ultra-high pressure materials synthesis and science are presented. Several topics are presented and discussed from the viewpoints of materials syntheses using supercritical fluid in high pressures in the ranges more than $\sim \text{GPa}$, such as growth of rutile-type rectangular hollow

crystals and nano-carbon crystals. Besides, some novel metal nitrides are also presented, which are synthesized in nitrogen supercritical fluid. It is also presented that novel xenon compound single crystals are grown in high pressures.

361 The analysis of inhomogeneous deformation behavior in dual-phase steel using by the crystal plasticity fast fourier transform method

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In general, the deformation behavior of crystalline materials is inhomogeneous. The mixed constitution materials such as dual-phase steel were developed in order to enhance their mechanical property. These materials deform more inhomogeneously because displacements across the phase boundary must be continuous. It is necessary to clarify the local stress-strain relations for understanding such a inhomogeneous deformation behavior. But, it is difficult experimentally. Thus, in this study, the inhomogeneous deformation in a dual - phase steel has been simulated by crystal plasticity fast Fourier transform method (CP-FFT). The construction of the model for the calculation was based on actual 3D-structure that was investigated from serial sectioning and electron back scattered diffraction pattern method. The microstructure of the model composes 90% ferrite and 10% martensite. On the basis of the results, the influence of the characteristic of deformation inhomogeneity on the mechanical property of dual-phase steel is discussed. The result indicated the formation of characteristic bands whose strains much higher than the average values. The bands were inclined at 45-degrees to the tensile axis. And it was found that the deformation developed from the ferrite areas adjacent to the interface between ferrite and martensite.

362 Microstructural characterization of ultra-high strength steel welds by means of light optical microscopy and electron backscatter diffraction

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Matching the strength of modern high strength low alloy steels is a crucial task for the welding consumables industry. The alloying concept of the consumable as well as the welding parameters have to be optimized to obtain both a yield strength of at least 1100 MPa and a reasonable toughness. The influence of the microstructural properties on the toughness of ultra-high strength steel welds is the focus of current investigations. A refinement of the microstructure generally leads to an increase in toughness. A combination of light optical microscopy and electron backscatter diffraction (EBSD) allows an extensive characterization of the microstructure of steels. In the present study different etching methods were used to reveal the primary dendritic structure or the secondary austenite grain structure in a martensitic steel weld. These microstructural features were investigated by means of light optical microscopy. As a complementary technique electron backscatter diffraction in a scanning

electron microscope was used to gather information about the crystallographic structure of the martensite and the prior austenite grains. The results obtained by both techniques were evaluated and discussed concerning their suitability for further use.

363 Exploring the thermal, mechanical, and radiation stability of nanocrystalline metals via in-situ transmission electron microscopy

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Nanostructured metals have shown significant promise for various applications due to the exceptional set of mechanical properties that have been associated with them. Although the mechanisms dictating these properties are still significantly debated, it is generally accepted that all of them derive from the high density of internal interfaces or grain boundaries present in the metal. The introduction of these metals into industrial applications has been slowed due to the limited stability of the nanostructure under a variety of real world environments.

In order to characterize the stability of nanograins and grain evolution in nanostructured metals in response to varied thermal, mechanical, and radiation conditions, we have developed a unique tool that permits direct real-time monitoring of the evolution of the nanostructure with complementary mapping of the texture and grain boundary characters. The In-situ Ion Irradiation Transmission Electron Microscope (I³TEM) at Sandia National Laboratories directly couples a TEM to 6 MV Tandem and 10 kV Colutron accelerators, enabling the nanometer resolution observations needed to elucidate the mechanisms governing microstructure stability. The TEM sample can be irradiated with both high energy particles and implanted with low energy ions, either sequentially or concurrently. In addition, both the thermal and mechanical loading can be programmed and monitored, permitting direct correlation between the applied load or temperature and the nanoscale observations. This presentation will highlight recent observations of pure nanocrystalline metals exposed to these complex environments, and compare the evolution of the texture and grain boundary character maps to recent mesoscale models.

364 New materials from extreme conditions processing

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Extreme conditions involving the application of high pressures (p) and high temperatures (T) can induce unexpected chemical compositions, atom arrangements and electronic structures, leading to new classes of materials with unique properties. Although the high significance of extreme conditions is widely recognized throughout materials sciences, its exploitation for synthesis of novel technological materials has not been pursued systematically. Today industrial production activities are restricted to high-value abrasives, like synthetic diamond, c-BN and their composites with ceramics. To advance the field, the projects must embrace a wider range of chemical precursor strategies and synthesis schemes. For example high-p,T reactions using nanopowders as precursors or utilizing volatiles (such as flux and hydrothermal reactions) can offer new prospects for materials exploration and discovery along with kinetic control of

synthesis reactions and phase transitions. Here we report on new refractory boron silicide semiconductors using nanopowders as precursors, the synthesis of exotic hydrogen dominant materials, such as K_2SiH_6 , from gigapascal hydrogenations, and new forms of silica and aluminosilicates from the utilization of extreme hydrothermal environments. Common to these materials is that p-T conditions for their synthesis do not exceed ~10 GPa and ~1200 °C which makes them potentially accessible in industrial processing.

365 Evaluation of weldability of titanium alloy Ti-6Al-4V and aluminum alloy 6061 dissimilar welds produced by electron beam welding

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Aluminum and titanium alloys are among the most important and the most frequently used engineering materials due to their physical and mechanical properties. Especially in the automotive and aerospace industry these materials allow to reduce the weight of design which leads to reducing fuel consumption and environment pollution. These materials are often used together which leads to problems with junction between these materials. In addition to the mechanical joints, there is an effort to produce quality welded joints. Series of works focused to welding of Al/Ti joints by conventional and nonconventional welding methods were published. By reduction of dimensions of molten material is possible to reduce the amount of emerging intermetallic phases and welding defects. Electron beam welding appears as suitable method for welding Al/Ti joints because it allows production of very narrow welds. The benefit is also necessity to perform electron beam welding in vacuum which is required for decrease energy losses of incident beam and simultaneously prevents reaction of molten metal with ambient atmosphere. This paper is focused to determine of appropriate parameters for electron beam welding of dissimilar welds of titanium alloy Ti-6Al-4V and aluminum alloy 6061. Metallographic evaluation, analysis of chemical and phase composition were made on the test welds in order to describe present phases. On the selected welds the influence of intermetallic phases on the mechanical properties was evaluated. The obtained results will be used for further experiments focused to optimize the process of electron beam welding of Al/Ti alloys.

366 Studies of degradation mechanisms of PEFC catalyst layers through an in-situ SEM/STEM technique

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Commercialization of fuel cell vehicles (FCVs) has led to increased general attention toward a hydrogen energy society. Since electrocatalysts for PEFC have an import role to increase FCV performance, the development of new catalysts is desired for the next generation, and then both their activity and durability should be studied fundamentally. In order to understand degradation mechanisms, we introduce accelerating degradation tests using Environmental TEM (Hitachi HF-3300). In this study, degradation derived by carbon oxidation, usually seen for cathode

catalysts, is focused on. Common cathode catalysts, Pt deposited carbon materials, were observed by SEM/STEM under the air introduction as an oxidant. For accelerating carbon oxidation, the specimen holder was heated up to 200 °C. Then, degradation of electrocatalysts was observed in the real-time. Although degradation mechanisms of Pt have been studied for decades, a new phenomenon of Pt embedding into Vulcan carbon particles was observed. In order to further investigate degradation mechanisms, experimental conditions were varied. Even with Nafion ionomer, Pt embedding occurred. When graphitized Vulcan carbon was used, embedding was suppressed, and agglomeration was rather accelerated. In case of hydrogen introduction instead of air, embedding did not occur. Consequently, Pt embedding is most likely derived from carbon oxidation catalyzed by Pt nanoparticles. Pt particles continuously oxidize carbon under them, leading to Pt embedding.

367 Effect of added elements on microstructures and joint strength of lead-free Sn-based solder joint dispersed IMC pillar

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To create a high reliability solder joint for automotive applications, the method to disperse pillar-shaped IMCs in the solder joint with Cu was examined with several Sn-based lead-free solder. In the joint with Sn-0.7Cu-0.05Ni (mass%), growth of pillar shaped $(\text{Cu,Ni})_6\text{Sn}_5$ IMCs which connect Cu plates on both sides was observed when bonding was conducted at 300°C for 30 min. In the joint with Sn-3.0Ag-0.7Cu-5.0In (mass%), coarsen columnar Cu_6Sn_5 IMCs which include a few mol % In grow in relatively random directions in bonding at 300°C for 30 min. The growth rate of IMCs in bonding is the largest among solder investigated in this study. In the joint with Sn-5.0Sb (mass%), thick columnar Cu_6Sn_5 IMCs grow from both Cu sides although there are no IMCs to connect Cu plates on both sides in bonding at 300°C for 30 min. For joints with Sn-3.0Ag-0.7Cu-5.0In and Sn-5.0Sb, an effectively IMC dispersed joint is expected to be fabricated by optimization of bonding conditions. In the joint with Sn-3.0Ag-0.5Cu (mass%), a thick scallop shaped IMC layer forms at the joint interface and thus it is difficult to fabricate pillar shaped IMCs to connect Cu plates on both sides. In the paper, joint strength of each joint by die shear test will be also reported.

368 Material and mechanical aspects of CMAS damage progression on thermal barrier coatings and its non-destructive detection

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At high temperatures exceeding 1200°C thermal barrier coatings (TBCs) on turbine blades in gas turbine engines can be damaged by calcium-magnesium-alumino-silicates (CMAS) resulting from the ingestion of siliceous minerals with the intake air and from unclean fuels. In this study, the CMAS damage progression was investigated at lab scale by employing a synthetic CMAS product spread on Air Plasma sprayed TBC specimens, followed by

isothermal exposure and thermal cycling at different temperatures up to 1300°C. CMAS infiltration into the top coat occurred at temperatures above 1210°C. With increasing exposure time reaction between the CMAS and the top coat occurred, resulting in various microstructural changes. Furthermore, delamination of the top coat was observed on the CMAS infiltrated specimens. The elastic modulus of the CMAS infiltrated top coat, which was measured through a vibrating reed machine, was about four times that of the non-infiltrated ones. The effect of CMAS interaction with the top coat on the delamination behavior was then assessed based on thermal stress analysis. Furthermore, measurements of electric capacitance by the AC impedance method for TBC specimens were also carried out, in order to explore the non-destructive detection of CMAS damage in the present study. The capacitance showed higher values at the CMAS infiltrated area, compared with that at the non-infiltrated ones. These suggest that the CMAS infiltrated area and its infiltrated depth would be detected by measuring the capacitance. The background of the change in capacitance was also discussed using simple condenser models.

369 Comparison of Self-annealing Behaviors in (001)- and (111)-Oriented Electrodeposited Silver Films by In Situ EBSP Analysis

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Self-annealing behavior of an electrodeposited Ag film which has been preferentially oriented in (001) direction was investigated by in situ electron backscatter diffraction pattern (EBSP) analysis. There are few clear grains of micrometer order in the initial state just after electrodepositing. The result shows that the electrodeposited film would be in amorphous state or could be consisted of very fine grains which size cannot be analyzed by EBSP. Self-annealing starts in storage at R.T. for a few hours and recrystallization almost completes in the storage time of 6 h. Recrystallized grains with relatively random orientation and a few micrometer order size have nucleate in storage time of a few hours. Afterward, (001) oriented grains preferentially grow in the self-annealing process. During self-annealing, the area fraction of (001) oriented grains increases rapidly and saturates in the range from 70 % to 80 % at the storage time over 6 h. The area fraction of (111) oriented grains slightly increases with increasing storage time, but it becomes at most 10 % even when self-annealing process has completed. On the contrary, the area fraction of (101) oriented grains decreases with increasing storage time and it becomes almost 0 % at the storage time over 6 h. In the paper, comparison of self-annealing behaviors in (001) oriented and (111) oriented electrodeposited silver films will be also reported.

370 Novel steels with niobium microalloying

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Production of microalloyed flat steel products around the world has increased many folds in tandem with end-user requirements for optimised balance mechanical and functional properties. These microalloyed steels demonstrate that effective combination of thermo-mechanical processing and phase transformations is a powerful way to develop and control microstructures,

generally unattainable by either route separately. While carbon, manganese, silicon, and aluminium make up the base alloying concept small amounts of niobium, titanium, vanadium, chrome and molybdenum are added to either improve the steel's property profile or to widen the processing window. The amount of niobium necessary to achieve these effects and thus the involved alloying cost is also quite small. It is well established that niobium is not only the most efficient alloying element in providing grain refinement but also it also supports transformation hardening and precipitation strengthening under suitable processing conditions. The optimum use of these niobium-related effects requires a careful design of the alloy as well as the consideration of the entire processing route in the steel mill. This presentation will highlight some of the recent results on the development of novel niobium-microalloyed structural, automotive and linepipe steels.

371 Microstructure and mechanical properties of friction stir welded 40 mm thick Al-Zn-Mg (A7N01-T5) alloy plate

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With the rapid development of high-speed railway, aluminum alloy is now widely used in high-speed train manufacturing. Welding technology of aluminum alloy directly affects the body structure and manufacturing cost. As known, there are many problems in the aluminum alloy fusion welding, such as porosity, inclusion, hot cracking and joint softening, etc. These problems become prominent when welding thick plate and limit the application of aluminum alloy thick plate in making key components of high speed train, such as corbel and coupling board, by conventional fusion welding method. Friction stir welding (FSW), a solid state joining method, is considered as the most effective one to overcome those problems occurred during fusion welding process of aluminum alloy, especially for aluminum alloy thick plate. In this paper, 40 mm thick A7N01-T5 aluminum alloy plate was friction stir welded using a double side process. The microstructure evolution of different zones of the joint, which is significantly affected by the strains and thermal cycle subjected during FSW, were investigated using various characterization methods. Mechanical properties of the joint were evaluated through tensile tests and micro-hardness measurements. It was shown that the tensile strength of the joint reached about 88% of the base material, and all the tensile specimens were fractured at heat affected zone, which was confirmed as softening region by microhardness measurement. This phenomenon was discussed and attributed to the re-solution and overaging of strengthening precipitates during the welding thermal cycle.

372 Effects of Ag addition on the microstructures and properties of Al-Mg-Si-Cu alloy

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Effects of Ag addition on the microstructures, ageing characteristics, tensile properties, electrochemical properties and intergranular corrosion properties of Al-1.1Mg-0.8Si-0.7Cu-

0.5Mn-0.02Ti alloy were investigated using optical microscopy, scanning electronic microscopy (SEM) and Energy Dispersive Spectroscopy. With increasing Ag addition (>0.4wt.% Ag), both the strength and elongation of alloys decrease, the strength and elongation significantly decrease 70MPa and 4%, respectively. The best aging temperature of alloys is 180°C. When ageing at 140°C, the hardness increases gradually and no peak hardness is observed, the hardness is low. When ageing at 220°C, the time to peak hardness shortened, and the peak hardness is low. Adding Ag into base alloy, the spontaneous corrosion potential firstly decreases and then increases and reaches a maximum value when Ag content is 0.4wt.%, and decreases again when Ag content is 0.7wt.%. The corrosion current density is lower than that of base alloy in the range of 0.1%~0.7%, and is smallest when Ag content is 0.4wt.%. The intergranular corrosion (IGC) test shows that IGC susceptibility of 0.4wt.% Ag alloy is lowest; 0.4Ag alloy has the best intergranular corrosion resistance under aged state, and corrosion resistance deteriorates under peak aging and over-aging states.

373 Driven mixing and nanostructure formation of metallic multilayers with repeated cold rolling and folding

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The repeated rolling and folding of arrays of metallic foils has proven to be very effective as a bottom-up approach for processing of bulk nanolaminate materials. While initially pursued as a technique to synthesize bulk amorphous alloys, much emphasis has been placed in the last 15 years to understand the formation of nanoscale microstructures during the roll and fold process. For many metal combinations, it is possible to achieve bulk samples with individual layer thicknesses well below 100 nm. Based on the model systems Cu-Mo and Cu-Ni, the complex interrelations will be highlighted between solution thermodynamics, crystallographic, and mechanical properties. The two main questions that will be addressed are the mixing phenomena at the layer interfaces and the morphology evolution of the layer array, which can unfold as layer codeformation or a shear-band driven formation of particle-matrix microstructures. Deformation-induced mixing occurs regardless of the values of the thermodynamic solution properties such as the heat of mixing. But the experiments performed to date suggest that the heat of mixing has an impact on the “kinetics” of the mixing process. The localized interfacial texture evolution also likely contributes to the mixing process and the formation of shear bands. The shear band formation and the overall layer morphology evolution are also affected by flow stress differences between the individual layers. These complex and interrelated aspects of the layer mixing and morphology evolution will be highlighted with the Cu-Mo and Cu-Ni model systems along with possible structural and energy applications.

374 High ductility AHSS grades: Improved formability by advanced microstructure control

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In recent years, the usage of 1st generation AHSS grades (Dual-Phase (DP), Complex-Phase (CP), and TRIP steels) has been increased dramatically by the automotive industry. Especially, DP steels up to a minimum tensile strength of 980MPa are applied frequently, nowadays. However, as both main driving forces for light weight construction in the car industry - improvement of fuel economy and increase of passenger safety - are still valid, there is the need to further decrease the weight of cars. One option is hot forming, which is now well introduced, and plays an important part in the automotive light weight construction. However, the heat treatment of the hot forming steel in the press shop also increases the complexity of production. Another possibility to save weight would be, to increase the formability of AHSS significantly, so that more complex parts can be formed out of these grades, which is especially important for 980 and 1180 grades. That would be a very cost-efficient way for a further weight reduction of the body in white. Exactly this was the motivation for the development of the High Ductility grades by voestalpine. In this work, it will be demonstrated, that by optimising the microstructural design of AHSS and by keeping the evolution of the microstructure within tight limits, a significant improvement of the formability can be achieved. High Ductility AHSS are a group of steel grades with strength levels of 590, 780, 980, and 1180MPa. For each strength level, a drawing type (DP-type) and a bending type (CP-type) can be developed.

375 Creep rupture, oxidation and corrosion of a Z-phase stabilized steel tested with welded tubes

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Fossil fired steam power plants are operated to supply the majority of the world's electricity demand. To minimize the CO₂ emissions of these plants, the life and reheat steam temperature and pressure need to be increased as much as possible. This requires - among others - boiler tube materials for final superheater and reheater components with sufficient creep strength as well as good oxidation and corrosion behavior for highest metal temperatures of up to 650°C. State-of-the-art material choices are austenitic boiler tubes, as 9-12% Cr steels do not meet the required properties for long-term operation. A cost efficient alternative for superheater material could be a newly developed 12% Cr steel, which utilizes a fine distributed Z-phase as a thermodynamically stable strengthening phase in the steel. Within the framework of the European FP7 project "Z-Ultra" RWE has carried out two different types of component tests to investigate the behavior of this Z-phase stabilized steel. Pressurized creep rupture tests are carried out at temperatures of 625 and 650°C on welded tube samples with a calculated lifetime of 10,000 hours to examine the creep behavior of the weld connection and its failure behavior. A pressurized combined oxidation and corrosion test is conducted to investigate the outside corrosion and inside oxidation behavior over 8,000 hours. The test tube with a simulated weldment is in contact with boiler ashes and humid flue gas at the tube outside and water vapor at the inside at realistic temperatures between 600 and 650°C at a pressure of 24 bars.

376 Influence of high-pressure torsion on the microstructure and the hardness of a Ti-rich high entropy alloy

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High Entropy Alloys (HEAs) are in the focus of materials science due to their high strength, good ductility and excellent resistance to wear, corrosion and softening at high temperatures. HEAs are disordered solid solutions, containing at least five chemical elements with similar concentrations. The high strength of HEA materials is caused by the strong resistance of the disordered crystal lattice to dislocation motion. Therefore, a very yield strength (about 900 MPa) can be detected even for HEAs with very large grain sizes (about 1 mm). Additional grain refinement by severe plastic deformation (SPD) may increase this strength to a very high level. High-Pressure Torsion is one of the most effective SPD techniques in grain refinement. The goal of our study was to investigate the influence of HPT on the microstructure and hardness of a Ti-rich coarse-grained HEA material. The evolution of the grain size and the dislocation density due to 1 turn of HPT was studied by ACOM-TEM and X-ray line profile analysis. Besides the refinement of the microstructure a phase transition also occurred during HPT. The initial bcc structure transformed into hcp structure throughout the material. The features of this phase transformation were studied on samples compressed to low strain values. The hardness as a function of the distance from the center in the HPT-processed disk was measured and correlated to the microstructure.

377 Magnetic shape memory effect in Ni-Mn-Ga single crystal

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The magnetic shape memory effect is as an example of the multiferroic effect combining ferro-elasticity and (ferro) magnetism. It is similar to conventional shape memory effect but the shape of the material is manipulated by magnetic field. Here we will focus on magnetic field induced structure reorientation (MIR). The reorientation occurs due to motion of twin boundaries. In the Ni-Mn-Ga martensite it results in giant deformations of up to 12% in moderate magnetic fields. As the magnetic field is relatively weak force compared with mechanical stress, the high mobility of twin boundaries is the basic condition for the effect. The reason for extreme mobility is not known but it may be connected with the shear instability of the lattice. The effect occurs only in particular modulated martensite structure of Ni-Mn-Ga alloy. We will discuss the properties of martensite relevant for this effect. In 10M modulated martensite, two types of mobile twin boundary (type I and type II) are observed with complex layered microstructures consisting of a hierarchy of twinning systems. The boundaries strongly differ in the magnitude and temperature dependence of the twinning stress. We show that heat treatment can affect the shear stability of the alloy and thus it may affect the mobility of the twin boundaries. We will search for analogue with non-magnetic Cu-Ni-Al shape memory alloy and follow some in-situ TEM experiments which may enlighten the character of modulated martensite phase.

378 Modelling grain coarsening in the framework of rational extended thermodynamics

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During heat treatment of metallic materials, growth and shrinkage of individual grains is a well discussed thermomechanical process. Often, grain growth and shrinkage is described without using a thermodynamic approach, like in the classical model by Hillert (1965). In this lecture, we show how a thermodynamically consistent model for grain coarsening in metallic materials can be derived in the framework of rational extended thermodynamics. The present approach links the microstructure description to continuum mechanics by a unified thermodynamic framework. Therefore, a free energy function incorporating the energy storage due to grain boundaries is introduced. The required evolution equations for representing the growth and shrinkage of the individual grains are motivated from the entropy principle. Of course, the discussed thermodynamic framework can also be used to develop more elaborate material models. Despite the rather different approaches used, we obtain a simple but powerful grain growth model which is similar to existing ones and can be regarded as a thermodynamic extension of that by Hillert to more general systems. Therefore, the relation of the developed model to existing ones will be discussed. Only physically motivated material parameters have been included in the suggested model. To demonstrate its applicability, we compare our simulation results to grain growth experiments in pure copper by different authors. The experimental observations can be predicted very accurately. In addition, we study the implications of the energy release due to grain growth on the energy balance and further interesting phenomena.

379 Development of three-dimensional porous titanium web for bone defect filling

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Titanium is widely used for dental implants and orthopedics because of its excellent biocompatibility and sufficient mechanical properties. A disadvantage of titanium is its higher elastic modulus than bone tissues, resulting in bone atrophy. A solution in reducing the elastic modulus is introducing pores. However, conventional porous titanium using titanium powders indicates disadvantages of low toughness and low deformability. Therefore, the authors have developed porous titanium mesh blocks using three-dimensionally woven titanium fiber to address the two disadvantages. The aim of this study is to evaluate new bone-formation of titanium mesh block with three different porosities. A mesh tube was woven using titanium fiber of 20 μm diameter. It was set into a metal mold with 3 mm inner diameter, and then pressed to the height of 3 mm. In the present study, the porosities of mesh block were 88%, 69% and 50%. New bone-formation was evaluated using rabbits. Three bone defects of 3 mm depth and 3 mm diameter were drilled in each tibia, and were randomly designated to three groups of porosities; A: 88%, B: 69%, and C: 50%, and bone-formation 4 or 12 weeks after implantation was evaluated using micro-CT. The micro-CT analysis indicated that bone-formation with 50% porosity at 4 weeks was significantly higher than that of the other two

porosities, and the new bone-formation was significantly higher for 69% porosity at 12 weeks. New bone-formation in the mesh block is possibly optimized by optimizing porosity.

380 Graphite-alumina and carbon nanotube-alumina sol-gel composite coatings on 304-L stainless steel for tribological applications

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The risk of seizure in moving parts as well as losses due to friction and wear both have a huge economic impact. Moreover, the continuous development in the mechanical industry and regulation changes require a constant improvement of lubrication solutions. Self-lubricating materials, preventing the need for liquid lubricants, are of particular interest. This study aims at investigating carbon-alumina sol-gel composite coatings on 304-L stainless steel. Here, carbon will be in the form of either graphite platelets or multi-walled carbon nanotubes (MWCNTs). The required amounts of carbon are incorporated into boehmite sols. Coatings are prepared by dip-coating. After gelation, drying and the appropriate thermal treatments, carbon-alumina coatings 1-10 μm thick are obtained. Their composition, structure and microstructure are investigated using a variety of techniques including electron probe microanalysis, X-ray diffraction and electron microscopy. Ball-on-disc wear tests with 316L stainless steel ball have been carried out at room temperature (2 N normal force, 10 m.s^{-1} sliding speed, 250 m sliding distance and 40% relative humidity) in order to investigate the friction coefficient and wear of the pure alumina, graphite-alumina and carbon nanotube-alumina coated systems. The friction coefficient for the composite coatings is significantly (about 4 times) lower than for the uncoated steel substrate, where it is higher for the alumina coating. The corresponding wear volumes are significantly lower too. The influence of the nature of carbon (graphite or MWCNTs) is discussed in details, notably with respect to the formation of a carbonaceous lubricating film during the wear process.

381 Measurement of grain boundary chemistry and crystallography by atom probe tomography and correlated electron microscopy

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The amount of solutes that segregate to an interface is governed by the crystal defect's five macroscopic crystallographic parameters. A full picture characterization of grain boundary segregation requires accurate detection of subtle compositional changes within the first few nanometers around the interface, the measurement of grain orientations and planes and all of this ideally for a high number of interfaces. Atom probe tomography is an ideal tool to quantify interface segregation. However, its spatial resolution is material dependent and only in special

cases is high enough to capture crystallographic information. This talk covers the state of the art techniques to quantify grain boundary segregation by the correlated use of atom probe tomography and electron microscopy, viz. electron backscatter diffraction, transmission Kikuchi diffraction and nanobeam diffraction. Results are discussed for the bcc Fe-C system and a fcc Fe-Mn-C alloy.

382 Dendrite growth kinetics in undercooled melts of Zr-based alloys

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Solidification needs an undercooling to drive the solidification front. If large undercoolings are achieved metastable solid materials are solidified from the undercooled melt. Containerless processing provides the conditions to achieve large undercoolings since heterogeneous nucleation on container walls is completely avoided. In the present contribution both electromagnetic and electrostatic levitation is applied. The velocity of rapidly advancing dendrites is measured as a function of undercooling by a High-Speed-Camera. The crystal growth dynamics is investigated in undercooled melts of Cu-Zr and Ni-Zr glass forming alloys. Even though not involved in glass formation growth dynamics of any crystalline phase is essential to determine the glass forming ability of the respective materials. A maximum in the growth velocity – undercooling relation is observed in those alloys of good glass forming ability. This is understood by the fact that the temperature dependent diffusion coefficient counteracts the thermodynamic driving force for rapid growth if the temperature of the undercooled melt is approaching the temperature regime above the glass transition temperature.

383 Precipitation hardening against slip and twinning in magnesium alloys

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Magnesium alloys are promising structural materials due to their weight-saving possibilities. However, magnesium wrought products are typically characterised by distinct yield stress asymmetry, restricting many of their potential applications. This is attributed to the polarity of twinning and the relative values of the critical resolved shear stresses (CRSSs) for the various deformation mechanisms, mainly affected by solutes and particles. In this work, the hardening effect of precipitates against slip and twinning is investigated. For this purpose, an extruded bar of the MN11 alloy (Mg-1 wt.% Mn-1 wt.% Nd) and a rolled sheet of the AZ80 alloy (Mg-8 wt.% Al-0.5 wt.% Zn) were thermally treated in order to obtain different amounts and sizes of precipitates, these being prismatic and basal plates, respectively. Afterwards, the particle-containing materials were tested under tension and compression at room temperature. A quantitative analysis of precipitation in both alloys, as well as examinations on the interaction between particles and dislocations or twins were performed by means of EBSD and TEM. Adequate expressions of the Orowan equation were developed for the predominant deformation mechanisms in Mg. However, the comparison between the predictions of such expressions and the experimental data revealed that Orowan relationship alone cannot explain precipitation

hardening of mechanical twinning, where other effects such as that of plastic strain discontinuity at the particle-matrix interface or back-stress must be also considered. In addition, the so-called reversed yield asymmetry, promoted in certain Mg alloys, including MN11, by precipitation is related to the predicted relative CRSSs.

384 Modelling of microstructure evolution with dynamic recrystallization in 3D phase-field environment of martensitic steel

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In order to predict material properties after hot forming of steel, it is necessary to understand the microstructural processes during forming. A new simulation environment is developed to simulate the evolution of microstructure and the corresponding flow stress during rolling and softening. A crystal plasticity hardening model is coupled to grain evolution-, recovery- and recrystallization kinetics within the three dimensional phase field framework OpenPhase. The crystal plasticity module takes grain orientation into account which enables further texture analysis. Grain structure evolution kinetics are extended to consider dislocation distribution. Crystal plasticity, recovery and recrystallization kinetics are treated separately to differentiate between individual hardening and softening effects. The simulation is applied to an initial hot forming process of high-strength martensitic steel MS-W 1200. Hot compression tests at 900-1000 °C with a strain rate of 0.1 s⁻¹ were performed to identify input parameters and validation purposes.

385 Plastic deformation of single crystals of iridium

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Iridium, a member of the platinum group metals with the FCC structure, possesses high hardness, melting point and corrosion-resistance. Although usual FCC metals such as aluminium, copper, and so forth, exhibit ductility and malleability, polycrystalline iridium fails by brittle intergranular fracture exhibiting considerably low ductility over a wide range of temperature. Although the mechanical properties of polycrystalline and single-crystal iridium have been investigated by various researchers, there remains an open question as to why iridium exhibits such a low ductility. Moreover, it has been reported that single crystals of iridium show a significant anisotropy in CRSS in tension, which cannot be expected for usual FCC metals. In the present study, compression deformation behavior of single crystals of iridium was investigated as a function of loading axis orientations in order to understand its plastic deformation mechanisms. Observations on the deformation microstructures by optical and transmission electron microscopy indicated that {111}<110> slip operates in iridium similarly to other usual FCC metals as well as that, unlike other usual FCC metals, iridium shows frequent cross slip from initial stage of deformation and an extremely large extent of work hardening.

386 Surface structuring by pulsed laser implantation

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Micrometric surface topologies are required for a wide range of technical applications. Structures with lowered surface features are already utilized for bearings or cylinders of combustion engines. There are also other fields of application, where the potential of elevated surface features is theoretically known, e. g. for metal forming tools. However, the demand for a high wear resistance of these structures often inhibit an industrial realization. Techniques for surface structuring basing on a relocation of material are not able to improve the wear resistance significantly, since the wear resistance is limited by the intrinsic material properties of the substrate. Therefore, wear resisted coatings must typically be applied.

Another solution is offered by techniques which apply additional material to a surface. A promising approach is the localized dispersing of hard ceramic particles by pulsed laser radiation, the so-called laser implantation. In this paper, the potential to adjust the geometry as well as the mechanical properties of laser implanted surfaces is described by means of microstructural and topographical investigations. Afterwards, different applications for this novel structuring technique are discussed and first results of wear tests are given. It can be shown that structures with a highly improved durability can be produced.

387 Influence of carbon addition on mechanical properties of thixomolded magnesium alloy

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Magnesium alloy has been applied as an alternative for plastic moldings in body frames for electronic equipments by means of excellent properties. Most of these are produced by the thixomolding process, and AZ91D magnesium alloy chips are ordinarily used. We have reported that mechanical property and fluidity were improved by using the AZ91D magnesium alloy chip fixed the carbon nanoparticle. On the thixomolding process, the blow hole may occur based on the solidification contraction, when wall thickness of the part increases. Therefore, the mechanical property of these parts may lower. It should be necessary to clarify the mechanical property in order to apply for structural members such as the automobile. In this study, the effect of carbon modification to the AZ91D magnesium alloy chip surface on fatigue property was examined by the rotary bending test. The carbon modification to the chip surface was quite useful for the control of the internal defect for the thick part on the thixomolding process. The depression of the internal defect by the carbon nanoparticle modification is closely related to the improvement of flowability, and that is presumed to be based on promoting the filling of the molten metal into the metal die inside. The tensile strength and elongation were respectively improved by the carbon addition. In addition, the fatigue strength also improved over 15%. It might be concluded that the magnesium-carbon alloy produced by the thixomolding process cloud be applied for a structural material such as automobile.

388 Nanoplatfom based on vertical nanographene for green technology applications

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Carbon nanowalls (CNWs) are composed of few-layer graphenes standing almost vertically on the substrate forming 3-dimensional structure. CNW film with large surface area has many graphene edges and CNW sheet itself is composed of nanographite domains of a few tens of nanometers in size. These nanographenes are modified with several types of surface termination including nanoparticles and biomolecules. Therefore, the structure of CNWs can be suitable for the platform of electrochemical and biosensing applications.

It is known that hydrogen peroxide (H_2O_2) is an important biological substance in the fields of chemistry and biology. For example, H_2O_2 is produced as by-product in the oxidation of glutamate in the presence of L-glutamate oxidase enzyme. Since the H_2O_2 is electroactive at electrodes such as platinum (Pt), glutamate sensing can be realized by detecting H_2O_2 . In this work, CNWs were grown on carbon fiber paper (CFP) using plasma-enhanced chemical vapor deposition with CH_4/Ar mixture. Subsequently, CNW surface was decorated with Pt nanoparticles by the reduction of H_2PtCl_6 in solution or by the metal-organic chemical fluid deposition employing supercritical fluid. Electrochemical property of Pt-decorated CNWs was evaluated by cyclic voltammetry. Pt-decorated CNW/CFP was successfully used as an electrode for H_2O_2 sensing. Moreover, Pt-decorated CNW/CFPs were also successfully used as electrodes for fuel cell. Electrochemical experiments indicate that nanoplatfom based on vertical nanographene with large surface area and high electrocatalytic activity offers great promise for providing a new class of nanostructured electrodes for electrochemical sensing and energy applications.

389 Fatigue and fracture of ultrafine-grained and nanocrystalline materials

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Tensile, fatigue and fracture mechanics properties of nanocrystalline materials are rare, because the available material volume is usually very small. Severe plastic deformation (SPD) is a method to produce ultrafine grained or nanocrystalline materials in large quantities. This enables the determination of the fracture, fatigue and ductility properties of these types of materials. High pressure torsion allows for the SPD of most metallic materials. Hence, and it allows the transformation of typically microcrystalline materials into an ultrafine or nanocrystalline state.

The large HPT equipment at our institute permits now to determine standard tension and fracture mechanics parameters on a large variety of metals and alloys. This contribution will give an overview of new results obtained in pure metals, alloys, and nanocomposites. Special attention will be devoted to the developed anisotropy of the fracture toughness and fatigue propagation, which is extremely pronounced in some materials.

390 Effects of warm working on microstructural and shear deformation properties of TRIP-aided martensitic steel

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The effects of warm working temperature on microstructural and shear deformation properties of a 0.2C–1.5Si–1.5Mn TRIP-aided martensitic steel for applications to automotive frame parts and forging parts. When post cooling rate is 5 °C/s after working, warm working at 550–700 °C after austenitizing refined the microstructure and increased the volume fractions of retained austenite and martensite-austenite like phase in the steel. The size of microstructure and these volume fractions decreased and increased with decreasing working temperature, respectively. The increased volume fractions were caused by that recrystallized austenite phase was refined by working at lower temperature. The shear displacement and the length of shear section increased with decreasing working temperature, accompanied with a decrease in maximum shear stress. This was associated with the suppressive void formation due to the strain-induced transformation of retained austenite, refined microstructure and a small difference in flow stress between martensite-austenite like phase and matrix structure.

391 Separating technology of pure zirconia from zircon-sand by the Ar-H₂ arc plasma fusion and the microwave leaching

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Zirconia (ZrO₂) is excellent for high toughness, high strength, thermal stability and high corrosion resistance. Thus, recent zirconia has been spotlighted as a dental material. Most of zirconia, because it is produced in zircon sand (ZrSiO₄), refining process (wet, dry), which can be separated only of zirconia have been developed. The plasma fusion method has the advantages that processes can get easily and quickly sinter-ability good fine powder as compared with traditional wet method. Thus in this study, zircon-sand is separated into zirconia and silica by using the Ar-H₂(hydrogen) arc plasma refining. And then silica is removed by the microwave leaching method to produce a high quality zirconia. Argon gas, hydrogen gas, copper anode and tungsten cathode are used for the plasma arc generation. To facilitate zirconia and silica separation, carbon of 1~3 molar ratios are added with zircon-sand. Plasma melting were sequentially conducted two processes. After a reduction process using an Ar gas only, it was refined using a mixed gas of Ar-H₂. After melting and water cooling in chamber, the solid phases composed with zirconia and silica obtained at 240°C, and 10% sulfuric acid solution used as an leaching materials to obtain a recovery ratio of 60% and high purity zirconia (more than 95%).

392 Characterization of the acoustic emission response and mechanical properties of Mg alloy with LPSO phase

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Mechanical properties of extruded WZ72 magnesium alloy with long-period stacking ordered (LPSO) phase were investigated during compression loading at room temperature and at a constant strain rate of 10^{-3} s^{-1} . The specimens had 3 different orientations of their LPSO fibers with respect the loading axis – longitudinal, 45° and transversal. Concurrently, with the deformation tests, the acoustic emission (AE) response of the specimens was recorded. The statistical analysis of the AE data revealed that the both twinning activity and the kinking of the LPSO phase significantly depend on the orientation of LPSO fibers. The investigation of the microstructure proved the results of the AE experiments.

393 Effect of Fe addition on mechanical properties of Ti-Au near-eutectoid alloys

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Ti-Au near-eutectoid alloy is a hopeful biomaterial since the additional element Au can be expected to enhance X-ray radiography and biocompatibility. Besides, Co-added Ti-Au near-eutectoid alloy exhibits good shape memory effect. In this paper, phase constitution, microstructure and mechanical properties of Ti-4at%Au (Ti-4Au) containing 0 to 7at%Fe alloy were investigated. It was found that the apparent phase at room temperature shifts from α' (hcp) to β (bcc) by 3at%Fe in addition to Al₁₅ Ti₃Au. The size of Ti₃Au was a few micrometer size, and the amount of Ti₃Au increased with increasing Fe content. Then, Fe addition promotes the formation of Ti₃Au. The Vickers hardness value increased with increasing Fe content: HV293 in Ti-4Au-3Fe and HV384 in Ti-4Au-7Fe. By tensile tests at room temperature, Ti-4Au-4Fe exhibited large elongation around 27% in addition to 800MPa in UTS. However, the elongation largely decreased and the strength increased with further increasing Fe content. Moreover, Ti-4Au-3Fe and Ti-4Au-4Fe possessed some shape memory effect. This work was supported by Grant-in-Aid for Scientific Research Kiban S 26220907 from Japan Society for the Promotion of Science (JSPS).

394 Effects of grain size and particle dispersion on the work hardening behavior of austenitic stainless steel

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Austenitic Stainless Steels (A-SS) show significant work hardening during plastic deformation. It is considered that, without the stress induced martensitic transformation, the dislocation accumulation dominates the work hardening behavior in case of A-SS. In that case, grain

boundaries and second phase particles which act as obstacles to dislocation motion are expected to play important roles in work hardening behavior. Clarifying the effects of both obstacles on the work hardening behavior can be the key to control and enhance the performance of A-SS. However, studies on work hardening behavior of stable A-SS are limited and the interactions between dislocation and the obstacles have not been well-understood. In this study, two 100kgf VIM ingots were prepared, of which chemical compositions, Fe-12Cr-18Ni-V-C, were tailored so that they do not transform martensite by heat treatment or cold-working. In addition, their chemical compositions were set to have the same solutal element concentration after solution treatment for one steel and after precipitation heat treatment for the other. Vanadium carbide (VC) was employed as precipitate. Grain size was changed with solution temperature. With these preparations, it is expected that effects of grain size and secondary particle on work hardening behavior will be separately discussed. Test specimens were served for hardness measurement, uniaxial tensile test, electron backscatter diffraction (EBSD), X-Ray diffraction, and TEM. In the presentation, we will report the experimental results and also discuss the details of work hardening behavior during the plastic deformation in terms of interactions among dislocation, grain boundaries and VC precipitates.

395 Gate stack technology for silicon carbide based metal-oxide-semiconductor devices

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Silicon carbide (SiC) is one of the promising wide bandgap semiconductor materials for high-voltage and high-temperature power electronic devices because of its excellent physical properties. Although SiO₂ gate insulator can be grown on SiC by conventional thermal oxidation similar to Si, low channel mobility and poor gate oxide reliability are the critical issues for SiC power metal-oxide-semiconductor field-effect transistors (MOSFETs). The post-oxidation annealing (POA) in NO or N₂O ambient is generally performed to improve the interface properties. The reduction of interface states would be attributed to the nitrogen atoms incorporated in SiC side at the SiO₂/SiC interfaces. However, the channel mobility of SiC MOSFET is still lower than expected from the bulk mobility. It has also been pointed out that the hole trapping during bias stressing is enhanced regardless of the reduction in electron traps, which leading to threshold voltage instability hence a serious concern in device reliability. Therefore, further development of SiC-MOS gate stack technology is highly desired. In this study, we systematically evaluated an impact of SiO₂/SiC interface nitridation on charge trapping characteristics in SiC MOS devices, and proposed a novel but simple method to improve interface property by using high-temperature processing.

396 Phonon excitations in Pd_{42.5}Ni_{7.5}Cu₃₀P₂₀ bulk metallic glass by inelastic x-ray scattering

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Pd_{42.5}Ni_{7.5}Cu₃₀P₂₀ is well-known to form the most excellent bulk metallic glass with the critical cooling rate of 0.067 K/s [1]. To investigate the atomic dynamics of this BMG, we have carried out inelastic x-ray scattering (IXS) experiment using a high-energy-resolution IXS spectrometer installed at the beamline BL35XU of the SPring-8. An IXS experiment was already performed by Ichitsubo et al. [2], where only the longitudinal acoustic (LA) excitations were discussed. In our new IXS results, the largest excitations are the LA modes, which show the clear dispersion relation with Q, and the position of the excitation energies coincides with the previous data [2]. There are other excitations burying the hollows between the quasielastic peaks and the LA excitations, which would be the transverse acoustic (TA) modes showing a slight dispersion relation with Q. Besides the LA and TA modes, an unknown optic-like mode can be realized as a long tail at energies higher than that of the LA mode. This mode looks unchanged in excitation energy with Q, and hindered beneath the strong LA mode. The spectral feature resembles the so-called Boson peak. Detailed analysis of the measured IXS spectra is now in progress. [1] N. Nishiyama and A. Inoue, Mater. Trans., JIM 37, 1531 (1996). [2] T. Ichitubo et al., Phys. Rev. B 76, 140201(R) (2007).

397 In-situ tensile texture analysis on a new Mg-RE alloy

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A new Mg-RE alloy was previously developed by micro-alloying method (RE < 0.4 wt.%), which achieves a high ductility and good corrosion resistance. In-situ tensile test via neutron and synchrotron diffraction were performed to investigate first the deformation behavior; and second the texture evolution which can be related to the deformation mechanism, and finally to understand why the as-cast Mg-RE alloys show such a high tensile ductility. Preliminary results showed that a dominated basal fiber texture was gradually developed with the increase of tensile strain. However, before the sample was broken a (10.0) fibre texture showed a similar intensity to that in (00.2), which means more activations of the non-basal slip planes during tensile deformation. This could also contribute to a relatively high elongation of this new Mg-RE alloy at room temperature. Further explanations will be showed together with the microstructures.

398 Two-step annealing for grain refinement in twin-roll cast Al-Mn alloys

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A two-step annealing process has been developed to obtain a fine grain structure in twin-roll cast Al-Mn alloys. The annealing treatment included a pre-recovery process at low temperature and a subsequent recrystallization process at high temperature. It was experimentally demonstrated that the pre-recovery consumed appreciable stored energy and resulted in a segregation of needle-like precipitate. Recrystallization nucleation at the next high temperature annealing stage was enhanced by pre-recovery treatment, which leads to a fine grain structure.
Keywords: Al-Mn alloy; Recrystallization; Grain refinement; Pre-recovery; Precipitation

399 Effect of intermetallic particles on the microstructure and elevated-temperature properties of Zr-added A356 alloy

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The microstructure and intermetallics of Al-7Si-0.3Mg and Al-7Si-0.3Mg-0.15Zr (wt.%) casting alloys have been studied by Optical microscopy, Scanning electron microscope (SEM) with energy dispersive x-ray analysis (EDS) and Transmission electron microscope (TEM). The intermetallics formed in the microstructures for as-cast and solution treated had been examined. The effect of Zr addition and precipitation of AlSiZr intermetallics on the elevated-temperature properties of the Al-Si-Mg alloy was investigated. It has been found that the primary AlSiZr phase and AlSiZr precipitates with different morphologies and sizes are formed in the Al-7Si-0.3Mg-0.15Zr casting alloy. The primary AlSiZr phase exhibits flake-like morphology, while the AlSiZr precipitates exhibit nanobelt-like, rectangle-like and needle-like shapes. The investigation indicates the intermetallic phases formed during solution treatment, whilst particular emphasis has been placed on its growth mechanism or orientation relationship with the aluminum matrix. Corresponding, the elevated mechanical properties were improved by the addition of Zr, i.e. the tensile strength, the elongation and yield strength.
Keywords: Al-Si-Mg cast alloys, Zr, precipitate, solution treatment.

400 Microstructure evolution of 304L stainless steel during variable thermo-mechanical processing conditions: Experiment and simulation

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The thermo-mechanical processing (TMP) conditions in real industrial production usually changes continuously in terms of deformation temperature and/or strain rate. In this study, the microstructure evolution of 304L austenitic stainless steel during hot deformation was characterized with the temperature and strain rate changing in a controlled manner between two constant TMP conditions. The varying of temperature and strain rate was performed on Gleeble 3800 both before and after recrystallization is initiated. In particular, the transient area during and after fast and slow change of temperature and strain rate are analyzed and discussed in detail with respect to flow stress, grain size and dislocation density. A well-established mean field model, which uses grain size and dislocation density as the internal variables, is used to simulate the microstructure evolution at these variable conditions. The results show that more internal variables might be needed for the model to accurately account for the microstructure evolution in varying conditions.

401 Ultra-strong nano-twinned steel with large tensile elongation

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A twinning-induced plasticity (TWIP) steel strengthened by elegant arrays of laminated nanotwins was manufactured by a simple thermomechanical treatment consisting of cold rolling and recovery annealing. Different to other lab-scale methods making nano-structured materials, the present simple thermomechanical treatment is suitable for large-scale production in the steel industry using existing facilities, which makes the present steel being an attractive structure material. The steel achieved a high yield strength (1450 MPa), high ultimate tensile strength (1600 MPa) and considerable uniform tensile elongation (20%). During the recovery annealing, the nanotwins are thermally stable so that they remain in the sample after recovery annealing. On the contrary, the dislocation density was reduced greatly after recovery annealing. The deformation mechanism of the present nanotwinned steel is investigated by synchrotron X-ray diffraction, transmission electron microscopy, nanoindentation and electrical resistivity, illustrating that the dislocation density increases dramatically with strain while the volume fraction of nanotwins remains constant. Therefore, the work hardening behaviour of the nanotwinned steel is mainly provided by the accumulation of dislocations.

402 2.1 GPa ultra-strong nanostructured steel with unexpected large ductility

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There has been an increasing demand for high-performance steels for the construction of energy-efficient and lightweight structures in aerospace and automotive industries. High-performance steels require ultrahigh strength as well as good ductility. Nevertheless, strength and ductility are mutually exclusive. It is extremely challenging to design an ultrahigh strength steel with good tensile ductility, especially steels with yield strength higher than 2 GPa. The present work proposes a new method to meet such a challenge to produce an ultrastrong and ductile steel with a yield strength of 2.1 GPa and an unexpected large tensile uniform elongation of 20%. To the best of our knowledge, this steel may be the best so far in terms of the combination of yield strength and uniform elongation compared to those reported in the open literature. The present novel steel possesses a dual-phase hierarchical nanostructure consisting of nanosized precipitates, nanotwins, and lamellar nano- and ultrafine-grained austenite and ferrite. This novel steel has been strengthened by all the available strengthening mechanisms, including solid-solution, precipitation, dislocations, grain boundary and twin boundary strengthening. Besides dislocation plasticity, the novel steel also experiences transformation-induced plasticity (TRIP) as well as twinning-induced plasticity (TWIP). More importantly, different to other lab-scale methods of producing ultrahigh strength alloys, such as the severe plasticity deformation (SPD) technique, the present novel steel has been produced by conventional processing routes currently used in the steel industry. No additional facilities are required for the steel industry to produce this novel steel, which will facilitate its future industrial application.

403 Microstructure and mechanical properties of a spray-formed and hot worked ultra-high strength aluminum alloy

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The microstructure and mechanical properties of a spray formed and hot worked ultra-high strength aluminum alloy is researched in this paper, and the hot working process is consist of hot isostatic pressing (HIP), homogenization treatment, hot extrusion, solution and aging treatment. Metallographic microscope, scanning electron microscope (SEM) and tensile test are used to research the microstructure and mechanical properties. The Al-Zn-Mg-Cu alloys with five different chemical composition are spray-formed. These alloys are treated by a two-step homogenization (440°C/12h+472°C/24h, 36h, 48h) after hot isostatic pressing (HIP) with 450°C/8h. Hot extrusion are carried out at 380~400 °C in order to get as deformed microstructure. And then solution and aging treatment are carried out with 440°C/1h+472°C/3h and 120°C/12h, respectively. When the weight percent of alloy elements except aluminum exceeds 14wt.%, the secondary phase is very difficult to dissolve back. The porous defects of spray-formed ingot can be mostly eliminated by HIP and hot extrusion, and the tensile strength

of the alloy reach 800MPa through the process of HIP, homogenization treatment, hot extrusion, solution and aging treatment.

404 Effect of cold rolling on the size and shape of second phase particles in an Al-1% Si alloy

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In nanostructured Al alloys, high strength and high ductility can be achieved simultaneously by introducing well dispersed spherical fine particles. It is of significant importance to explore suitable heat treatment parameters to obtain high density spherical fine particles. In this study, an Al-1wt% Si alloy was designed and produced using an extremely pure Al(99.9996%) as the base material. The ingot was solid solution treated at 580°C for 12 hours and water quenched to obtain a supersaturated solid solution. The alloy was cold rolled to reductions of 30 and 80% before aging treatment. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used to analyze the sizes and shapes of the second particles after aging at a series of temperatures for different times. The results were also compared with sizes and shapes of the particles obtained from the same alloy without cold rolling before aging. The results showed that the higher density and smaller spherical particles can be obtained in the 80% cold rolled sample by long time annealing at low temperatures.

405 Enhanced strength and ductility by architecturing laminate structures of alternative fine and coarse grain sizes

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Architecturing the three-dimensional distribution of structural features such as the size and morphology of grains in single and dual (or multiple) phase alloys and phases in dual and multiple phase alloys is a new strategy to achieve property improvement. Bimodal or multimodal distribution of grain sizes is one typical example of such three-dimensional architecturing approaches to obtain improved combination of strength and ductility. Another example is the production of continuous gradient grain structure of grain sizes from nanometer to micrometer, which even leads to simultaneous enhancement in strength and ductility. In this work we apply produce a new three-dimensional architecture of laminate structure composed of alternating ultrafine and coarse grain sizes and investigate the mechanical properties and thermostability in a single phase Mg alloy and a dual phase steel. The results showed significantly improved strength and ductility due to activation of new strength and work hardening mechanisms.

406 Recovery or non-recovery in Al-0.1 Mg and Al-1 Mg alloy during high-pressure torsion processing

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Both Al-1 Mg and Al-0.1 Mg alloys were processed by high-pressure torsion (HPT) at room temperature. In Al-1 Mg alloy, hardness values in disc centre area are lower than disc edge area after ½ and 1 turn, and the area of lower hardness values in disc centre decreased as the number of turns increased from ½ to 1 turn, in the end the hardness values appeared to be reasonably homogenous along disc diameter as the number of turns increased to 5 and 10 turns. Al-0.1 Mg alloy displayed different hardness evolution behaviour: hardness values in disc centre area are higher than disc edge area after ½ and 1 turn, and the area of higher hardness values decreased as the number of turn increased from ½ to 1 turn, and the hardness values evolved towards homogeneity along disc diameter after 5 and 10 turns. EBSD microstructure investigations in Al-0.1 Mg alloy reveal that grain sizes are smaller and a few low-angle boundaries exist at the disc edge area after ½ and 1 turn, whereas grain sizes are much larger and high proportion of low-angle boundaries exist in disc centre area after ½ and 1 turn. This indicates the higher hardness values in disc centre area after ½ and 1 turn in Al-0.1 Mg alloy are related to the quick recovery in disc edge area, where material is subjected to the heavy shear strain.

407 Challenges of thermomechanical processing of a beta-stabilized gamma-TiAl alloy in a near conventional forging process

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Low density, high specific stiffness, elevated temperature strength retention as well as good environmental resistance make gamma-TiAl based alloys a promising substitute for currently used nickel-based superalloys as low pressure turbine blade application in modern aircraft engines. High and balanced mechanical properties in next generation aircraft engines favor a hot working strategy. Thermo-mechanical processing, however, of these gamma-TiAl alloys is a challenging task due to a narrow “processing window”. A “near conventional” thermo-mechanical processing technology has been developed by Böhler Schmiedetechnik GmbH & Co KG. Die temperatures above 700 °C and processing at standard atmosphere necessitate the usage of nickel-based superalloys as die material. Main requirements for die materials to produce sound forgings in an economic hot-die forging process are good mechanical resistance and microstructural stability at die temperature, low thermal expansion, good machinability, good weldability for repair reason and oxidation resistance at operating temperature and standard atmosphere. The present work describes the efforts on materials selection for die material as well as results of low pressure turbine blade forging trials at industrial scale.

408 Impact of silicon, magnesium and strontium on feeding ability of AlSiMg cast alloys

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In this paper it has been shown how characteristic solidification temperatures including rigidity temperature can be used to quantify the various feeding mechanisms that occurs during solidification of AlSiMg alloys. In additions, the impact of Silicon, Magnesium and Strontium on the temperature intervals of various feeding regions have been also analyzed.

409 New trends in high-pressure chemistry of materials

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In the last decade, systematic high-pressure experiments up to maximum pressures of 25 GPa have been carried out in our group focusing on high-pressure solid state chemistry using the multianvil technique [1]. In the context of these investigations, several new high-pressure polymorphs of known compositions were discovered. These investigations led to fundamental insights into the structural behaviour of oxoborates under high-pressure conditions. Next to the syntheses of new modifications, new compositions were realized in our group. For example, all attempts to produce rare-earth metal (III) oxoborates with the ratio $\text{RE}_2\text{O}_3:\text{B}_2\text{O}_3 = 2:3$, $1:2$, and $3:5$ failed under normal-pressure conditions. In contrast, the corresponding high-pressure experiments led in most cases to phase pure rare-earth metal (III) oxoborates. Our latest results opened up new fields of syntheses, which impressively underline the importance of the parameter pressure for the synthesis of new materials in solid state and materials chemistry [2]. [1] H. Huppertz, Chem. Commun. 47 (2011) 131-140. [2] G. Sohr, N. Ciaghi, M. Schauperl, K. Wurst, K. R. Liedl, H. Huppertz, Angew. Chem. Int. Ed. 54 (2015) 6360-6363.

410 Additive processing of materials with a higher carbon content

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Additive manufacturing processes (3D-printing) are regarded as a key technology in meeting challenges such as shorter development cycles, increasing competition for raw materials, the increase in component variants and the individualisation of products. It is possible to directly manufacture complex parts components with integrated functions and undercuts without using tools. The main advantages of additive manufacturing are the good material utilization and the possibility of producing complex geometries as with any other manufacturing process. Moreover, it is possible to process a variety of materials, such as Cr-Ni stainless steel, bronze, Ni, Ti, Al-based alloys, and CoCr alloys. Furthermore, the processing of other materials, such as ceramics, tool steels with a higher proportion of carbon, mixed materials and coatings with SLS, are also examined. The required high melting temperatures of metals lead to unfavorable stress conditions in the component. By using localised high-energy lasers, thermal stresses are

induced into the component. The resulting voltage gradients can lead to delamination between the layers and distortions in the part. Especially in the processing of materials with a higher carbon content and titanium alloys, cracking is observed. This problem can be minimized by the process control and preheating of the powder. It was found that the preheating of the powder at 300 °C is not sufficient to process the materials with a higher carbon content. Only a preheating of the powder to a temperature of about 400 °C allows for the crack-free processing of the materials with a carbon content higher than 0,3%.

411 Characterization and mechanical properties of a 0.2C steel produced by Q&P

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The third Generation of Advanced High Strength Steels produced by Quenching and Partitioning has been proposed as a novel heat treatment in order to produce steels with improved strength-ductility combinations. In the present work, the impact of quenching and partitioning conditions on the microstructure of a 0,2 %C steel was assessed. The microstructural evolution was characterized using SEM, EBSD, XRD and TEM. Phase quantification was further completed by dilatometry data. The obtained Q&P microstructures are mainly composed of tempered martensite, bainite and retained austenite.

The small fraction of bainite formed during the partitioning step is proportional to the amount of untransformed austenite, and thus to the quenching temperature. Furthermore, the austenite fractions after Q&P treatments showed significant differences when compared to the calculated values. The measured austenite fractions were found to be less sensitive to the quench temperature and were never larger than the predicted maximum fraction. Competing reactions such as austenite decomposition, carbide precipitation, interface movement and carbon trapping at lattice defects may explain this behavior. TEM observations were carried out and allowed the detection of carbides and interlath retained austenite. Moreover, the effect of the retained austenite and the influence of multiphase microstructures on mechanical properties of Q&P steels were studied. More specifically, tensile properties were linked to the microstructural quantifications and nano-indentation hardnesses. This is critically discussed and compared to quenched and tempered as well as to austempered microstructures.

412 Effect of alloying element on deformation behavior of binary magnesium alloys

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As the lightest of structural alloys, Mg alloys offer significant potential for weight reduction, but have yet to see significant application in automobiles, particularly in sheet form. One of the major drawbacks preventing such application is their poor formability at room temperature, originating from their strong basal texture and limited number of slip systems. Therefore, numerous studies have been conducted to improve the formability of Mg alloys by alloy modification and weakening/randomizing the texture. Although the effect of alloying elements on the modification of texture is relatively well understood, the intrinsic effect of alloying

elements on deformation behavior of Mg alloys is not clearly understood yet.

The present work is aimed at having a better understanding of the effect of alloying elements on deformation behavior of Mg alloys. Among various elements utilized in Mg alloys, four representative alloying elements have been chosen; Al and Zn that are most commonly used alloying elements in Mg alloys, Sn that is known to increase ductility and promote significant precipitation hardening in Mg alloys, and Y that represents rare earth elements used in numerous Mg alloys. The binary alloys containing these elements have been cast and subjected to various thermomechanical treatment to have the similar grain size. Their deformation behavior has been analyzed by TEM, EBSD and neutron diffraction analysis and compared with the VPSC simulation analysis to identify the role of each alloying element in the deformation behavior.

413 Non-classical crystallization of thin films and nanostructures synthesized by chemical vapor deposition

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Extensive studies have been made on non-classical crystallization, which refers to the crystal growth by the building unit of nanoparticles. In parallel with the non-classical crystallization in the crystal growth in solution, the non-classical crystallization in the gas phase synthesis of thin films and nanostructures by chemical vapor deposition (CVD) and some physical vapor deposition (PVD) has been studied extensively. Here, the charged nanoparticles (CNPs) are spontaneously generated in the gas phase and become the building block of thin films and nanostructures. Charged nanoparticle-based crystallization appears to be very general, including the growth of diamond, Si, ZrO₂, GaN, ZnO films as well as nanowires. The generation of CNPs in the gas phase was experimentally confirmed in many systems and their mass distribution was shown to play a decisive role in the microstructure evolution of films, nanowires, and nanotubes. The fact that CNPs can be a building block of crystals without leaving any void behind and of nanowires with smooth surface indicates that CNPs are quasi-solid, having a liquid-like property in diffusion. This means that the charge enhances the atomic diffusion, which is a newly discovered physical phenomenon. This means again that charge weakens the bond strength. Small nanoparticles can be liquid-like even if singly charged but large nanoparticles should be multiply charged to be liquid-like.

414 Microstructure and processing map development of Ti-Al-Fe alloy

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Ti-Al-Fe alloy is a recently developed alpha+beta type titanium alloy which contains Fe as a beta stabilizer to replace expensive beta stabilizing elements such as V, Mo. The alloy exhibits high strength and excellent hot workability compared to Ti-6Al-4V alloy. The hot deformation behavior and microstructural evolution of Ti-Al-Fe alloy have been investigated in the present study. The hot deformation behavior of the alloy has been characterized by flow curves obtained

by hot compression tests. Processing map of hot compressed Ti-Al-Fe alloy was developed by using Dynamic Materials Modeling (DMM) approach for finding optimum working conditions. Microstructural analysis of the alloy was investigated to consider phase transformation during thermo-mechanical processing using SEM and TEM. The volume fraction of beta phase in Ti-Al-Fe alloy was higher than that of Ti-6Al-4V alloy because Fe is strong beta stabilizing element. Temperature and strain of the alloy were controlled to obtain the fine grained microstructure through dynamic recrystallization (DRX) during thermo mechanical processing. The microstructure of the alloy which is solution heat treated at alpha+beta phase region consists of alpha and metastable beta. When the alloy was solution heat treated and hot deformed at 880°C, fine B2 precipitates which have high Fe segregation and coherent relationship with beta matrix were observed. The fine B2 precipitates were disappeared in the alpha phase, and moved to the boundaries of alpha and beta phase.

415 Micro-scale strength evaluation for bonding interface of cold sprayed coatings

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The local and micro-scale strength of the bonding interface of cold spray coating is an important factor to determine the macroscopic strength. In order to understand the strength characteristics of the entire cold spray coating, SEM-EBSD structural evaluation and the micro-strength test utilising the FIB (Focused Ion Beam) system have applied for the interface.

416 Magnesium spinel oxides undergoing spinel-to-rocksalt transition for magnesium battery cathodes

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Magnesium rechargeable batteries (MRBs) research is a very challenging field, and we currently have many issues to be overcome to accomplish MRBs. For example, there are few cathode materials with high capacity (> 150 mAh/g) and high potential (> 2 V vs Mg) for MRBs. Thus, in order to change the energy storage paradigm, we have to seek cathode materials for polyvalent cations including Mg cations. Here, we focus on magnesium spinel oxides as candidates for MRB cathode materials. The lattice sites in the spinel structure are denoted as 8a, 16d (cation sites), and 32e (oxygen sites) in the Wyckoff position in the space group No. 227, while those in the rocksalt structure are denoted as 16c, 16d (cation sites) and 32e when it is assigned to the same space group. Thus, spinel can be regarded as rocksalt whose 16c sites are vacant and instead the 8a sites are occupied by cations. In this work, we demonstrate that Mg ions are inserted at high potentials (2-3 V vs Mg) onto 16c vacant sites in the spinel lattice with a “push-out process” of the already-existed cations from 8a to 16c, which leads to the formation of rocksalt phase.^{1,2} Finally, we discuss the feasibility of Mg insertion/extraction into/from the spinel oxides in terms of stabilities of the resultant rocksalt phases. 1. T. Ichitsubo, S. Okamoto et al., J. Mater. Chem. A 3, 10188 (2015). 2. S. Okamoto, T. Ichitsubo et al., Adv. Sci. 2, 1500072 (2015).

417 Thermal conductivity and tensile property of Mg-Zn-Y casting alloys with long-period stacking ordered phase

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The Mg-Zn-Y alloy with long-period stacking ordered (LPSO) phase is known as attractive for automotive lightening engine components with strength at elevated temperature. On the other hand, it is considered that the thermal conductivities of magnesium alloys are lower than those of commercial aluminum alloys. Low thermal conductivity causes engine performance degradation. However, there is little study on the thermal conductivity of Mg-Zn-Y casting alloys. It is important study, if we consider the application of hot parts for engine components. Then we developed the Mg-Zn-Y casting alloys with strength at elevated temperature and good thermal conductivity. Our developed alloys have the fine LPSO phase network for higher strength and the pure magnesium matrix for better thermal conductivity. It is important that there is little or no solute element in the magnesium matrix for thermal conductivity. We accomplished these two good characteristics at the same time by optimizing the amount of zinc and yttrium contained in the Mg-Zn-Y casting alloys. $\text{Mg}_{95.8}\text{Zn}_2\text{Y}_2\text{Zr}_{0.2}$ casting alloy had a good thermal conductivity of over $100(\text{W m}^{-1} \text{K}^{-1})$ at 473K. This is almost identical to the thermal conductivity of heat resistant aluminum casting alloys for conventional engine components.

418 Visualization of elastic strain around various interfaces by TEM image analysis

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Transmission electron microscopy (TEM) is well known as one of powerful tools to investigate not only its microstructure but also chemistry at a nano-scale in the crystalline materials. Through the recent development of the digital image acquisition by means of TEM, the quantitative image analysis has attracted with considerable interest. In this presentation, I will show some results about the quantitative image analysis of the elastic strain around the various interface. As one example, the quantitative image analysis of the misfit dislocation at a Si/SiGe hetero interface in the strained-SGOI wafer has been studied. The elastic strain around the misfit dislocation, which is introduced to relax the misfit strain between Si and SiGe layer during annealing, are distributed at the tip of the dissociated partial dislocations within a few nm.

419 Neutron scattering studies of aluminum-based hydrides by high intensity total diffractometer (NOVA)

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A high intensity neutron total diffractometer, NOVA, at J-PARC can observe crystalline structure as well as amorphous and liquid structure in a short time, and clarify the structural changes during hydrogen absorption and desorption reactions of the hydrogen storage materials. Aluminum trihydride, AlH_3 , is one of the potential candidates for hydrogen storage materials because of high gravimetric and volumetric hydrogen densities (10 mass% and $149 \text{ kgH}_2/\text{m}^3$) and a simple hydrogen desorption reaction ($\text{AlH}_3 \rightarrow \text{Al} + 3/2\text{H}_2$) at 370-470 K. We investigated the structures of $\text{AlD}_3/\text{AlH}_3$ before the hydrogen desorption reaction by NOVA/X-ray diffraction (BL02B2 at SPring-8) measurements. The presence of $\gamma\text{-Al}_2\text{O}_3$ on the surface may prevent the deuterium/hydrogen desorption reaction of $\text{AlD}_3/\text{AlH}_3$ particles at room temperature. Also, the local structures of amorphous phase during the direct thermal decomposition reaction from $\text{LiAl(ND}_2)_4$ to Li_3AlN_2 and AlN were analyzed by PDF (Pair Distribution Function) analysis. Furthermore, mechanism for reversible disproportionation reaction of $\text{NaAlD}_4\text{-TiCl}_3$ was studied by in-situ neutron scattering measurements under deuterium gas atmosphere. This work was partially supported by the New Energy and Industrial Technology Development Organization (NEDO) under “Advanced Fundamental Research Project on Hydrogen Storage Materials (HydroStar)” and “Feasibility Study on Advanced Hydrogen Storage Materials for Automotive Applications (2012)”, JSPS KAKENHI Grant Numbers 23686101, 24241034, 15K13810, the Inter-university Cooperative Research Program of the Institute for Materials Research, Tohoku University (Proposal No.15K0110) and the Neutron Scattering Program Advisory Committee of IMSS, KEK (Proposal No. 2014S06).

420 Stress and strain analysis in an Fe-Ga alloy single crystal

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It is well known that Fe-Ga alloys show large magnetostriction values more than 200 ppm. They are expected to be used as a high performance vibration power generation apparatus by electromagnetic induction. Previous papers suggest that the magnetostrictive properties are closely related to the stress state in this alloy. For example, compressive stresses are shown to enhance the magnetostriction along the loading direction¹⁾. Thus, it is very important to clarify the relationship between the stress/strain states and magnetostrictive properties. In this study, we carried out in situ x-ray diffraction measurements of an Fe-18at%Ga alloy single crystal under magnetic fields. We used a Goss-oriented square plate (dimensions : 10 mm × 10 mm × 1 mm height) cutting from the as-grown single crystal ingot produced by the Czochralski method. The magnetic fields were applied in-plane. The influence of magnetic field direction on the stress/strain states was precisely analyzed by using our original x-ray single crystal

stress/strain measurement method. As a result, we obtained tri-axial magnetostriction values.

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421 Fiber texture of groove rolled Ti-Nb-Al biomedical shape memory alloy

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Effect of reduction rate (r) and heat-treatment temperature on fiber texture of groove rolled Ti-24Nb-3Al biomedical shape memory alloy was investigated by X-ray pole figure measurement and electron back scatter diffraction patterns analysis. An alloy ingot was fabricated by Ar-arc melting, homogenized at 1273K for 7.2ks and quenched into water. Groove rolling with $r = 60\% \sim 99\%$ was made. The apparent phase of the alloy was the beta-phase (bcc) at RT. The fiber texture of the as-rolled material was $\langle 100 \rangle$ at $r = 60\%$. $\langle 110 \rangle$ fiber texture was developed for $70\% < r < 95\%$ and $\langle 102 \rangle$ fiber texture appeared at $r = 99\%$. The $\langle 102 \rangle$ fiber texture is suitable for the bending deformation because tensile and compressive components of lattice deformation are well balanced along this direction. Recrystallization at various temperatures for 3.6ks changed the fiber textures. Optimum condition of thermomechanical treatment is discussed on the view point of fiber textures, crystallographic anisotropy of lattice deformation and Young's modulus.

422 Mechanism of recrystallization texture evolution during solution treatment for age-hardenable Al-Mg-Si alloy sheets fabricated by cold rolling and asymmetric warm rolling

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We recently succeeded in developing a favorable $\{111\}\langle 110 \rangle$ texture for deep drawing in solution-treated materials of AA6022 alloy sheets fabricated by cold rolling at high reduction and subsequent differential-speed warm rolling at low reduction. To more intensify the $\langle 111 \rangle$ //ND texture components, this study has focused on the mechanism of $\{111\}\langle 110 \rangle$ recrystallization texture evolution during solution treatment for the combined rolling processed AA6022 sheets containing a large number of particles. The $\{111\}\langle 110 \rangle$ recrystallization texture developed significantly during high-temperature annealing for an extremely short time. TEM observations in the as-rolled or recovered condition revealed that $\{111\}\langle uvw \rangle$ orientations exhibited lower dislocation density within a subgrain and fewer fine particles at subboundaries than other orientations. EBSD results showed that the $\{111\}\langle 110 \rangle$ oriented regions tended to possess the approximately 40 degrees rotation relationship around common $\langle 111 \rangle$ axes with neighboring areas at a relatively high frequency in the early stage of recrystallization, and that particle-stimulated nucleation hardly contributed to the formation of $\{111\}\langle uvw \rangle$ recrystallized grains. Additional shear deformation and precipitation during asymmetric warm rolling would be closely related to the evolution of $\{111\}\langle 110 \rangle$

recrystallization texture.

423 Effect of annealing upon retention of He and H in irradiated SiC

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The retention of hydrogen isotope fuel and helium ash from the fusion environment, as well as the interaction between radiation damage and these retained species, requires further investigation and the results will help to understand and predict failure mechanisms of components, the lifetimes for retention of acceptable limits of tritium, and the degradation of SiC thermal properties. SiC samples were irradiated with He ions of energy up to 30 keV and a fluence up to $10^{16}/\text{cm}^2$, to produce damage in the near-surface region. A duplicate set of He ion irradiated SiC samples, as well as undamaged SiC, were also irradiated with H_2^+ ions of energy up to 20 keV and a similar fluence, to study the interaction of H species with SiC and radiation-damaged SiC by He. Modification to the surface following irradiation is observed via Raman spectroscopy, which exhibits development of damage states such as disordered carbon and Si-Si peaks. The retention of H and He were measured using elastic recoil detection analysis using 7.8 MeV C^{3+} ions. Changes to the surface chemistry were characterised using X-ray Photoelectron Spectroscopy and Near Edge X-ray Absorption Fine Structure spectroscopy. Samples were annealed in steps of 200 K, from 473 K to 1273 K, and ERDA was performed after each anneal step. Only minor changes in the H and He profiles were observed up to 1,073 K, however after the 1,273 K anneal the H and He profiles changed considerably, with a marked difference between samples irradiated only with He and those irradiated with He and H.

424 Superelasticity of rolled Ti-Nb-Zr alloy

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Superelastic alloys Ti-Nb-Zr obtained the recognition as biocompatible materials for orthopedic and dental surgery. Superelasticity includes two stages: (1) elastic deformation by $\sim 0,2\%$ due to with interaction of neighboring atoms, (2) elastic deformation by $2\div 3\%$ and more due to martensitic transformations (MT). Features of superelastic deformation in the foil of alloy Ti-22%Nb-6%Zr (at. %) were studied by methods of X-ray texture analysis. At low stress, less than the yield point of beta-phase, in the course of MT the phase beta with BCC crystalline lattice reversibly turns into the phase alpha" with orthorhombic lattice. The orientation relationship between both phases is a such, that the increase of interplanar spacings by the transition from beta-phase to alpha"-phase in the case of tensile test promotes to growth of elastic deformation up to degree of 2,5%, whereas in the case of compressive test it impedes the elastic deformation of sample above degree of 1%. According measurements of X-ray line broadening, which characterizes the substructure condition of reflecting grains, that MT in the textured bent foil develops inhomogeneously – by the deformation degree of surface layer 1,5% at the compressed side of foil MT is localized in less fragmented grains with the rolling plane

(001), while in grains with the rolling plane (111) MT is practically absent. Reversible broadening of X-ray lines is conditioned by fragmentation of beta-grains in the course of MT due to spreading of martensitic needles, whereas irreversible broadening is connected with dislocation plastic deformation.

425 Effect of microstructural change by rolling and annealing on hydrogen permeability of Nb-TiNi and Nb-TiCo eutectic alloys

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The Nb₁₉Ti₄₀Ni₄₁ alloy shows very fine lamellar structure consisted of the bcc-(Nb, Ti) solid solution and B2 intermetallic compound in the as-cast state. On the contrary, similar microstructure is observed in the as-cast Nb₃₀Ti₃₅Co₃₅ alloy consisted of the bcc-(Nb, Ti) and B2-TiCo phases. These alloys show good hydrogen permeability with resistance to hydrogen embrittlement, so that they are strong candidates for non-Pd hydrogen separation and purification alloys. When these alloys are rolled and annealed, hydrogen permeability is drastically reduced in the Nb-TiNi alloy but unchanged in the Nb-TiCo alloy. That is, hydrogen permeation behavior in these alloys is very different, although they have similar metallurgic characteristics. In the present study, their hydrogenation behavior is compared and controlling factors of hydrogen permeation are discussed on the basis of experimental results.

After rolling and annealing, microstructures of these two alloys were changed to the granule bcc phase embedded in the B2 phase. Because hydrogen permeability Φ is expressed as the product of hydrogen solubility coefficient K and hydrogen diffusion coefficient D , the values of K of these two alloys were obtained by PCT measurements. Hydrogen solubility coefficient K of the Nb-TiNi alloy was five times higher than that of the Nb-TiCo alloy. On the other hand, hydrogen diffusivity D , estimated by the relationship between Φ and K , was 200 times higher in the Nb-TiCo alloy. Hydrogen permeability was provided by large K for Nb-TiNi alloy but by large D for Nb-TiCo alloy under the same experimental conditions.

426 Hydrogenation behavior and structural change of LPSO Mg-based alloys

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The LPSO phases stably exist in the Mg-Tm-Y (Tm: transition metals) alloy systems. They have complex structures such as long period stacking, Tm-Y condensed planes and chemical ordering configuration in Tm-Y condensed planes, which improve mechanical properties of Mg-base alloys. They also have attracted interest of many researchers as metallic hydrogen storage media. In the present study, hydrogenation behavior and structural change of LPSO Mg-Zn-Y alloys are investigated. The alloy ingot, which was prepared by a high-frequency induction melting method, was pulverized using a diamond file and the alloy powder was sieved through a #150 mesh screen in air. Equilibrium pressure-composition-temperature (PCT) measurements were performed using Sievert-type apparatus. The sample was heated to 673 K and held for 1 h to achieve activation. After activation, 7N hydrogen gas was introduced into

the chamber. The structure of the sample was identified using a powder X-ray diffraction (XRD). The LPSO Mg-Zn-Y alloy absorbed about 0.15 H/M hydrogen below 0.1 MPa H₂ at 673 K, the alloy decomposed into hcp phase and YH₂ hydride. Therefore, the LPSO alloy was broken by hydrogenation. The wide plateau pressure was observed at around 1.5 MPa H₂ for 673 K. Finally, the sample absorbed 4.6 (H/M) hydrogen and decomposed into YH₂, YH₃, MgH₂ and MgZn₂ phases. After PCT measurements, 0.3 H/M of hydrogen still remained in the sample. The LPSO phase was not reformed under the present experimental conditions.

427 Effects of the pore size on mechanical property and tissue response to porous carbonate apatite made by the setting reaction of carbonate apatite granules

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Carbonate apatite (CO₃Ap) is replaced to bone. The process may be accelerated by introduction of the porous structure. In this study, therefore, interconnected porous CO₃Ap was fabricated based on setting reaction of CO₃Ap granules, and effects of pore size on mechanical properties and tissue response to the interconnected porous CO₃Ap was evaluated histologically.

CO₃Ap granules with 100-200μm and 300-600μm in diameter were exposed to acidic calcium phosphate solution. CO₃Ap granules were bridged by the DCPD crystals. When the porous body was further immersed in NaHCO₃ solution, DCPD formed on the surface of CO₃Ap granules was transformed to CO₃Ap, resulting in the fabrication of interconnected porous CO₃Ap. Compressive strength and pore size were 1.5MPa and 2.2MPa, and 100-200μm and 30-100μm, for porous CO₃Ap fabricated using large and small CO₃Ap granules.

When porous CO₃Ap was implanted in the rabbit tibiae, bone penetration inside the porous structure was confirmed within 4 wks in both case. In contrast, no bone penetration was observed in the case of dense CO₃Ap. After 12 wks, larger amount of bone was found inside the porous CO₃Ap, and CO₃Ap was replaced to the bone. Interestingly no significant difference on replacement to bone was observed based on the pore size of the porous CO₃Ap under the condition used in this experiment. We concluded, therefore, interconnected porous CO₃Ap is promising as the artificial bone replacement.

428 Apatite orientation and material property of bone are enhanced by artificially elevated load

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Bone adapts to mechanical environment and rebuilds their macro- and micro-structure to make the bone stronger to the environment, which is defined as functional adaptation of bone. Although the anisotropic arrangement of bone extracellular matrix (ECM) is clarified to be an important contributory factor for bone mechanical function [1, 2], bone functional adaptation through the change in the ECM orientation remains understood. The present study aims at clarifying how bone changes its anisotropic ECM arrangement under artificial load in order to clarify bone's adaptive response to the mechanical environment. SD rats were received axial cyclic compressive load that is higher than physiological load onto their ulnae by utilizing

fatigue testing machine. The bone formation rate, preferential orientation of ECM dominantly composed of biological apatite and collagen, bone mineral density (BMD), and Young's modulus were analyzed in the artificial load-induced newly formed bone. The artificially loaded ulna showed enhanced bone formation at the bone surface comparing to the control bone that was not artificially loaded. The artificial load-induced bone possessed significantly higher BMD and the degree of apatite along the bone long axis, resulting in improved mechanical function. This finding might lead to a deeper understanding of bone adaptation to in vivo mechanical environment not only through bone mass but also anisotropy in bone material parameter. [1] T. Nakano et al.: Bone 31 (2002) 479-487.
[2] T. Ishimoto, T. Nakano et al.: JBMR 28 (2013) 1170-1179.

429 Hydrogenation properties of supported metal nanoparticles on graphene

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It has been reported that Fe nanoparticle on graphene can absorb hydrogen at low temperature by density functional theory (DFT) calculation [1]. In this work, we experimentally investigated hydrogenation properties of metal nanoparticles of Fe, Ti and Au on graphene. The metal nanoparticles were evaporated on single layer graphene on Cu substrate by vacuum evaporation method. The samples were hydrogenated under 1.0MPa H₂, for 10 min, at 77K, 373K, and dehydrogenated from room temperature to 673K and analyzed using mass spectrometer (MS). Hydrogen desorption was observed around 440K for Fe, around 420K for Ti, and not observed for Au. Observed hydrogen desorption was caused by metal nanoparticles because we could not observed hydrogen desorption when we performed same experiment to graphene on Cu substrate. [1] K. Takahashi et al., Low temperature hydrogenation of iron nanoparticles on graphene, Scientific reports, 4: 4958 (2014)

430 Effect of Ni on the coefficient of thermal expansion and Young's modulus of Fe-Ni-Nb-Ti Invar alloys

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Fe-Ni-Nb-Ti Invar alloys with low coefficient of thermal expansion (CTE) and high Young's modulus have been used for many applications where high dimensional stability is required. However, the expression mechanism of material properties such as CTE and Young's modulus of Fe-Ni-Nb-Ti Invar alloys has not been thoroughly clarified. In this study, the effect of Ni on CTE and Young's modulus of Fe-Ni-Nb-Ti Invar alloys has been investigated. Fe-(36,38,40,42,44 and 46wt%)Ni-3.3wt%Nb-1.4wt%Ti alloys were fabricated by vacuum induction melting. Ingots were hot forged and then solution-treated at 1373 K for 1 hr followed by ageing at 873 K for 100 hrs. The microstructure, CTE and Young's modulus were examined. In aged samples, gamma double prime phase precipitated and 40wt%Ni alloy showed the minimum of the CTE value. Thermodynamic calculation revealed that the amount of solute Ni in the matrix of 40wt% alloy was almost 36wt% after gamma double prime precipitation. Therefore, it seems that the low CTE of the aged 40wt%Ni alloy would be brought by its solute

Ni amount which equals to the one in conventional Fe-Ni Invar alloy. The effect of Ni content on Young's modulus in aged samples also is explained by the amount of solute Ni. In this presentation, we will discuss the effect of gamma double prime precipitation on CTE and Young's modulus in Fe-Ni-Nb-Ti Invar alloys.

431 Structural change in melt-quenching Ni-Zr glassy alloy due to the deuterium absorption

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Structural features of Ni-Zr glassy alloys have been widely studied due to the continuing interest in the relation with their glass forming ability. These glassy alloys are also interesting from the perspective of the hydrogen absorption because their hydrogen absorption properties differ from those of corresponding crystal alloys. However, only a few structural studies have been conducted on their hydride (deuteride) samples. We have performed pulsed neutron diffraction and synchrotron X-ray diffraction measurements on melt-quenching $\text{Ni}_{0.67}\text{Zr}_{0.33}$ glassy alloy and its deuteride to investigate the structural change due to the deuterium absorption. The comparison of the pair distribution functions for these two samples shows that the local elongations of the Ni-Ni and Ni-Zr correlations are nearly equal to an average elongation but that of the Zr-Zr correlation is larger than the average elongation. Namely, the glass structure is inhomogeneously expanded due to the deuterium absorption. We report detailed differences in the structural features between the $\text{Ni}_{0.67}\text{Zr}_{0.33}$ glassy alloy and its deuteride based on the three dimensional structure models obtained by reverse Monte Carlo simulation.

432 Preparation of Mg-TM-Y (TM=Transition metal) alloys with long period stacking ordered phase and their superior mechanical properties

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Magnesium alloys have high potential as structural components in aerospace and automotive applications, because of their low density and high specific strength. In particular, Mg alloy with long period stacking ordered phase can be expected as excellent mechanical property. It is known that the LPSO phase is forms in Mg-TM (transition metal) -RE (Rare earth metal) systems as stable phase. Therefore, Mg alloy with LPSO phase was developed using a conventional method, cast, extrude, or rolling and they showed excellent mechanical properties. In present study, we have developed the high strength Mg-TM-Y alloys with the LPSO phase. The Mg-TM-Y alloy ingots were prepared by using an electric furnace in Co_2 atmosphere. The Mg-TM-Y alloys sheets were prepared by hot-rolling at 623K, and subsequently annealed at 673K. Microstructure was investigated by SEM and TEM. The Mg-Ni-Y alloy sheets prepared by hot-rolling exhibited high 0.2% proof stress at room temperature. For example, The $\text{Mg}_{94}\text{Ni}_2\text{Y}_4$ rolled-sheet which is consist of Mg and LPSO phases exhibited high 0.2% proof stress of 393MPa and elongation of 7%. The 0.2% proof stress of the Mg-TM-Y alloy tend to increase to increasing of volume fraction of the LPSO phase. The Mg-TM-Y alloy sheet combining high strength and reasonable elongation was achieved by hot-rolling and subsequent

appropriate thermal annealing, using the LPSO phase as a strength factor.

433 Scaling-up the high pressure torsion for processing of ultrafine-grained billets

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High Pressure Torsion is one of the most effective Severe Plastic Deformation techniques in respect of microstructure refinement and properties enhancement in processed materials. The main limitation of this method is small dimensions of final samples. Up to now, a major disadvantage of the HPT method has been the small dimensions of the samples (typically disks of 10 mm diameter and less than 1 mm thickness), which prevents its commercial use for structural applications. Recently we proposed to scale-up the HPT method for industrial processing of advanced materials with at least three order of magnitude larger yield using a new technical approach. The novel machine allows the continuous processing of long rods instead of the thin plates used in conventional HPT. The basic idea of the method relates to the continuous transmission of the shear deformation zone along the metallic rod that is pushed through the die. The proposed method has an important advantage when compared to other well-known large scale SPD techniques, i.e., equal channel angular pressing and accumulative roll bonding, as it allows the processing of the billet in one pass without intermediate steps and, thus, allows for continuous fabricating nanocrystalline rods. Furthermore, continuous HPT (C-HPT) provides exciting opportunities for the purposely design of nano- and microstructures. Several examples of application of C-HPT for processing of Cu and Al alloys will be presented and discussed.

434 Microstructural and mechanical properties of welded joints of 690 MPa grade QT and TMCP steel

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The microstructure and mechanical properties of 8 mm thick GMAW welded joints of two 690 MPa grade steels: quenched and tempered (QT) and thermomechanically controlled processed (TMCP) are studied. It is shown that the heat affected zone (HAZ) of the same grade steels welded under the same welding conditions have a considerably different microstructure and properties. Analysis of the hardness distribution is clearly indicated that the maximum decrease in hardness of the 690 grade steels reaches 20-30% of the base metal. Impact toughness measurement shows that heat input in the range 1.0 to 1.7 kJ mm⁻¹ has a weak effect on the impact energy in the HAZ.

435 Comparative study of microstructural and mechanical inhomogeneity of laser and friction stir welded joints of Al-Mg-Si alloy

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Microstructural and mechanical inhomogeneity of laser (LBW) and friction stir (FSW) welded joints of 4 mm thick 6082-T6 Al-Mg-Si alloy was studied. Thermal cycles in heat affected zone (HAZ) were measured during welding and analysed. It had been found that tensile properties and ductility of joints is significantly lower than those of the base metal. Total elongation of FSW joint is 4 times lower than that of the base metal, LBW joint - 6.5 times lower. Measured local microhardness of joints and local strain in tensile specimens revealed that fracture of the joints corresponds to the HAZ as a region with minimum hardness. It is explained by the overaging (coarsening of the strengthening particles) in HAZ. Two types of post weld heat treatment (PWHT) were studied: (i) solution heat treatment (SHT); (ii) SHT followed by an artificial aging (SHTA). Microstructural examination of PWHT joints did not reveal any grain growth. SHTA treatment improves both tensile properties and ductility of joints but not to base metal level.

436 Microstructure of the Mg₉₆Zn₂Y₂ alloy joints welded by ultrasonic spot welding

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Development of the welding technique for the Mg-Zn-Y alloys joint is indispensable to extend their application. We applied ultrasonic spot welding to Mg₉₆Zn₂Y₂ alloy plates to obtain high strength joints with low welding energy. During ultrasonic welding, plastic deformation and heat rise are expected to contribute to the formation of the welded microstructure. In this study, the microstructure of the welded interface was observed in detail, and the controlling factor of the interface formation and the effect of the ultrasonic welding on the microstructure of the Mg₉₆Zn₂Y₂ alloy were discussed. The sample we used was an extruded Mg₉₆Zn₂Y₂ alloy. Plates were cut from the extruded alloy and welded using an ultrasonic welder. The microstructure of the joints exhibited the maximum weld strength was observed. Cross-sections of the welded interface were observed by FE-SEM and STEM. In the joints of the Mg₉₆Zn₂Y₂ plates, the narrow band consisted of fine Mg grains was observed along the welded interface. Outside the band, the original Mg grains were grown. The LPSO phase in the Mg₉₆Zn₂Y₂ alloy restricted the width of the narrow band and the size of the grown Mg grains. At the vicinity of the welded interface, the band was corrugated showing severe plastic deformation. The Mg₉₆Zn₂Y₂ alloy joint microstructure showed the variation of the interface formation mechanism by the difference of places around the welded interface.

437 Tensile and wear properties of TiB/Ti and TiC/Ti composites with different Ti powders prepared by spark plasma sintering

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Titanium and titanium alloys are widely used in aerospace, chemical, and biomedical applications because of their high strength-to-weight ratio, high fatigue strength, good corrosion resistance, and good biocompatibility. However, they are underutilized in industry due to their poor wear resistance. The addition of TiB or TiC in titanium enhances their wear resistance. To further improve their wear properties, TiB- and TiC-reinforced Ti matrix composites (TiB/Ti and TiC/Ti) were produced by the spark plasma sintering (SPS) process. Hydride-dehydride (HDH) and gas-atomized (GA) pure Ti powders with different powder sizes were used as a matrix, and TiB₂ or TiC powders were used as a reinforcement. We investigated the microstructures, Vickers microhardnesses, and the tensile and wear properties of the composites. The ultimate tensile strengths, the stiffnesses, and the Vickers microhardnesses of TiB/Ti composites were higher than those of TiC/Ti composites. On the other hand, the TiC/Ti composites exhibited better wear resistance than that of the TiB/Ti composites.

438 Enhanced fatigue-properties of high strength aluminum alloy by coating with metallic glass thin films

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7075 aluminum alloy is widely used in sports equipment, automobile bodies, and aircraft frames because of its high specific strength/weight ratio. However, the 7075 aluminum alloy performs poor fatigue property in comparison to steel. In this study, 200-500 nm Zr-based and 200 nm Ti-based metallic glass thin film (MGTF) coatings were successfully coated on the 7075 aluminum alloy substrates by DC sputtering method. Then the fatigue properties of these samples were tested by following the standard of ASTM-C1161-02c. The fatigue life of 7075 aluminum alloy can be improved about 24.4 and 8 times than the bare one at 250 MPa stress level by coating with 200 nm-thick Zr-based and Ti-based MGTF, respectively. In parallel, the fatigue limit can be improved from 150 MPa (bare one) to 235 MPa and 205 MPa by coating with Zr-based and Ti-based MGTF, respectively. The formation of offsets and cracks from the surface of specimen was revealed to be effectively restricted by both Zr-based and Ti-based MGTF coatings. This is attributed by the high strength, good flexibility, and strong adhesion of both MGTF coatings, which can provide effective compressive stress to suppress the slip band protruding. In addition, the fatigue life and fatigue limit shows opposite trend with increasing the thickness of MGTF due to the changes of surface roughness and residual stress. The optimum performance was revealed on the sample coated with 200-nm-thick Zr-based MGTF. In summary, Zr-based MGTF coating is believed a promising coating material for improving 7075-T6 aluminum fatigue properties.

439 Improving the control of precipitation strengthening during thermomechanical processing of V-bearing micro-alloyed steel by application of in-situ EBSD and phase field modelling methods

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Current generation of automotive precipitation hardened V-bearing micro-alloyed steels is developed with aim to deliver extended stretch-flange formability through C content reduced below 0.05 wt. %, limiting the microstructure to a single phase ferrite. The metallurgical challenge is to maintain the yield strength above 650 MPa to allow thin final gauges for light-weight automotive components. It has been established that interphase precipitation during austenite to ferrite transformation may lead to a dense arrangement of closely packed sheets of V containing carbides and nitrides. However it is not yet fully understood under which conditions of austenite to ferrite transformation, e. g. cooling rates, isothermal holding temperatures and holding times, maximum precipitation strengthening can be achieved. Hot Stage Electron Backscattered Diffraction in combination with High Resolution Transmission Electron Microscopy is applied in this paper to characterise in-situ the role of cooling rate on the austenite to ferrite transformation and indirectly on the distribution of interphase precipitates. Crystallographic analysis of overlapping austenite/ferrite EBSD maps is performed with aim to explain the role of austenite to ferrite orientation relationship on the interphase precipitation morphology. Complementary to in-situ experiments, a phase-field method is employed to simulate interface precipitation. The growth of precipitates is simulated in 3D and various anisotropy conditions are studied, showing that the elastic energy contribution to precipitate morphology becomes dominant at later stages of precipitation. Experimental modelling results shows a good correlation between TEM and phase field modelling with regard to shape, size and distribution density associated with interphase precipitation.

440 Producing Ti-based amorphous/nanocrystalline powder using high-energy mechanical milling

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Ti-based crystalline alloys have numerous beneficial properties but their high strength can be further enhanced by amorphous/nanocrystalline structure. The present research reports on high energy ball-milling of crystalline Ti-based alloy. Ingots with composition of Ti_{43.2}Cu₃₈Ni₁₀Zr_{7.8}Al_{0.5}Si_{0.5} (at.%) were prepared by arc-melting a mixture of constituent elements in a Ti-gettered argon atmosphere. The structure of the initial crystalline alloy was examined by X-ray diffraction and scanning electron microscopy. Scanning electron microscopic observations revealed the presence of different solid solution phases. The master alloy was grinded and fractioned to a particle size below 300 µm for ball milling. The maximum milling time was 25 hours. It was found that drastic structural changes took place after first 5 h of milling based on the X-ray diffraction; (Ti,Zr)(Cu,Ni) phase remained largest volume

fraction among the initial phases. No significant change can be observed between the diffraction pattern of the milled powders after 10 and 15-20 h milling time.

441 High quality tmcp production and metallurgy of niobium bearing steels

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The technological and metallurgical advancements of value-added niobium (Nb) microalloyed thermo-mechanical controlled process (TMCP) plate steels continue to be developed for more demanding end user requirements. The market demand for reduced fuel consumption and CO₂ emissions in the automotive and construction sectors have further increased the demand for these new and advanced higher quality Nb-bearing steel grades. Often, the transition from laboratory melted and TMCP hot rolled heats to the production scale requires some continuous casting, thermal and mechanical metallurgy adjustments from the laboratory results in order to accomplish proper industrial continuous casting and hot rolling processes. These advanced high strength steels are microalloyed with Nb, Mo and/or other elements which affect the austenite-ferrite transformation. Niobium enables achievement of substantial grain refinement when the plate is rolled with the proper reduction and thermal schedule. The effects of these microalloying elements on the continuous cooling transformation behavior must be carefully controlled during the reheating and rolling process to successfully achieve the desired mechanical properties. TMCP applications have been successfully developed in numerous product sectors with thickness exceeding 120 mm. Since the very fine grained microstructure improves toughness and increases the yield strength, this TMCP process enables the required tensile properties with the growing trend to leaner chemical composition designs (less than 0.10%C) and excellent toughness properties. The consequence of leaner chemical compositions, especially lower carbon content and lower carbon equivalent, enhances mechanical properties, fabrication and weldability.

442 The optimal placement of sensors by minimizing the maximum probability of non-detection using genetic algorithm

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Optimally placing the sensors without compromising the performance is a challenge and its application is found in Structural Health Monitoring, Load Monitoring, Vibration Control, and other areas. Every sensor has predefined region within which the source of disturbance is detected if it is present. This paper examines the use of minimax concept on simple discretized plate using Genetic Algorithm to optimally place the sensors. This has been achieved by introducing maximum non detection probability in the objective function and the fitness of objective is minimized through genetic algorithm solver in MATLAB. The effectiveness of the present algorithm is then checked by comparing the solution with the solution obtained by implementing this concept on a continuous unit square plate using fminimax solver in MATLAB. The solution obtained in both the methods matched to that in the literatures. The

study shows that the algorithm developed can be effectively adopted in discretized structures to optimally place the sensors.

443 Surface engineering of metal oxide photoelectrodes for improved band alignment in solar water splitting cells

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Proper alignment of semiconductor energy levels with the water reduction and oxidation potentials is critical for achieving unassisted photoelectrochemical water splitting. Several earth-abundant transition metal oxides and mixed oxides (e.g. CoO, Cu₂O, Fe₂O₃) having a suitable band gap for solar water splitting exist, but energy level alignment is often sub-optimal. For example, the required straddling of the H₂ and O₂ evolution potentials may not be satisfied, or an excessive driving force for one reaction or the other may be present. We are exploring the possibility of tuning the semiconductor-solution energy level alignment by using ionic (e.g. Li⁺, F⁻) or polar molecular adsorbates (e.g. substituted benzoic acids) to modify the interfacial dipole layer. In this presentation I will report on our progress in applying this strategy to various metal oxide and mixed oxide photoelectrodes. This will include discussion of insights gained into interfacial energetics through in-situ spectroelectrochemical measurement of band energies and studies of interfacial charge transfer rates.

444 Effects of deformation induced structural variations on recrystallization of metals

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Although it is well recognized that the processes occurring during annealing of deformed metals are determined by the local environment in which they occur, much understanding, analyses and modelling are based on macroscale considerations.

In this presentation, recent achievements concerning the understanding of deformation induced structural variations on several length scales are summarized. It is now clear that deformation microstructures are hierarchical structures with dislocations and deformation induced boundaries subdividing the original grains. There are also variations from grain to grain due to the different orientations of the grains. Finally, processing may also introduce structural variations on the sample scale. The main objective of this talk is to address how such structural variations affect recovery and recrystallization. It will be shown:

1. how the deformed microstructure may coarsen due to triple junction motion
2. that nucleation typically happens at structural heterogeneities with high local stored energy. Focus will here be on orientation relationships between the nuclei and the parent deformation microstructures
3. how the recrystallization boundaries migrate through the deformed microstructures and it will be discussed how the deformation morphologies may affect the growth.

445 Tailored sever plastic deformation by a novel repetitive continuous extrusion forming (R-Conform process) in Al alloy

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A novel repetitive continuous extrusion forming, defined as R-Conform process, was performed on Al-Fe-Cu alloy and Al-Mg-Si alloy. By optimized die designing and wheel revolving speed controlling, various extruded temperatures could be tailored. The results showed that improved microstructure homogeneity and refined grains could be obtained under both high and low temperature conditions. However, these microstructural evolution processes were faster at high temperature. Particle refinement and/or re-dissolution were observed in both alloys at high temperature. Dynamic precipitation strengthening effect was implemented in Al-Mg-Si alloy at low temperature.

446 Al-5Cu alloy processed by equal-channel angular pressing

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Equal-channel angular pressing (ECAP) is an established method to produce ultra-fine-grained (UFG) materials with extraordinary mechanical properties. An ECAP-processed UFG Al-5Cu alloy was characterized by various electron-microscopy-based methods such as backscattered electron contrast and electron backscatter diffraction (EBSD). A skilful combination of all methods reveals the complete information about the microstructure which is needed to understand the results of the tensile tests. As a result, a bimodal grain structure was developed accompanied by micron-sized grains after 4 passes. In the present work, high strength (~501 MPa) and large uniform elongation (~5%) were achieved simultaneously in a binary Al-5 wt.% Cu alloy by using room-temperature equal channel angular pressing (ECAP) in route A. The high strength is due to a combination of strengthening by solute, the high density of dislocations and ultrafine grains. The high uniform elongation is due primarily to the enhanced work hardening resulted from the solute Cu content and the bimodal grain structure. Most importantly, the present work revealed that inhomogeneous deformation during ECAP can be utilized as a strategy to generate desirable bimodal grain structure, gaining both a high strength and high uniform ductility.

447 Metastable phase structure and evolution in the Al-Si-Mg-Hf Alloy

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The morphology, composition and structure of precipitates in an Al-Si-Mg-Hf alloy after heat treatment at 560°C for 20h were studied by means of Cs-corrected high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) and energy dispersive X-ray spectroscopy (EDS) with atomic resolution and traditional high-resolution transmission electron microscopy (HRTEM) and first-principle calculation method. Three different morphologies of precipitates, i.e., square-like, rectangle-like and nanobelt-like were identified to be metastable $(\text{Si}_{2-x}\text{Al}_x)\text{Hf}$ phase, which was formed with Al replacing some Si in orthorhombic Si_2Hf lattice yielding to a ternary ordered phase. First-principle calculations showed lower formation energies for Al atom replacing the Si6 and Si8 sites in the Si_8Hf_4 structure, which could be the preferential sites for Al incorporation. While some nanobelt-like precipitates have been identified as an equilibrium Si_2Hf phase.

Keywords: Aluminum alloys; Precipitates; HAADF-STEM; Si_2Hf

448 Analysis of Thin Strip Shape and Profile in cold rolling: A way to Improve Strip Profile and Mechanical Properties

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Market requirements for increasingly thinner gage strip such as the thickness less than 0.2mm with improved quality have driven the efforts to develop technology for predicting and controlling thinner strip profile. Strip shape control has been recognised as a challenging aspect during cold rolling of thin strip. Key rolling parameters such as work roll cross angle and work roll shifting have significant impact on microstructure, mechanical properties and strip shape and profile of the rolled strip. In this study, the influences of work roll cross angle and work roll shifting value on the strip profile and microstructure of low carbon steel have been investigated under dry condition at speed ratio of 1.3 using Hille 100 rolling mill. With an increase of work roll cross angle from 0° to 1°, a significant improvement of strip profile was observed, but no significant change of microstructure (grain size and shape) was found. Work roll shifting values from 0-8mm were also found to improve the strip profile, but not as significant as that of work roll cross angle. However, there is a slight change in grain size and shape. The significant drop in rolling force was similar in both cases. Furthermore, even when the reduction ratio increases, there is no change in microstructure of the rolled strip. Of all these parameters, the work roll cross angle has the most significant effect on strip profile, and the rolling force can be significantly reduced by either increasing the work roll cross angle or work roll shifting value.

449 Solidification of immiscible alloys under the effect of a direct current

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Continuous solidification experiments were carried out with immiscible alloys under the effect of a direct current. The experimental results demonstrate a direct current shows a significant effect on the migration of minority phase droplets (MPDs) in continuously solidified immiscible alloys. It can promote the formation of a well dispersed microstructure or a core/shell microstructure. A model describing the kinetics of the microstructure evolution in a continuously solidified immiscible alloy was developed. The microstructure formation in the alloys was calculated. The numerical results are in favorable agreement with the experimental ones. They demonstrate that a direct current may affect the microstructure development through changing the spatial motions of MPDs. The alloys show a well dispersed microstructure when they are solidified with such a direct current density that the direct current causing motion of the MPDs is almost equivalent to the radial component of the Marangoni migration velocity of the MPDs. The alloys show a core/shell microstructure when they are solidified with such a direct current density that the direct current causing motion of the MPDs dominates the migration of the MPDs along the radial direction of the sample. A wire or rod with well dispersed microstructure or a core/shell microstructure can be prepared by solidifying monotectic alloys under the effect of a direct current properly chosen.

450 Study on the porosity in Al-Zn-Mg-Cu high strength alloy DC ingot

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An Al-Zn-Mg-Cu high strength alloy ingot produced by Direct-Chill casting was used in this study. The distribution of porosity in the cross section of the DC ingot was investigated by the precision density method (Archimedes' principle), also X-ray microtomography technique was used to quantitatively analyze porosities in typical positions. The pattern in the cross section as well as in the thickness and width direction was obtained. The results show that: in the cross section of the ingot, porosity was increasing gradually from the surface to the center of the ingot; porosity shows an overall escalating trend from the surface to the center of the ingot both in thickness direction and in width direction; porosity was closely related to the cooling rate in the ingot; oxide inclusions have an effect on the formation of porosity to some extent.

451 The influence of molybdenum on precipitation in strip cast steels containing niobium

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Steels containing both Nb and Mo have been reported to exhibit superior strength as compared to those containing only Nb. This has been explained by the formation of harder microstructures, such as bainite and acicular ferrite, and denser and finer precipitates promoted by the addition of Mo. This research explores the effect of Mo on precipitate formation in steels processed with the recently developed direct strip casting process. Direct strip casting is a revolutionary casting technique that integrates casting and subsequent rolling together with rapid solidification and cooling rates, which not only simplifies the process, but also confers superior energy-saving as compared to conventional alloy thermomechanical processing. In this work, the role of Mo in precipitation is investigated under direct strip casting conditions. Small-angle neutron scattering (SANS) and transmission electron microscopy (TEM) are utilised to investigate the kinetics of precipitation. The structure and chemistry of the precipitates will be analysed at the atomic scale by the combination of atom probe tomography (APT) and high resolution transmission electron microscopy (HRTEM).

452 Study on strain distribution in high-temperature superconducting coils by using synchrotron X-ray diffraction

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It is known that it is a challenge to observe the X-ray diffraction peak through a few millimeter width of metal or ceramics. Strain measurement of the materials in coil of high-temperature superconductor such as Bi2223 and REBCO is much important for their application in high-field magnet, which coil of 4-5 mm width is commercially available. In this study, a white X-ray diffractometer at SPring-8 was chosen to measure the strain distribution in the coils. By appropriate measurement conditions in the 2 θ and beam width, a clear (400) peak of the Bi2223 phase was successfully obtained that is the first observation in coiled high-temperature superconducting wire using synchrotron X-ray diffraction. This method is considered to be useful to develop the future high-field magnet. The experiment method and the results will be presented in THERMEC'2016.

453 Local surface phase stability during cyclic oxidation process

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Surface properties are essential for many engineering materials design issues, such as fatigue and corrosion performances. Austenitic stainless steels used in high-temperature applications, as for instance components in biomass-fired power plants, needs sufficient corrosion resistance. At temperatures above 600 °C and in water vapor environment, Cr-vaporization will create Cr depletion, causing a local change in chemical composition. This local change in chemical composition leads to phase transformation in some austenitic stainless steels. This paper reports the surface properties regarding the local phase transformation during thermal cycling in water vapor environment. Three commercial austenitic stainless steels are investigated, AISI 304, AISI 316L and Sandvik Sanicro™ 28. The thermal cycling was performed at 650 °C in a 15 mol% water vapor environment. AISI 304 shows local surface phase transformation related to martensitic transformation due to locally changed chemical composition and increase in the martensitic transformation temperature (M_s). However, the other two austenitic stainless steels don't show this martensitic transformation. The phase transformation is discussed using thermodynamically simulations and microstructural investigations using electron backscatter diffraction (EBSD).

454 Ammonia for hydrogen storage

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Ammonia has been promoted as a viable candidate as an indirect hydrogen fuel vector, due to its high hydrogen content (17.8 wt%) [1]. Ammonia can be safely stored in very high gravimetric and volumetric density in solid state halide materials [2-3], for example, at 109 gL⁻¹ for Mg(NH₃)₆Cl₂. We are interested the storage, characterisation and utilisation of ammonia as an energy vector, whether it be for massive scale energy buffering or mobile applications. Here I will present some recent work on three aspects of our studies; the development of new catalyst systems for ammonia cracking [4], understanding the physical properties that determine the strength of ammonia binding within solids [6] and the development of new characterisation techniques for solid state ammonia stores [7].

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455 Stability, kinetics and prospects of high entropy alloys at elevated temperatures

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High Entropy Alloys have been the subject of an enormous research effort since their inception just over a decade ago and a vast array of different alloys have been reported upon. In the as-cast state, these multi-principle element materials are often found to exist as solid solution phases with relatively simple crystal structures. As a result, it was suggested that the configurational entropy of these phases was very large, sufficient to overcome enthalpic effects and stabilise the solid solutions against the formation of intermetallic phases. These alloys were also reported to exhibit several other key benefits, including sluggish diffusion kinetics, good mechanical properties and significant resistance to environmental attack. Such properties are highly desirable for high temperature service, making these materials of great interest, especially since the entropic term should become increasingly dominant at elevated temperatures. However, much of the fundamental science that underpins these alloys remains under debate. Here, we will present detailed characterisation of two exemplar high entropy alloys following prolonged exposure to elevated temperatures. We consider the stability of the solid solution phases in these alloys and investigate the kinetics of microstructural evolution. We will also discuss the prospects that these materials offer for high temperature applications and which systems show the most promise.

456 Influence of the processing variables on the microstructure evolution of a bainitic carbide-free steel

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New trends focused on achieving higher performance steels has led to a so-called 3rd Generation Advanced High Strength Steels (AHSS), in which the typical polygonal ferrite found in TRIP steels as a matrix phase is replaced by harder phases as Carbide-Free Bainite (CFB) and/or (tempered) martensite. Besides, large volume fractions of retained austenite (RA) with adequate stability are aimed for to improve the formability of the steels. Si containing steels are regarded as most suitable to retard cementite formation and consequently reach high volume fractions of RA. In this work, CFB annealing schedules were applied to dilatometer samples of Fe-0.22C-2.0Mn-1.3Si. The overaging temperature T_B was varied between 390°C and 540°C, and other processing variables investigated were the austenitizing temperature T_{aus} , and the bainitic holding time t_B . The annealed samples analyzed with LOM, FEG-SEM, EBSD and X-rays diffraction techniques show that markedly different complex microstructures made up of bainite, (tempered) martensite, ferrite and retained austenite are accomplished depending on the specific thermal cycle. These results are described in detail and discussed in relation to the dilatometry measurements.

457 Processing shape memory alloys and its composites by powder metallurgy

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Shape memory alloys (SMAs) have found multiple applications in several industrial sectors, due to their specific properties of shape memory and superelastic effects, which are linked to a thermo-elastic martensitic transformation. In the last years there has been a growing interest in developing C-Al-Ni SMA, because they exhibit some competitive advantages over the worldwide spread TiNi SMA. However the main drawback of these alloys is their brittleness in polycrystalline state, intrinsically associated to their high elastic anisotropy producing high-stress concentrations at the grain boundaries and fracture. The processing route of Cu-based SMA by powder metallurgy is being developed to solve the above problem. In the present talk we will do an overview of the progress obtained by this powder metallurgy methodology, using gas atomization of the SMA, compaction by HIP and hot rolling, with an optional step including mechanically alloying the SMA powders before compaction. The microstructure of the alloys and the functional properties of the SMA obtained by such powder processing methodology will be reviewed. Alternatively, atomized powders of SMA can be used to produce Composites. This is a promising new route for manufacturing metal matrix composites taking advantage of the specific properties of the SMA. In particular, we have developed In-Sn matrix composites with spherical particles of Cu-Al-Ni SMA, which exhibit ultra-high mechanical damping behaviour. The microstructure and properties of these new functional composites based on powders of SMA will be also reviewed in the second part of the lecture.

458 Evaluation of bending response of heat-treatable aluminum alloys using crystal plasticity model

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Aluminum alloys, due to their low density, strength, and formability, have become standard for hang-on parts in many vehicle models. Sheets or profiles are usually formed into final parts by cold deformation. Thus, bendability becomes an important attribute of aluminum alloys in their applications.

Constituent particles, grain morphologies and texture are found to be important factors in shear band formation or strain localization during bending deformation. In this work, extensive analyses on the effect of each factor as well as combination of the factors on mechanical properties and bending stability have been performed using crystal plasticity finite element simulations. Local stress and strain state as well as orientation changes are examined with different particle size and spatial distribution, grain morphologies and initial texture. Variations in particle size and spatial distribution, grain morphology are synthetically made with DREAM3D software. Common textures such as random, rolled, or cube textures are used to evaluate their effect when combined with different particle and grain size distribution. Experimental measures are made and compared with initial microstructure used for simulation to ensure that the synthetic microstructures used for the simulations are realistic.

459 The effect of sample preparation on the microstructure of duplex stainless steels

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Sample preparation in metastable austenitic steels can have significant effect on the microstructure, i.e. by the formation of strain-induced martensite. As a result, these steels often have a complex microstructure with phases of relatively similar crystal structures such as ferrite and martensite, making it very difficult to analyse. However, the quantitative analysis of such microstructure and the effect of the sample preparation are very important for the further study of the steel. In this research, the effect of sample preparation in metastable duplex stainless steel was studied by using three different sample preparation methods. In addition to conventional mechanical etching with colloidal silica, electro polishing and FIB (focused ion beam) was used to create optimal sample surfaces to be further analysed with EBSD. FESEM/Optical micrographs were obtained from each sample before and after sample preparation, and the microstructure was analysed with EBSD, Ferritoscope and XRD. It was observed that the amount of martensite was dependant on the sample preparation, therefore the most martensite was formed in mechanical polishing and least in FIB ion milling. The amount of martensite was distinguished from the ferrite using band slope and band contrast imaging with various other techniques, like grain averaging, on EBSD software.

460 Solidification cracking susceptibility for dissimilar weld metal of austenitic metals

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Austenitic metals such as stainless steels and Ni-based alloys have high corrosion resistance, high heat resistance and so on. However, these metals are high susceptible to hot cracking (especially solidification cracking) during welding. The susceptibility becomes higher in the case of dissimilar welding using the austenitic metals. In addition, the dissimilar welds might increase for fabrication of multi material components. Thus, it is necessary to prevent from the solidification cracking occurrence by understanding the cracking susceptibility during fully austenitic solidification. The aim of this work is to investigate the influence of dilution ratio in dissimilar weld metal of austenitic metals on the solidification cracking susceptibility during welding. In order to change the chemical composition of specimen systematically within austenite region in Schaeffler diagram, the specimens were fabricated using 304L stainless steel as a base material and several filler wires of Ni-based alloy and stainless steel. Trans-Varestraint test was carried out to evaluate the solidification cracking susceptibility quantitatively. Distribution of the solidification cracking susceptibility in Schaeffler diagram could be evaluated quantitatively by measuring brittle temperature range and crack length. The susceptibility became the highest at Cr equivalent of 20% and Ni equivalent of 30 % in the diagram and the decreased toward a marginal area in the austenitic region. However, the susceptibility varied with the type and content of alloying element when the equivalents of Ni and Cr were nearly the same. Especially, containing Nb deteriorated the susceptibility significantly.

461 Effect of red scale on the bendability of ultra-high-strength steel

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The effect of red scale on the bendability of a thermomechanically rolled and direct quenched pilot-scale strip steel has been studied by comparing the bending behaviour of adjacent areas with and without red scale. The yield strength of the studied 8 mm thick strip was 960 MPa. The local microstructure and texture below the different scale surfaces were characterized using FESEM and FESEM-EBSD, chemical compositions were determined using GDOES, surface roughness and microhardness profiles were measured and bendability was determined using three-point brake press bending. Red scale was found to significantly affect bendability especially when the bend axis is transverse to the rolling direction. The minimum punch radius for defect-free bends in the absence of red scale was 12 mm (1.5 x thickness) while under red scale it was 30 mm (3.75 x thickness). Beneath the red scale the microstructure 50 to 400 micron below the surface was clearly different to that in the absence of red scale. Without the red scale the microstructure was mainly granular bainite with small fraction of upper bainite and polygonal ferrite. Below the red scale the microstructure was a mixture of upper bainite and granular bainite. As a result of the microstructural differences, the hardness in subsurface area changed substantially from 360 HV in the absence of red scale to 410 HV with red scale. Possible explanations for the observed effects of red scale on subsurface microstructure, and microstructure on bendability, are discussed in the paper.

462 Development of an electrically-debondable smart dental cement

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Dental devices are bonded to tooth and sometimes have to be removed later; for example, removal of crown prosthesis for root canal treatment, dental braces after orthodontic treatment is completed, and temporary sealing in resuming treatment. They are usually removed mechanically, and therefore, the processes have risks of tooth damage. Thus, the authors suggest a “non-mechanically” debondable bonding system. The system should normally indicate a strong bonding property for a long time and if necessary, the bonding strength can be reduced drastically for easy removal. The authors have developed a new dental cement containing a debonding trigger and succeeded in producing an electrically-debondable dental cement. In this study, a new dental cement was prepared by mixing a commercial dental cement and an ionic liquid (IL) as a debonding trigger. The shear bonding strength (SBS) of the IL-containing cement and the IL-free cement (control) was evaluated. SBS of the IL-containing cement was slightly higher than that of control. Thus, addition of IL did not reduce SBS. After activation of the debonding trigger (applying of direct current, 19V, 30s), SBS of the IL-containing cement was significantly lower than that before activation. Thus, activation of the debonding trigger reduced SBS significantly. The IL-containing cement in this study could perform both strong bonding and easy debonding simultaneously. Hence, the IL-containing cement is “Smart” and shows the feasibility of an “Electrically-debondable smart dental

cement”.

463 Nanoparticle addition into molten steel

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This investigation focuses on the development of a novel steel grade with enhanced combination of strength and toughness through microstructural control by means of nanoparticle safe addition into molten microalloyed steel. The main objective of the investigation was to set the parameters for the future implementation of the addition method during the industrial continuous casting, and the subsequent replacement of the steel grades currently used in automotive cold forged components that require more energy consuming processes, with these new steel grades. Apart from the steel composition, nanoparticles addition, and the application of specifically designed processes, acicular ferrite nucleation is one of the mechanisms here studied for property enhancement through grain refinement, which contributes to an overall increase of strength and toughness.

During the whole research several obstacles had to be overcome, such as, the dispersion and posterior localization of nanoparticles in the steel due to the small amount added, and of course the identification of the benefits obtained due to the addition, having used for that purpose, several and very different characterization techniques.

464 Effects of additional elements on hydrogen storage properties for vanadium alloys

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Vanadium or V-based solid-solution alloys with a bcc structure are known to show fully hydrogen content 3.8 mass% (H/M~2), and they form beta phase as a monohydride and gamma phase as a dihydride. Reversible hydrogen capacity is equivalent to the amount of stored hydrogen for transformation between beta and gamma phases.

The author group has reported that increasing Cr content in 20V-Ti-Cr alloys turned out to low hydrogen concentration in unstabilizing the beta hydride phases. Moreover, higher hydrogen desorption capacity was considered to be achieved by increasing Cr content. Therefore, the purpose of the present study is to investigate the effects of additional elements on hydrogen storage properties for vanadium alloys. With increasing Cr content in V-xCr binary alloys (x=0-16), fully hydrogen content of the alloys slightly decreased until less than 9 at.%Cr. A clear distinction of the PC isotherm curves between the 15 at.%Cr alloy and the other alloys is observed. V alloys with an excessive Cr addition would come not to form gamma phase (dihydride). This led the drastic decrement of the hydrogen content in the alloys. Meanwhile, the Cr addition in V alloys was effective to low hydrogen concentration in unstabilizing the beta hydride phases. In addition, it was found that the addition of X elements in V-Cr alloys (X=Al, Mo, Ti, W) was effective to expand the gamma-phase forming range of Cr amounts. As the results, high reversible hydrogen-capacity, 2.68mass%H, was obtained in a V-18Cr-2Ti-0.5Al alloy.

465 Evaluation of rolling contact fatigue by using an X-ray diffraction ring analyzer

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In this report, an analysis of X-ray diffraction ring (Debye-Scherrer ring) for the evaluation of the rolling contact fatigue (RCF) progression is introduced. The progression of RCF is evaluated based on tri-axial stress state and the degree of martensite grain orientation which are characterized by two parameters: σ_{eq} and S/S_0 , respectively. The σ_{eq} is calculated by six components of tri-axial stresses, and the S/S_0 is defined as the variation of peak intensities for the central angle of the Debye-Scherrer ring. From the behavior of the σ_{eq} and S/S_0 during RCF, it is found that the progression of RCF can be divided into three stages as follows. First, σ_{eq} approaches the yield stress of SUJ2 in the early loading stage; about 10^3 cycles. The S/S_0 does not change in this stage. After drastically increasing the σ_{eq} up to 10^3 cycles, the S/S_0 increases. This shows that the martensite grain orientation on the RCF surface begins to be formed after the 10^3 cycles. Finally, σ_{eq} gradually decreased while increasing peeling on the raceway although the orientation of texture continues to progress. These are new findings obtained by applying the Debye ring analyzer to the evaluation of RCF. The evaluation of RCF by Debye ring is a promising method, not only to investigate mechanism of RCF, but also to allow for quantitative estimation of the progression of RCF.

466 Effect of electroless Ni-P plating on mechanical properties of Al-4%Ge alloy

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In this study, the effect of electroless Ni-P plating on the mechanical properties of Al-4%Ge alloy was investigated. As the results, the following points were clarified. (i) Tensile strength of the specimen subjected to the Ni-P plating after aging treatment or furnace cooling treatment was improved by about 10% in comparison to one of the non-processed specimens. (ii) Breaking elongation of the specimen subjected to the Ni-P plating after aging treatment showed no significant changes in comparison to one of the non-processed specimens. On the other hand, breaking elongation of the specimen subjected to Ni-P plating after a furnace cooling treatment was reduced to 70% in comparison to one of the non-processed specimens. (iii) Fatigue strength of the specimen subjected to the Ni-P plating after a furnace cooling treatment was overall reduced rather than one of non-processed specimens. (iv) Fatigue strength of the specimen subjected to the Ni-P plating after aging treatment was overall reduced, except for the low-stress region, rather than one of the non-processed specimens. (v) In the specimen subjected to Ni-P plating after a furnace cooling treatment or aging treatment, clear hydrogen desorption was recognized. On the other hand, there was only hydrogen desorption from a few of the non-processed specimens. Especially, it is considered that the poor fatigue strength and ductility of the plating materials are mainly due to the interaction between the surface precipitates and hydrogen gas.

467 Infrared solar cells using plasma processed semiconducting single-walled carbon nanotubes thin films

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In order to greatly increase the solar cell efficiency, a full use of the solar spectrum is one of the crucial issues, and therefore, development of multi-junction thin-film solar cells is strongly desired. Especially, for fabrication of an infrared solar cell as the bottom cell, single-walled carbon nanotubes (SWNTs) are attracting much interest for photovoltaic energy conversion because of their broad absorption bands including the infrared range (0.2 ~ 1.3 eV). To realize the solar cell with SWNTs [1,2], it is necessary to establish a method for carrier type-, density-, and position-controllable doping into SWNTs. In this study, we have fabricated the p-n junction embedded SWNTs solar cell using SWNTs thin films. We have demonstrated the controllable and stable n-type carrier doping into semiconducting SWNTs thin films by position selective cesium (Cs) encapsulation into SWNTs with a plasma ion irradiation method [3]. Optoelectrical features were also measured and rectifying features can be observed for the p-n junction embedded SWNTs. Furthermore, the open circuit voltage and the short circuit current can be also clearly obtained with light illumination. This is the first result showing the clear solar cell performance with p-n junction embedded SWNTs thin films. [1] R. Hatakeyama, Y. F. Li, T. Y. Kato, and T. Kaneko, Appl. Phys. Lett. 97 (2010) 013104.[2] Y. F. Li, S. Kodama, T. Kaneko, and R. Hatakeyama, Appl. Phys. Lett. 101 (2012) 083901.[3] T. Kato, E.C. Neyts, Y. Abiko, T. Akama, R. Hatakeyama, and T. Kaneko, J. Phys. Chem. C 119 (2015) 11903.

468 Carbon enrichment in austenite during bainite transformation in Fe-2Mn-1.5Si-C alloys

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In TRIP steel, it is essential to understand behavior of carbon enrichment into γ during bainite transformation to control retained austenite (γ) distribution. Recently, it becomes possible to measure carbon enrichment in γ directly by three-dimensional atom probe and field emission electron probe microanalyzer. Therefore, the objective of the present study is to investigate the carbon enrichment into γ during isothermal bainite transformation in Fe-2Mn-1.5Si-(0.2, 0.4)C (mass%) alloy over the temperature range 500 - 400°C using those methods. It was found that the incomplete transformation (ICT) of bainite appears in transformation at 400 °C in both alloys and volume fraction of BF in ICT decreases with increasing carbon content. The carbon contents in ICT in both alloys exceed T_0 composition. Interestingly, it was revealed that the higher the bulk carbon content, higher the carbon content in untransformed γ . The difference in carbon content in untransformed γ implies that volume fraction of BF influences energy barrier for growth of BF in diffusional theory or strain energy in displacive theory.

469 Constitutive modelling of high Mn TWIP steels: Composition and temperature dependencies of tensile behaviour

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The tensile behaviour of high Mn TWIP steels is studied with a dislocation density based constitutive model. Quasi-static tensile tests were carried out for Fe-18Mn-0.5C and Fe-24Mn-0.7C (wt%) at 23°C and 110°C. Model parameters are critically reassessed based on previous studies. The most important parameters are (i) the shear modulus which increases with temperature near to the Neel temperature due to magnetic ordering, and (ii) the dynamic recovery coefficient, which is related to the easiness of cross slip. By considering cross slip as a thermally activated process, the temperature dependency of the dynamic recovery coefficient is estimated, and dislocation density evolution is predicted accordingly. Although the temperature dependency of the dislocation density evolution is similar in the both alloys, that of the flow stress is different since their shear moduli vary abnormally depending on their Neel temperature.

470 Applicability of interatomic potentials for Fe-C systems to simulate martensitic transformations with molecular dynamics

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Using molecular dynamics (MD) simulations we can study mechanisms controlling phase transformations from austenite to martensite in pure Fe and Fe-C systems. The thorough understanding of these phase transformations is important to further improve the properties of advanced high strength steels. In the first part of this study, we will estimate the accuracy of the of two interatomic potentials available in the literature for MD study of Fe-C systems (Johnson et al. 1964, Acta Metall., P. 1215-1224; and Lau et al. 2007, PRL, P 215501). In particular, we will evaluate the resulting stability of the phases with respect to temperature and carbon concentration. These potentials are chosen for the present study based on the previous literature studies which have shown that they predict several properties of austenite and martensite phases well, such as lattice constants and temperature stability. In the second part, we will use these potentials to study the effect of carbon concentration and temperature on the martensitic transformations in single crystal Fe-C systems. The simulations will capture the main features and mechanisms of nucleation, growth, and pathways of martensitic transitions. This study will help answer the questions such as how does carbon affect the nucleation and growth of martensite transformations at atomic scales which can be difficult to analyze experimentally.

471 Fiber laser beam welding of Ti6242 - effect of parameter variation on microstructural and mechanical properties

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The object of the present work is the fiber laser beam welding of Ti-6Al-2Sn-4Zr-2Mo sheet (denoted as Ti6242) used for high temperature applications. An experimental study based on the design of experiment was performed to identify a process window, where butt joints with fully penetrated welds and good shapes can be achieved. The effect of laser beam power, focus position and advance speed on the geometry and microstructure of the butt joints was investigated. The identified parameters used for detailed microstructural and mechanical studies were the laser beam power of 5 kW, the focus position of 0.0 mm and the advance speed of 6.2 m/min. The Ti6242 base material is characterized by a globular ($\alpha + \beta$) microstructure. The heat input during laser beam welding has led to the formation of martensitic α' fusion zone. The heat affected zone consisted of globular grains and acicular crystallite. These local transformations were connected with a change of micro-texture, average grain sizes and β content. Furthermore, the microhardness increased from 330HV0.3 to 450HV0.3 due to the martensitic transformation. The mechanical behaviour of laser beam welded Ti6242 butt joint under tensile load was determined by the properties of the Ti6242 base material. The local hardness increase delivered a shielding effect protecting the Ti6242 butt joint against mechanical damage.

472 Preparation of poly(lactic acid)-based composites containing calcium carbonate with core-shell-type fibrous structure

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We have reported cotton wool-like composites consisting of siloxane-doped vaterite and poly(L-lactic acid), SiVPCs, for filling various irregular-shaped bone-voids. The composites can supply calcium and silicate ions for enhancing bone formation. They can be also used by being mixed with bone marrow aspiration (BMA) or autologous blood, as necessary. Since they are mechanically brittle, they may be carefully handled during the mixing. In the present work, for improving their mechanical toughness, the composite fiber was coated with a soft, thin layer consisting of poly(lactic-co-glycolic acid) (PLGA) by coaxial-electrospinning. The resulting cotton wool-like materials were comprised of the "core-shell"-type fibers with 5~20 μm of diameters, consisting of SiVPC coated with a PLGA layer of 1~2 μm -thickness. The fibers were mechanically tough: even when a uniaxial load of 1.5 kPa was applied to the cotton wool-like material, it was compressed without the fracture of the fibers. After removing the load, it showed the recovery of ~60 %: the toughness of the composites have been drastically improved by the thin PLGA coating. In Tris buffer solution, calcium and silicate ions were released slowly from the "core-shell"-type fibers after 1 day. Since the cotton wool-like "SiVPC-core/PLGA-shell"-type materials have highly-porous structure for cells migration and ions-releasing ability for enhancing cellular activities, it is expected to have an excellent performance in use as bone-

void fillers.

473 Manufacturing of aluminum metal matrix cast composites with carbon based additives for thermal management applications

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This work focuses on the production of new high conductive carbon based MMC or co-cast components obtained by casting processes. These novel thermally conductive structures are designed to face modern heat management challenges in critical fields such as power micro-electronics, e-mobility and (renewable) energy generation as well as highest performance combustion engines. The sought parts will be assembled by different heat conductive aluminum-carbon composites and for this reason different heat conductive MMCs have been studied. The three main pillars are the aluminum matrix, medium heat conductive with isotropic thermal properties and highly conductive and un-isotropic phase. The combination of these MMCs into once cast Aluminum part will allow the part to meet applicative needs for heat management challenges. Some preliminary empirical and numerical results are presented in this article. The thermal behaviour of the obtained materials has been studied by means of theoretical (EMA) and numerical (FEM) approaches in order to determine the effective thermal conductivity in the different directions of heat dissipation. Different Aluminum-Carbon MMCs are investigated for their thermal and properties, namely aluminum-graphite strips, and aluminum with particles additives. The effects of thermal resistance at the interfaces between matrix and inserts have been considered in order to numerically evaluate influence of casting process parameters which determines the quality of interfaces between the different materials of composites. The numerical results have been validated by direct thermal conductivity measurements on samples of the obtained materials by means of a thermofluximeter instrument (NanoFlash Light Flash System).

474 The microstructure change of Sb added 60/40 Cu-Zn alloy by annealing

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Hardness variation in 60/40Cu-Zn alloy is occurred by phase transformation from β_1 single phase to α phase by annealing process. The crystal structure of α phase is 9R structure, changes to fcc structure by annealing. The effects of phase decomposition in 60/40 Cu-Zn alloy by additions of transition elements (Sn, Zr and Ge) have been reported with annealing treatment. Previous studies have been reported that reduce the stacking fault energy of Sn, Zr and Ge added Cu alloy. Since Sb has also been reported that decreasing the stacking fault energy of Cu, the hardness change due to annealing in Sb added alloys to determine the number density change of per unit area of the α -phase, and compared with each alloy. Hardness measurement was conducted using Vickers microhardness tester. Number density of the α -phase is counted using scanning electron microscopy (SEM). Also, microstructure observation of α -phase and

β -phase were conducted using transmission electron microscopy (TEM). When Sb added 60/40 Cu-Zn alloy is compared to other alloys, maximum hardness was high. When measuring the number density per unit area of the α phase by SEM observation, Sb added alloys were the most highest number density.

475 Compression deformation of single crystals of the equiatomic CrMnFeCoNi high-entropy alloy

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Multi-component alloys, consisting of five or more elements, may exist as a stable single-phase solid-solution crystallizing in simple crystal structures such as the BCC and FCC structures due to their high mixing entropy. There alloys are recently called high-entropy alloys (HEAs) and are known to exhibit excellent mechanical properties. Among various HEAs, the equiatomic CrMnFeCoNi alloy with the FCC structure has been most intensively investigated and shows considerably high fracture toughness at low temperatures. However, since the properties of the HEA have been obtained only for polycrystals, its deformation mechanisms as well as the value of CRSS have yet to be clarified. Moreover, it is impossible to evaluate the extent of the solid solution strengthening in the HEA unless the bulk CRSS is known. In the present study, we have performed compression tests of single-crystal micropillars with different sizes at room temperature to elucidate the bulk CRSS value by extrapolating the power law scaling of CRSS against micropillar size. Since we have succeeded in growing large single crystals of the CrMnFeCoNi HEA during the micropillar experiments, we have also conducted compression tests of bulk single crystals in a wide temperature range from 77 to 1073 K. The CRSS value estimated by the micropillar compression tests agrees well with that of the bulk single crystals obtained at room temperature. Observations of the deformation microstructures indicate that the HEA exhibits mechanical characteristics similar to binary dilute FCC alloys.

476 Prestrain memory on subsequent cyclic behavior of FCC metallic materials presenting different dislocation slip character

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The forming processes of metallic industrial parts often involve strong plastic strains which modify the subsequent fatigue properties compared to cyclic behaviour of the equivalent fully annealed material. However, the mechanisms responsible for this memory effect are still unclear. For FCC metallic materials, several researchers investigated the influence of dislocations slip character on the memory effect. Purely planar slip materials were reported as strongly sensible to memory effect [1,2] whereas wavy slip materials have been presented as weakly sensitive to prestrain history [1,3] or fully sensitive [4].

The objective of this work is to investigate the influence of tensile and cyclic prehardening steps on the fatigue behavior for three materials with different slip character: NiCr alloy (planar slip), pure copper (easy cross-slip) and 316L (planar and cross-slip). First, the CSSCs of prestrained

specimens and virgin ones were compared showing that the three materials appear to be sensitive to the prestrain if a plastic prestrain threshold is overcome. Second, fatigue tests were performed for different strain amplitudes for both virgin and prestrained samples. For a given plastic strain amplitude in fatigue, larger stress amplitudes were observed for prestrained samples compared to virgin ones. These results are discussed in terms of backstress and effective stress evolution, dislocation structures and sample roughness. References: [1] K. Schoeler et. al. Int. J. of Fatigue 23 (2001), 767-775. [2] H.-J. Christet. al. Mat. Sci. Eng. A201 (1995), 1-12. [3] C.E. Feltner and C. Laird, Acta Met., 15 (1967), 1612-1653. [4] L. Kunz et. al., Mat. Sci. Eng., (2001), 1-6

477 In situ X-ray diffraction studies on rapidly solidified alloys under additive manufacturing conditions

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The emerging beam-based additive manufacturing (AM) technologies put new requirements on material properties. Alloys developed for conventional processing are suddenly subjected to high cooling rates that are orders of magnitude higher than the processes the alloys were designed for. Consequently, materials scientists need novel experimental tools to study the alloy behavior under controlled conditions similar to beam-based AM. The fast changes in temperature in small volumes require techniques with high temporal and spatial resolution. Synchrotrons can provide the intense radiation that allows such measurements. In this work, an in situ method was developed to study the behavior of alloys during rapid heating and cooling combining laser-based heating with synchrotron micro X-ray diffraction (microXRD) and high-speed imaging. Small metallic specimens were melted using a laser-based heating system consisting of two diode lasers and subsequently rapidly solidified using fast heat extraction into a Cu support structure. High-speed camera imaging provides location-specific information on temperature and solidification front positions. Simultaneously, microXRD was performed in transmission geometry at the microXAS beamline at the Swiss Light Source (Paul Scherrer Institut, Switzerland). Time-resolved peak evolution and temperature-corrected microXRD data provide clear observations of solidification and solid state phase transformations under non-equilibrium conditions similar to beam-based AM for alloy screening and design. The presented approach is applicable to many materials and provides a tool for materials scientists working in the area of AM to achieve deeper insights into alloy behavior and non-equilibrium conditions.

478 Mechano-chemical synthesis of refractory alloys nanometric powders

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The synthesis of Nb-W, Nb-Mo, Mo-W and Nb-Mo-W stoichiometric refractory alloys has been performed by inducing the reduction of their respective oxides (Nb_2O_5 , WO_3 , MoO_3)

by magnesium, in excess, the reaction being triggered using high energy planetary ball milling. As these reduction reactions (so-called thermites or more specifically magnesio-thermitic reactions) are extremely exothermic, with a final temperature that may exceed 3500K, adjusting the product final temperature is necessary to avoid extensive melting. This has been done by adding an inert component, namely sodium chloride, to the mixture. Using a ball-to-powder ratio of 15:1, a mill velocity of 500 RPM and tempered steel milling media, the reaction starts within 3 to 12 minutes, depending on the composition and is characterized by a brutal temperature and pressure increase inside the milling jar. Products were then leached using 2M hydrochloric acid to remove NaCl and MgO, filtered, rinsed and dried before full characterization. SEM observation revealed a final microstructure of agglomerated particles with a mean diameter within the 80-150 nm range. BET specific surface analysis, X-ray diffraction analysis have also been performed; we will show how it is possible, through an adjustment of our synthesis parameters, to obtain a final product with a single stoichiometric composition, with a near perfect distribution of the 2 or 3 atom types within the alloys, at the nanometric scale.

479 Microstructure evolution induced by sliding-based surface mechanical treatments – application to pure copper

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The surface treatment notion is usually connected to the modification of surface properties by various actions of physical, chemical, thermal or metallurgical origin (quenching, nitriding, ...). However mechanical loadings alone may also result in modifications such as the creation of compressive residual stress fields or the sub-surface grain refinement without any thermal or chemical phenomena. It is often named as SMT (surface mechanical treatments). We distinguish treatments based on normal or quasi-normal contacts (like indentation) such as shot peening or hammering, and treatments based on tangential contacts (like scratching) such as burnishing, polishing and so on. In this paper, we address the issue of microstructural evolution induced by SMT processes based on sliding (friction). A tribometer designed for simulating contact pressures and cutting speed occurring during machining has been used to characterize the effect of sliding velocity on microstructural evolution induced in Copper. Numerical simulations are used to extract local variables around the interface pin/ copper bar, such as equivalent strain or temperature. These latter evolve sharply with sliding velocity and permit to justify that recrystallization and grain refinement happen only for the highest sliding speed tested. Thanks to these promising results, a new process based on repeated sliding friction is proposed. This one makes possible to create near-surface layer of Ultra-Fine Grains over a depth up to 100 microns in a very short time compared to usual impact-based treatments.

480 Modeling of the hot rolling: towards the industrial applicability

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The modeling of the hot rolling has been an exciting topic in the physical metallurgy over the past 3 decades. An empirical model of microstructural evolution during the hot rolling can very well predict the microstructure of closely related compositions of steels. This approach relies upon the time consuming experimental modeling in the laboratory scale. However, the application of the developed models onto prediction of the steel microstructure beyond the characterized chemistry is not really certain and can be used only for the first estimation. From this reason, the modeling moves towards the physically-based coupling of many interacting aspects: phase transformation, dissolution and precipitation of particles, grain growth, hardening, dynamic and static softening phenomena. The physical-based approach is very powerful, but it is still very intensive regarding the calculation. The semi-empirical approach to the modeling of the microstructural evolution during the hot rolling of austenite including grain growth, hardening and softening will be discussed in the frame of a general application concerning the activation energy of iron self-diffusion in austenite to the particular phenomena. The activation energy is depending upon the chemical composition of the steel and hence it influences the flow properties during hot rolling. Additionally, the precipitation sequences, the size distribution, Oswald ripening and interaction with softening are also included in the model of the microstructural evolution. The current model has been validated on the experimental results of more than 20 different steels in laboratory scale. Exemplary results are discussed in connection with the production parameters.

481 Modeling and experimental results in core-shell ferroelectric ceramics

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Modeling and experimental results in core-shell ferroelectric ceramics

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Structural and physical consequences of downsizing to form nanopowders or nanoceramics are very important both from applicative and fundamental point of view. Indeed many interesting effects have already been reported such as diminishing of permittivity and increase of diffusivity, cancelation of dielectric relaxation in relaxors, lowering of ground states, increase and rotation of polarization etc. In order to explain these experimental results, one must take into account not only size and microstrains effects but also existence of core-shell structure which can be modeled. Modeling also shows that there is optimal nanostructurations to maximize energy storage and electrocaloric effect. Calculations also show

the possibility to form new phases in which vortices of polarization compete with homogeneous polarization or in which homogeneous hypertoroidization of nanotori coexists with axial homogeneous toroidization. We will give a quick survey of these effects from our recent results.

482 Local deformation and fracture investigated using in situ electron microscopy

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Size effects regarding materials strength and deformation mechanisms become increasingly dominant once either the material microstructure or the sample size itself are reduced into the micrometer or nanometer regime. Not only the material strength can be subject to change, also changes in deformation behavior can occur. Quantitative miniaturized experiments performed in situ in the scanning or transmission electron microscope are a very versatile approach to not only measure material strength, but also directly observe the underlying deformation and strengthening mechanisms at the same time. These insights enable a better mechanistic description of the elemental deformation mechanisms in small dimensions. Within this presentation, we will present selected examples to highlight the benefits of such an approach. In detail we will address the cyclic deformation behavior of defect free nanowires, and the competing size and rate dependent deformation modes in hexagonal materials at small scales. Moreover, special emphasis is placed on very recent experimental advances regarding miniaturized quantitative fracture experiments in small dimensions, thus enabling insights into the dislocation nucleation and multiplication processes at crack tips.

483 The numerical simulation of precipitates dissolution interacting with grain boundary

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In the slab reheating process, mainly two phenomenon occur. First one is dissolution of precipitates, and second one is austenite grain growth. The phenomenon of grain boundary (GB) movement blocked by precipitates is well known as a pinning effect. However, the effect of GB on the precipitate dissolution is not clear. So, we have been researching the precipitate dissolution with taking the GB movement into consideration by phase-field simulation.

Numerical simulation was carried out in a following way. Many precipitates of TiC which are circular and the GB having curvature are defined in the Fe-C-Mn austenite matrix. Heat treatment is started at 1073K, and when temperature is reached at 1523K, it is soaked. As a result, following results have been obtained. 1) Precipitates which are NOT interacting with GB are dissolved retaining the initial shape. On the other hand, precipitates which are interacting with GB (= precipitates participating pinning effect) are dissolved with changing their shape from the initial shape to spindle shape like a rugby ball. This causes interface tension to be balanced at the triplet junction of GB/precipitate/matrix. Therefore, precipitates/matrix interface curvature changes into the spindle shape. 2) The interface energy is proportional to the interface curvature. Hence, driving force for the dissolution of precipitate is decreased as

the precipitates/matrix interface changes into the spindle shape which has small interface curvature. As a result, precipitates interacting with GB take much longer time for dissolving into the matrix perfectly compared to precipitates which are NOT interacting with GB.

484 Preparation of self-setting paste composed of hydroxyapatite/collagen bone-like nanocomposite

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Hydroxyapatite/collagen bone-like nanocomposite (HAp/Col) porous body is widely used in Japan as a bioresorbable bone-void filler due to its high biological performance that incorporated into bone remodeling process as well as viscoelastic property. In this study, self-setting paste was prepared using 3-(glycidoxypopyl) methyl-diethoxysilane (GPTMS) solution as a liquid phase. The HAp/Col powder was prepared by the simultaneous titration method; briefly, calcium hydroxide aqueous suspension and orthophosphoric acid aqueous solution with porcine dermal atelocollagen were simultaneously titrated into a reaction vessel with maintenance of pH at 9 and water bath temperature at 313 K. The HAp/Col composite fiber obtained was compacted by squeezing water with a specially designed mold and freeze-dried. The compact was crushed into a powder at 100 micrometer or less in size, and collagen in the HAp/Col was dehydrothermally crosslinked at 413 K for 12 h. Liquid phase was prepared by mixing of medical grade distilled water and GPTMS at 0.1 to 25 % in mass for 1 h. Powder and liquid (P/L) ratio was 0.20 to 2.00 g/cm³. Physical and biological properties of the paste obtained were examined according to JIS T 0330-4:2012, cell culture and animal tests. Viscosity and compressive strength of the paste depended dominantly on liquid amount. Decay ratio was minimized at P/L ratio of 1.0. Slight decreasing of initial proliferation of cells was observed by addition of the as-mixed paste; however, no further effect was observed afterwards. These results suggested that the HAp/Col-GPTMS paste is a good candidate of injectable bone-void filler.

485 Direct growth of pure SnO nano-wires and nano-platelets on CVD graphene/Au thin film layer by thermal evaporation

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The tin oxide (SnO) nano-materials have been studied as a functional material for gas sensors, and Li-ion battery. The synthetic method of nano-structured materials can be generally classified into several categories: vapor-phase growth, solution-phase growth, sol-gel, and template-based methods. Among these methods, thermal evaporation is one of commonly employed synthesis method of nano-structured materials. The tin oxide has various morphologies which are nano-belts, nano-slabs, nano-branches, and nano-disks depending on growth conditions. In this study, pure SnO nano-wires and nano-platelets are grown using a thermal evaporation process in a horizontal two-zone furnace system. The source material (SnO₂ powder, Sigma-Aldrich, purity: 99.9%) are vaporized at 1090°C in an alumina crucible

which is located at center of the first heater. The pure SnO nano-wires and nano-platelets are grown on CVD graphene/Au thin film layer at 424°C. The single layered CVD graphene was grown using a Cu foil by CVD and transferred on Au(50 nm)/SiO₂ (300nm)/Si substrate. The carrier gas was Ar (purity: 99.999%), and flow rate was 1000 SCCM. The quality of synthesized graphene and grown SnO nano-platelets are characterized by Raman spectroscopy. The morphology and crystalline property of the pure SnO nano-wires and nano-platelets are investigated by SEM and HR-TEM, respectively.

486 Towards the development of Mg alloys formable at room temperature

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The development of wrought magnesium (Mg) alloys, particularly in the sheet form, is essential to support the growing need of the automotive industry for lightweight components. For the feasible application of Mg sheet products, however, it is necessary that Mg sheets can be readily formable into complicated shapes at room temperature. Although Mg alloys, in general, show respectable tensile ductility at room temperature, their formability, e.g., limiting dome height measured by Erichson test, is rather low, mainly due to the dominance of basal slip during deformation and strong basal texture present in Mg alloy sheets. This presentation covers our recent activities on the development of Mg alloys formable at room temperature. The effect of alloying elements and thermomechanical treatments on the formability of various Mg alloys will be discussed. Several critical microstructural factors affecting the formability have been identified and suggestions will be made on how to improve the formability of Mg alloys at room temperature.

487 Understanding carbon redistribution processes during quenching and partitioning heat treatments

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Advanced high strength steels typically consist of complex microstructures that are optimised by both heat treatment and alloying composition. Understanding the redistribution of carbon between the different phases is fundamental in designing the steels, but it still remains a challenge due to the various carbon-trapping sites. For instance, Quenching-and-Partitioning (QP) steels rely on carbon partitioning from martensite into austenite, where at the end of the heat treatment the carbon-enriched austenite shows sufficient stability at room temperature. Nevertheless, recent literature gives evidence of more complex interactions involving carbon: (i) the occurrence of carbide precipitation during partitioning, and (ii) indication of higher carbon solubility in ferrite when in equilibrium with austenite. Both phenomena reduce the amount of carbon available to partition into austenite. The aim of the current study is to gain insight into carbon-competing processes by applying a series of QP heat treatments, with particular focus on the partitioning stage. Two model alloys are studied: Fe-1C-1Mn and Fe-1C-1Mn-2Si (wt.pct.). Various characterisation techniques such as dilatometry, X-ray

diffraction (XRD) and electron microscopy are put together in order to unveil carbon redistribution. In particular, establishing the tetragonality of the bcc-phase in XRD experiments will allow the distinction of primary martensite, which is carbon-depleted after the partitioning step, from the carbon-rich secondary martensite formed in the final quench of the heat treatment. Since the secondary martensite is directly formed from the austenite after partitioning, the relation between the two martensites is crucial for understanding the partitioning behaviour.

488 Kinetic analysis of densification by grain-boundary sliding/diffusion

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The densification behavior in the final and intermediate stage of sintering is analyzed for spherical isolated pores embedded in dense polycrystalline matrix. In the final stage, the shrinkage driven by grain-boundary diffusion is analyzed for small pores comparable to the grain size, and in the intermediate stage, the shrinkage driven by both grain-boundary sliding and diffusional accommodation is analyzed for large pores. From the analysis, the bulk viscosity, the densification rate and the pore-shrinkage rate are predicted as a function of the porosity, pore size, grain size and grain-boundary viscosity. The dependence of the pore-shrinkage rate on the relative density is opposite for the two sintering stages. In the intermediate stage, the shrinkage rate decreases with densification, whereas it increases in the final stage. With decreasing pore size, the contribution of grain-boundary sliding to the pore shrinkage decreases gradually due to restricted pore space, and further shrinkage occurs by grain-boundary diffusion only in the final stage. The present study suggests that the transition of the densification mechanism would occur during sintering. The mechanism transition is confirmed from the inflection observed experimentally in the pore-size distribution during sintering. We propose a new criterion to distinguish the sintering stage by a densification mechanism, not by a pore shape conventionally. The theoretical prediction of the present discrete model is also compared with the continuum model and experimental results. The dependence of the predicted densification rate on the porosity and on the grain size is well consistent to the experimental results for ceramics.

489 Microstructure evolution of rolled Al-Si-Mg alloys with Fe/Mn ratio

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Influence of rolling process on the formation behavior of the Fe intermetallic compounds in Al-Si-Mg cast alloys with Fe/Mn ratio has been investigated. Microstructure evolution of the rolled Al-Si-Mg based alloys was observed using SEM and TEM. Mn modifies the morphology and phase from the needle-like β phase into the small size and Chinese script and polyhedral α phase. The grain size of the rolled specimens became smaller compared with the as-cast specimen based on the EBSD results. The formation behavior of the Fe intermetallic compounds has been discussed in accordance with the reduction ratio of rolling process.

490 Feasibility study on characteristic of fatigue behavior using friction stir processing in high strength steel

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This study proposes a new postweld treatment method improving the weld bead shape and metal structure at the welding zone using friction stir processing (FSP) to enhance fatigue behaviour. For that, a pin-shaped tool and processing condition employing FSP has been established through experiment. Experimental results revealed that fatigue life improves by around 42% compared to as-welded fatigue specimens by reducing the stress concentration at the weld toe and generating a metal structure finer than that of flux-cored arc welding (FCAW).

491 Temperature tolerant polymer electrolytes for PEMFC

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PEMFCs have been attracting attention as a next-generation power generating device. The current study is in the high performance with the understanding of the material itself. Temperature tolerant electrolytes are in the applicants. A proton transport property depends on the degree of sulfonation and mobility in the polymer. Well controlled polymer can be displayed high proton conductivity in the temperature tolerant conditions.

We prepared a perfluorocarbon sulfonic acid and hydrocarbon polymers. The proton conductivity and cell performance of some polymers were higher than those of Nafion. We will discuss about that in the conference.

492 Novel siloxane-based copolymer for AEMFCs

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Anion exchange membrane fuel cells (AEMFCs) have been attracting interesting due to the possibility for the using of Pt-free catalysts. In the development of AEMFCs, high conducting anion exchange materials have been investigated to make a breakthrough in AEMFC performance. We prepared new siloxane-based anion exchange copolymer with soft segments of diphenylsiloxane (DPh). DPh parts are played a role to acting as impermeable filler, physical crosslink, and hydrophilic parts through grafting quaternary ammonium (QA) groups. QA functionalized DPh-DM copolymers can become an anion exchange electrolyte with the chloromethylation, quaternization and alkalization. The synthetic properties will be introduced in the conference.

493 Determination of the grain coarsening temperature in Nb microalloyed steels by multiphase-field model

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The grain coarsening temperature in Nb microalloyed steels is investigated using multiphase-field model. Rudnizki et al. [Computational Materials Science 49, 209 (2010)] have successfully simulated abnormal grain growth using effective mobility function under various initial conditions and constant pinning force. In this study, pinning force is treated to depend on the mean field kinetics of precipitates including precipitates volume fraction and their size. Using this model, the grain size distribution is calculated with time under temperature range from NbC dissolution temperature to highly precipitated temperature in order to investigate the effect of NbC precipitates on grain coarsening temperature. Through this model, it is possible to suggest the optimal conditions to obtain finer austenite grain size during processing.

494 The effects of ultrasonic nanocrystal surface modification technique temperature on microstructure and stress corrosion cracking of alloy 600

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Alloy 600 (UNS N06600) is an austenitic nickel-based alloy with superior corrosion resistance and high-temperature endurance, which determines its widespread applications in aeronautical, aerospace, marine and nuclear industries. Particularly, a number of nuclear components used Alloy 600 as their structure materials due to their high corrosion resistance, high-temperature endurance and excellent fabricant characteristics. Many failures have occurred in Alloy 600 with various forms of environmental degradations during long-term operation. In this study, an ultrasonic nanocrystal surface modification (UNSM) technique was applied to Alloy 600 at a room and a high temperature of 350 C. The effects of UNSM treatment temperature on the microstructure and slow strain rate test (SSRT) including hardness and compressive residual stress were investigated. The hardness and compressive residual stress were measured with respect to depth from the top surface. Also, the SSRT of UNSM-treated at a room and a high temperature Alloy 600 specimen was compared to that of the untreated specimen. The improvement in SSRT of Alloy 600 by UNSM technique was discussed in terms of increased hardness, refined grain size and induced compressive residual stress and modified microstructure.

495 Manufacture of high refractive index ophthalmic polymer lens with anti-reflection coating technology based high energy visible light blocking function

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The reduction of optical reflection is important for many applications such as solar cells and lenses. The high refractive index ophthalmic polymer lens with anti-reflection (AR) coating technology based high energy visible (HEV) light blocking function is manufactured using the lens monomer (MR series) of MITUI CHEMICAL INC (Japan). The high energy visible light from sunlight, LED light, LED TV, PC monitor, and mobile displays is harmful to eye care. It is known that the high energy visible light caused retinal damage. The effective graded-refractive index (RI) AR coating layer is designed a tri-layer structure with SiO₂, MgF₂, and ITO thin films for high refractive-index polymer ophthalmic lenses. The effective graded-RI AR coating is fabricated using the E-beam evaporation system. The optical properties of the HEV blocking ophthalmic lens were measured by UV-visible spectrometer. The ability to change the maximum reflectance band of an AR coating by simply changing the film thickness is advantageous for other potential applications.

496 Effect of large strain on texture formation behavior of AZ80 magnesium alloy during high temperature deformation

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In previous study, the formation behavior of texture and microstructure in AZ80 magnesium alloy under high temperature deformation was investigated. It was found that the basal texture was formed at stress of more than 15-20MPa and the non-basal texture was formed at stress of less than 15-20MPa. This means that stress of 15-20MPa is the change point of deformation mechanism. Therefore, in this study, uniaxial compression deformation of AZ80 magnesium alloy was carried out at high temperature deformation (stress of 15-20MPa). Behaviors of microstructure and texture development are experimentally studied.

The material used in this study is a commercial magnesium alloy extruded AZ80. The uniaxial compression deformation is performed at temperature of 723K and strain rate 0.003/s, with a strain range of between -0.4 and -1.3. Texture measurement was carried out on the compression planes by the Schulz reflection method using nickel filtered Cu K α radiation. EBSD measurement was also conducted in order to observe spatial distribution of orientation. As a result of plane strain compression deformation, the maximum value of the flow stress is observed at the true stress-strain curves, and the main component of texture and the accumulation of pole density vary depending on deformation condition.

497 Analysis of electric pulsing effect on mechanical behavior of magnesium alloy

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To investigate mechanism of effect of electric current on mechanical behavior of magnesium alloy, pulsed electric current was applied to the pre-tensioned magnesium alloy sheet. The mechanical properties of the specimen after electric pulsing were compared to those of the pre-tensioned specimen annealed at the measured maximum temperature during electric pulsing for total time of electric pulsing using oil bath to exclude Joule heating effect. The results showed that the change in mechanical properties after electric pulsing is solely associated with Joule heating. In-Situ TEM results also verified that dislocation structure does not change without heating during applying electric current.

498 Hot workability and extrusion characteristics of Al-Cu-Li-X and Al-Mg-Li-X alloys

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Aluminum-lithium alloys have low density, high strength, and high stiffness and therefore they have been extensively used for aircraft and spacecraft application. Wrought products of aluminum-lithium alloys are made by rolling, extrusion, and forging, and hot deformation condition should be precisely determined because it can play an important role in workability, microstructure evolution, and mechanical properties. In the present research, hot working behavior and extrusion characteristics of aluminum-lithium alloys were studied. Two different alloys, Al-Cu-Li-Mg-Ag-Zr and Al-Mg-Li-Zr alloys were prepared by vacuum induction melting, and hot compression tests were carried out using a thermomechanical simulator, Gleeble-3800. Processing maps were constructed using a dynamic material model, and deformation and failure modes during hot compression were studied. Indirect extrusion was used to investigate extrudability of the alloys, and the microstructural and strengthening mechanisms of the extruded alloys were suggested.

499 Extraction of high purity silicon for solar cell from Al die casting scraps

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A huge amount of Al die-casting scrap is produced from the casting industry every day due to the low volume ratio of the final product to its scrap, and its price is 0.4–2 \$/kg. If the cheap scraps can be used as source of high-purity Si, this application is more profitable than the re-use for die casting. We successfully fabricated solar-grade silicon (SOG-Si) feedstock using recycling aluminum die-casting scraps. 3N purity Si was extracted from A383 die-casting scrap by using the combined process of solvent refining and a novel centrifugal separation technique. The efficiency of separation depended on both impurity level of scraps and the starting

temperature of centrifugation. Impurities in melt and processing temperature governed the microstructure of the primary Si. The purity of Si extracted from the scrap melt was 99.963%, which was comparable to that of Si extracted from a commercial Al-30 wt.% Si alloy, 99.980%. The initial purity of the scrap was 2.2% lower than that of the commercial alloy. This result confirmed that high-purity Si can be extracted from die-casting scraps using the centrifugal separation technique.

500 Effects of die steel on the die soldering of aluminum alloy die-casting

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Die soldering is typical defects in die casting process of aluminum alloy. It has happened when aluminum sticks to die surface, made some metallic compounds. Until now, the various researchers have performed study about die soldering phenomenon but, most of them were studied effect of aluminum alloys on the soldering layer. So, in this study had carry out a die-soldering test using both H13 and carbon steel to confirm that which changes have occurred thereby using the two substrate materials. Each Aluminum alloys was melted and held at 680°C and both H13 and carbon steel were dipped for 2 hr in the melt. After dipping test, the specimens were air cooled and analyzed using a SEM and EPMA. The result of soldering test, reaction layer of H13 substrate has increased according to Si contents of aluminum, however, reaction layer thickness was decreased in the carbon steel substrate.

501 Microscopic elastic properties of polycrystalline Mg-Zn-Y alloys with long-period stacking ordered 18R phase by inelastic x-ray scattering

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Recently, a new series of Mg alloys with a synchronized long-period stacking order (LPSO), so-called KUMADAI Magnesium, [1] has attracted considerable attention owing to the excellent mechanical properties. By adding a small amount of Zn and rare-earth metals (Y or Gd) impurities, soft and flammable light weight Mg metal becomes harder and non-flammable. By taking these properties, the new Mg alloys might even be used for aircraft bodies. Macroscopic elastic properties were intensively investigated by several groups [2]. However, the microscopic information on the dynamics of this material is still lacking. We have measured polycrystalline Mg-LPSO alloys together with pure alpha-Mg by Inelastic x-ray scattering (IXS). Tiny but clear excitation signals of longitudinal acoustic (LA) modes are observed in the whole Q range. In addition, the transverse acoustic (TA) modes are detectable in the second Brillouin zone. For the Mg-LPSO alloys, the microscopic Poisson ratio is 0.22 ± 0.04 , slightly smaller than the macroscopic value of 0.26 and usual metal values of ~0.3, indicating a stiffness of the bond angles. In the presentation, we will compare the IXS results with the macroscopic

elastic constants [2], and discuss the microscopic elastic nature of the Mg-LPSO phase.
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[2] M. Tane et al., Acta Mater. 61, 6338 (2013).

502 Influence of prior-austenite grain structure on delamination toughening of ultra-high-strength low-alloy steels processed by warm tempforming

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We have recently developed a thermomechanical treatment, in which tempered martensite is deformed by tempforming at an elevated temperature. This is known as warm tempforming (WTF). When the WTF was applied to medium-carbon low-alloy steels, using multi-pass caliber rolling with a rolling reduction of 78% at a temperature of 773 K, ultrafine elongated grain (UFEG) structures with strong $\langle 110 \rangle$ //rolling direction (RD) fiber textures were formed and an excellent combination of ultra-high yield strength and notch toughness was achieved. The enhancement of the toughness for the warm tempformed steels is induced as a result of the occurrence of delaminations, in which cracks branch and propagate in the longitudinal direction of the notched bars. It is well-known that the prior-austenite grain size can influence the size of the blocks and packets in tempered martensitic structures. In the present study, 0.4%C-2%Si-1%Cr-1%Mo steels with different prior-austenite grain structures were quenched and tempered at 773 K and deformed by multi-pass caliber rolling with a rolling reduction of 78%. The influence of the prior-austenite grain structure on the delamination toughening was investigated for the warm tempformed steels. It was demonstrated that as the prior-austenite grain size decreased, the delamination toughening became significant at lower temperatures. The delamination toughening mechanism was discussed in relation to the UFEG structures and packet band structures that evolved through the extension of the blocks and packets, respectively.

503 Microstructure - mechanical properties relationship of MoSi₂/Mo₅Si₃-based eutectic composites

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MoSi₂/Mo₅Si₃ eutectic in-situ composites are promising candidates for ultra-high temperature structural materials because of their high eutectic temperature (1900°C) and fine microstructure of so-called script-lamellar type that is developed by directional solidification. However, poor fracture toughness at low temperatures and insufficient high temperature strengths have remained as drawbacks to be improved for their practical applications. Recently, we have systematically investigated the effects of ternary additions on microstructures and their thermal stability of MoSi₂/Mo₅Si₃-based eutectic composites in order to establish ways to overcome the drawbacks through controlling interfacial properties such as interfacial segregation of ternary elements and lattice misfits. Directional solidification experiments of MoSi₂/Mo₅Si₃ eutectic composites containing seventeen ternary elements were conducted by optical floating zone method. The ternary elements are classified into two types, group 1 and 2 mainly based

on the solubility of the ternary elements into MoSi_2 and Mo_5Si_3 . Among the group-1 elements (Ti, V, Cr, Nb, Ta and W) with relatively high solubility, the ternary additions of Ta and W are found to be beneficial in improving the fracture toughness by promoting the delamination along $\text{MoSi}_2/\text{Mo}_5\text{Si}_3$ interfaces. The group-2 elements (Fe, Co, Ni, Cu, Y, Zr, La, Hf, Ir, B and C) with negligibly low solubility are found to be effective in improving high temperature strength and fracture toughness through structure refinements and interface morphology modification caused by their strong segregation along $\text{MoSi}_2/\text{Mo}_5\text{Si}_3$ interfaces.

504 Material flow studies in friction stir welding: Part I - numerical material flow Modeling Part II - Cu inserts experiments, analysis

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The formation of joint in the friction stir welding, depends on Shoulder's Frictional contact and Pin's Shearing and Frictional Contact. In the present investigation, various non-circular pin profiles (Triangular to Hexagon) along with Straight cylindrical & Taper Cylindrical (30deg) are taken to study their impact on the weld quality. Initially Numerical modeling of the material flow were studied on Pure aluminum using the commercial DEFORM -3D FEA Code. Later experiments were conducted on pure aluminum with different copper inserts configurations (Centre; Perpendicular to Weld Advancing side (AS), Retreating Side (RS), Both; Parallel to Weld AS, RS), results were analyzed to understand the material flow behavior and weld formation. The initial results shows that the decrease in the z-axis load, and increase in the x-axis load as shearing and forging taking place during the weld. The flow cu insert configuration perpendicular to centre and along the weld shows the uniform dragging and mixing of cu-particles which suggests the flow region along the pin. The final flow results are correlated and concluded in all the aspects of XRD along with Texture and all other mechanical tests.

505 On the dynamic superplasticity

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Superplasticity is considered as a special state of the polycrystalline material plastically deformed at the low level of the stress with the retaining of the ultrafine-grained structure – structural superplasticity received at the previous stage or arisen during hot deformation independently from the initial grain size – dynamic superplasticity. For realization of the dynamic superplasticity it has to substitute an initial structural condition of material another, allowed to realize a superplasticity. The mentioned above changes are caused by the conforms of the proper strain rates and structural (phase) transformations of the evolutionary type in the open nonequilibrium systems. It is proposed an approach applying to the modelling of the deformation processes at the superplastic flow of commercial aluminum alloys taking into account the boundary regions in the framework the theory of self-organization of dissipative structures. An examples of the theoretical and experimental data correlation are given.

506 Bending deformation of Mg single crystals by three-point bending tests

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Magnesium single crystals with different orientations were applied to three-point bending tests, and the bending deformation behavior was investigated. When the specimen deformed due to basal slips, it showed gull-shape after deformation. On the other hand, when the specimen deformed due to {10-12} twinning, it showed V-shape. Relationships between bending yield stress of the specimens and critical resolved shear stress for the basal slip, or {10-12} twin was discussed.

507 DSC analysis of martensitic transformation temperature in casted Ti–Ni shape memory alloy

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The effect of cold rolling on martensitic transformation temperature in cast Ti-50.5at%Ni shape memory alloy from self-propagating high temperature synthesis (SHS) ingot were studied. Shape memory alloy specimens were casted by lost-wax process from SHS ingot. The heat treatment and the change of the transformation temperature by the rolling conditions were measured by differential scanning calorimetry (DSC).

508 Effect of oxygen potential gradient on mass transfer in alumina layer at high temperature

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The durability of thermal barrier coatings is strongly affected by interdiffusion of oxygen and aluminum along grain boundaries (GBs) in alumina scales formed on the bond coat in a high-temperature oxidizing atmosphere. However, the mechanism involved in the mass transfer phenomenon is still not clear. In the present study, the mass transfer mechanism in alumina wafers, which serve as models for the alumina scales, was investigated by accelerated tests of the oxygen permeability of the wafers for a large oxygen potential gradient ($d\mu_{\text{O}}$) at 1600 °C under dry and humid environments with $^{18}\text{O}_2$, and/or water vapor. The oxygen diffusion coefficient along GBs near the high oxygen partial pressure ($\text{P}_{\text{O}_2(\text{hi})}$) surface was experimentally determined from the ^{18}O depth profile along GBs measured by secondary ion mass spectroscopy for a cross section of the exposed wafer. The ^{18}O was concentrated at GB ridges on the $\text{P}_{\text{O}_2(\text{hi})}$ surface. Dissociative adsorption of $^{18}\text{O}_2$ occurred on the surface, and subsequently ^{18}O diffused to surface GBs, resulting in the formation of new alumina by reaction with aluminum diffusing outward along the GBs. The oxygen GB diffusion coefficient near the $\text{P}_{\text{O}_2(\text{hi})}$ surface was comparable to that calculated assuming an electronic conductor from the oxygen permeation data, and was clearly lower than that for oxygen GB self diffusion without

$d\mu_{\text{O}}$. Furthermore, in the case of large $d\mu_{\text{O}}$, water vapor included in the $\text{P}_{\text{O}_2}(\text{hi})$ environment enhanced GB diffusion of oxygen near the $\text{P}_{\text{O}_2}(\text{hi})$ surface.

509 Microstructure and creep property of silicon- and/or germanium-bearing near-alpha titanium alloys

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The effect of Si and/or Ge addition on microstructure and creep property at 650°C and 137MPa was investigated in near-alpha Ti-Al-Sn-Zr-Mo alloys. Si and/or Ge addition decreased minimum creep strain rate due to solid solution of Si and Ge in matrix, and precipitates of silicide, germanide and these solid solution. The decrease in the minimum creep strain rate in the alloy with 1 wt% Ge without Si was most significant due to very fine germanides. A marked shift in the lattice parameters of alpha and beta phases was detected for the alloy containing 4 wt% Ge. The characterization of alpha/beta interfaces using Moiré fringe observed in high-resolution transmission electron micrographs indicated that the structural ledges in the Ge-bearing alloy had a smaller ledge size and a greater number of ledges compared with those in Si-bearing and base alloys.

510 Short-time procedure to quantify the cyclic hardening potential of metallic materials by cyclic hardness tests - PhyBaL-CHT

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For the development of materials with improved properties, e.g. higher damage tolerance, an efficient estimation of cyclic properties is necessary. Quantifying the cyclic hardening potential with conventional methods requires time and cost consuming fatigue experiments. Hence, a short-time procedure - PhyBaL_{CHT} - was developed which requires planar, polished material sections for testing and is therefore suitable for characterization even if only small quantities of test material are available. To characterise the hardening potential the plastic response (i.e. width) of the force-indentation-depth-hysteresis is plotted vs. indentation cycle number. The resulting cyclic indentation hardening curve is characteristic for each material depending on alloy composition and heat treatment. In the present study, the PhyBaL_{CHT} is applied in the development of a damage tolerant TRIP-modified SAE 52100 steel. One of the main reasons initiating fatigue fracture in SAE 52100 components, i.e. roller bearing rings, are microstructural imperfections like nonmetallic inclusions. Stress concentrations around such defects should activate TRIP-induced local hardening and compressive residual stresses in order to inhibit crack initiation or crack growth, resulting in improved damage tolerance.

To assess alloying compositions and heat treatment variants with respect to cyclic hardening capability, PhyBaL_{CHT} was applied. An extensive parameter field was evaluated to detect alloy / heat treatment combinations with maximum cyclic hardening potential. One TRIP-modified SAE 52100 was selected for further fatigue investigations and the fatigue behaviour, the cyclic hardening as well as the damage tolerance were compared with conventional SAE 52100 steel.

511 Splat analysis and assessment of porosity in thermal barrier coatings produced by axial suspension plasma spraying (ASPS)

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Thermal barrier coatings (TBCs) used in gas turbines are commonly produced by atmospheric plasma spray technique (APS) or by electron beam physical vapor deposition (EB-PVD). Due to their columnar microstructure, EB-PVD coatings can accommodate thermal stresses better than APS coatings, providing them with a longer life-time. However, a major drawback of EB-PVD coatings is their high production cost. Axial suspension plasma spraying (ASPS) is a relatively new, innovative spraying technique using fine powder particles suspended in a liquid as feedstock. When injected into the plasma plume, the suspension breaks up and forms individual droplets, the solvent evaporates and the powder particles melt. Upon impact on the substrate, the molten particles are expected to flatten out and solidify building up the microstructure. The produced ASPS coatings have been shown to exhibit superior durability compared to conventional APS and EB-PVD coatings. This has been attributed to the fact that ASPS coatings do not show the typical splat structure with flattened particles. In fact, a columnar structure with vertical cracks and nanometer-sized pores is formed by the much smaller splats. The mechanism of coating formation during ASPS processing is not yet well understood. An important part in the characterization and property assessment of ASPS coatings is therefore the analysis of splats and the determination of porosity which will be addressed in this presentation.

512 Structure formation in Ni superalloys during high-speed direct laser deposition

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Additive technologies are replacing the conventional methods of casting and subsequent time-consuming machining because of its high productivity. Recent engineering development in the field of additive manufacturing allows increasing assortment of useful powder materials. Technology of high-speed direct laser deposition (HSDLD) is a one of most perspective new technologies. It allows realizing heterophase process during the manufacturing, which there is process of partial melting of used powder is realized. The product is formed from a metal powder, which is supplied by compressed gas-powder jet directly into the laser action zone, wherein the jet can be as coaxial and as non-coaxial. Ni-based alloys found their application in many industrial areas, mostly there are used engine systems, aircraft and shipbuilding, aeronautics. The unique combination of operational characteristics depending on the type of alloy makes them promising materials. Heating and cooling rates during direct laser deposition determine structure and affect on its properties. Research is focused on structure and phase formation within technological process of HSDLD for Ni-based superalloys. Mechanical tests were carried out on the creep-rupture test and static tensile test, microhardness was measured. Based on research results the high-speed direct laser deposition technology could be used for

manufacturing of products from different Ni-based alloys without subsequent heat treatment.

513 Synchrotron radiation investigations of niobium precipitates in HSLA steel

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Precipitation and dissolution behaviour of niobium carbo-nitrides is of particular interest for many technical applications. Reasonable equations for the solubility product at thermal equilibrium can be taken from literature but kinetics is largely unknown. Investigation of nano-scale niobium precipitates by TEM analysis or chemical extraction methods is common practice. However, TEM suffers from statistical relevance and chemical extraction will not give information on particle distribution and orientation. Information on kinetics is difficult to obtain from both methods, as samples have to be quenched from soaking to room temperature. Besides the considerable experimental effort the situation at soaking temperature is evaluated from the room temperature state. In contrast, investigation by high energy synchrotron X-ray of about 100 keV offers statistical relevance as volumes of several cubic millimeters are regarded. The wavelength of around 0.2 Å is able to analyze nanometer particles. In addition this technique enables in situ observation of the precipitation state as chemical composition, orientation and tilt towards the surrounding austenitic or ferritic matrix. This contribution summarizes results from high energy synchrotron X-ray investigations of the precipitation behaviour of nanometer-sized niobium-carbides (NbC) during thermomechanical processing of niobium microalloyed steel. This technique offers in situ observation of the precipitation state as chemical composition, orientation and tilt towards the surrounding matrix. Observed volumes of several cubic millimeters give statistic relevance to the obtained results. Synchrotron investigations on laboratory and industrial samples have been carried out at DESY in Hamburg. The results establish this technology for qualitative and quantitative analysis of nano-scale niobium carbo-nitride precipitates.

514 Molybdenum based materials and their challenges in production and applications

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Molybdenum and its alloys are representatives of high melting point metals also named refractory metals. These metals, in particular molybdenum and tungsten, exhibit an unique combination of properties – high temperature strength, high Young's modulus, excellent corrosion resistance against liquid metals and glasses, good electrical and thermal conductivity, and a low coefficient of thermal expansion, which is e.g. close to glasses and semiconductor devices. Primarily these metals were mainly used for high temperature applications, however during the last decades there is a clear trend that these metals have been applied more and more near room temperature, e.g. electronic applications. Nevertheless, it is important to have fundamental understanding of the material behavior at elevated temperatures in order to optimize manufacturing processes and to develop new products for usage under extreme conditions. Therefore, Plansee SE has been placing emphasis for more than five years on the

recrystallization behavior of pure molybdenum depending on texture, on the increase of strength in case of the precipitation – hardened molybdenum based alloy MHC and on improving grain boundary strength of molybdenum. The latter project is combined with developing suitable sample preparation as well as analytical methods to investigate grain boundaries having low segregation levels and atomistic modelling. After an introduction about the properties and applications of molybdenum in general, the presentation will give an overview about the work and the results of the running projects mentioned above.

515 Advances in the electrochemical synthesis of polymer electrolytes for microbatteries

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Microbatteries are needed for powering autonomous microdevices, such as microelectromechanical systems (MEMS), microsensors and actuators, implantable medical devices, or RFIDs. The current two-dimensional (2D) microbatteries show a relatively low areal capacity and energy density; in order to increase the energy density without sacrificing power density, a 3D design, using nanowire or nanotube electrode morphologies, is mandatory [1]. We work since several years on 3D lithium ion microbatteries based on TiO₂ nanotubes as negative electrode [2]. Concerning the solid electrolyte separator, our achievements include the electrodeposition of the polymer electrolyte PEO-PMMA inside TiO₂ nanotubes. A good cyclability was demonstrated with this separator [3]; however, the polymer electrolyte can gelify and dissolve in presence of water, for example during the ulterior deposition of the positive electrode inside the polymer lining. The objective of our work is now to develop a separator using ionic-conducting aromatic polymers with high mechanical stability and low solubility in water. In this talk, I will present the various approaches, including electrodeposition of PEO-PMMA and of sulfonated poly (phenylene oxide). I will talk about the electrochemical conditions for deposition, the characterization of the polymers by FTIR and NMR spectroscopies, scanning electron microscopic observations and the electrical and electrochemical properties of the new solid polymer electrolytes.

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516 Tensile and fatigue properties of miniature size specimens of Sn-5Sb lead-free solder

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Tensile and fatigue properties of Sn-5.0Sb lead-free solders were investigated using micro-size specimens. As-cast micro-size specimens with 0.5 mm diameter and 2 mm gauge length were fabricated from the solder wire. The tensile test was conducted at the strain rate ranging from 2×10^{-3} to $2 \times 10^2 \text{ s}^{-1}$ at room temperature. Specimens were not necked uniformly and chisel point fracture was observed. Tensile strength of Sn-5.0Sb becomes relatively low compared with those of Sn-3.0Ag-0.5Cu and Sn-37Pb solder. For elongation, Sn-5.0Sb has an excellent value

of approximately 90% at the maximum strain rate investigated. The value was larger than those of Sn-3.0Ag-0.5Cu and Sn-37Pb solder (approximately 40%). Fatigue test was conducted at the strain rate of $2 \times 10^{-3} \text{ s}^{-1}$ at 25°C and 125°C. In the fatigue test, strain was controlled using a symmetrical triangle wave and total strain range was controlled in the range of 0.5 % to 2.0%. The fatigue life was defined as a 20 % decrease in load. In the low-cycle fatigue, the fatigue life of Sn-5.0Sb is as same as that of Sn-3.0Ag-0.5Cu at 25°C. Since the fatigue life of Sn-5.0Sb is scarcely decreased with increasing temperature from 25°C to 125°C, it is superior to that of Sn-3.0Ag-0.5Cu at 125°C. In the paper, the effect of temperature on tensile properties of Sn-5.0Sb and high-cycle fatigue properties of it will be also reported.

517 First principle analysis for the effect of beta stabilizer in Ti alloys on the formation of alpha double prime phase

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Metastable alpha double prime phase would be formed from high temperature beta phase of Ti alloys containing beta stabilizer such as Nb and Mo during quenching. The compositions for alpha double prime phase formation in Ti-Nb and Ti-Mo systems are reported as 8-28at%Nb and 2-7at%Mo, respectively. The ranges are fairly different, i.e. 20at%Nb in Ti-Nb system and 5at%Mo in Ti-Mo system and the reason for such the difference has not been understood. The formation of alpha double prime phase would relate to the soft phonon mode of $1/2[110]T$ and $2/3[111]L$. The purposes of present research to examine the effect of beta stabilizer on the phonon dispersion curves of Ti alloys and to reveal the reason for such the difference in various Ti alloys. The force constant of a Ti alloy was calculated by using the VASP. Electron-electron interactions are treated within the generalized gradient approximation (GGA). Phonon dispersion was calculated by the Phonopy. The soft phonon mode of $1/2[110]T$ of a Ti-Mo alloy became non-soft mode by the addition of 10at%Mo and then that of $2/3[111]L$ became non-soft mode at Ti-20at%Mo. On the other hand, soft phonon mode of $2/3[111]L$ of a Ti-Nb alloy became non-soft mode by the addition of 20at%Nb, while that of $1/2[110]T$ remained in the soft mode. This implies that Nb addition in Ti does not stabilize phonon mode of $1/2[110]T$ resulting in wide composition range of alpha double prime phase formation in Ti-Nb system.

518 Effect of grain boundary microstructure on electrical conductivity in gold thin films produced by sputtering and subsequent annealing

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Improvement of electrical conductivity in metallic thin films is indispensable for development of high performance advanced electronic devices. It is well known that grain boundaries are resistant for electrical conductivity in metallic interconnects, depending on the character and structure of distinct grain boundaries. Therefore, electrical conductivity in metallic interconnects can be improved by optimization of grain boundary density and grain boundary

character distribution. However, grain boundary engineering has not been seriously applied to development of high performance thin films. The grain boundary microstructure in the gold thin films sputtered on the different kinds of substrate on was controlled by annealing in various atmosphere. The gold thin film specimens with various average grain size, namely grain boundary density, and fraction of low-sigma coincidence site lattice (CSL) boundaries were obtained by the development of sharp $\{111\}$ -texture resulting from surface energy-driven grain growth. The lower resistivity was obtained in the gold thin film specimen with the larger grain size (the lower grain boundary density), when the specimens had similar fraction of low-sigma CSL boundaries. Moreover, the higher fraction of low-sigma CSL boundaries induced the lower electrical resistivity in gold thin film specimens when the specimens had similar average grain size (grain boundary density).

519 Effect of pre-deformation on TiC precipitation kinetics in ferritic steel

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Fine alloy carbides are beneficial to strengthen steels with the addition of a small amount of alloying elements. It is of great importance to understand precipitation kinetics for the control of the size and number density of particles, which are factors of particle strengthening. Previously, we investigated the precipitation behavior of titanium carbide (TiC) in ferrite matrix with isothermal aging using a model steel with a low dislocation density, and observed the precipitation state from homogeneous nucleation to coarse growth. However, steels manufactured by actual production often have a high dislocation density due to deformation and/or phase transformation, which may influence the precipitation kinetics and strengthening. In this study, we investigated the influence of a dislocation density on the precipitation kinetics of TiC. The model steel (Fe-0.03C-0.10Ti-0.2Mn-3Al (wt%)) has no phase transformation and remains ferritic throughout the solution treatment. In order to make a difference in the dislocation density of matrix before aging, some of the solution treated steels were cold-worked by true strain of 0.5 (pre-deformation), and the others were without pre-deformation. We compared the steels with/without pre-deformation after aging at 580 °C for the same times for the precipitation of TiC. Atom probe tomography analyses revealed that TiC precipitate, not only on dislocations but also homogeneously in matrix, much earlier with higher number density in the pre-deformed steels than in the steels without pre-deformation. The reason for the difference is discussed in terms of the competition among C segregation to dislocation, precipitation of cementite and TiC.

520 The effect of thermomechanical processing temperature-strain-time parameters on the mesostructure formation

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The effects of Temperature-Strain-Time parameters at the Thermomechanical Processing (TMP) of austenitic, duplex and pearlitic structural steels on the mesostructure formation has been studied based on the laboratory, industrial experiments and TEM analysis. The fragmented dislocation substructure observed in steels with a different carbon, nitrogen, titanium, niobium content as well as recrystallization gave evidence that TMP effects the work-hardening and softening behaviour. The problem of mesostructure appearing in various steels and alloys due to various modes of TMP used hot and hot-warm deformation is discussed. The role of plastic strain in the formation of mesostructure and the relation between the changes in the crystal structure due to TMP and the mechanical properties of the steels are considered.

521 Structure of a non-glass forming oxide liquid

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Atomistic and electronic structures of high-temperature non-glass forming liquid, ZrO_2 was revealed to understand the origin of glass-forming ability in liquids. The X-ray structure factor, measured by the use of aerodynamic levitation technique, was well reproduced by density functional (DF) - molecular dynamics (MD) simulation. The first sharp diffraction peak (FSDP) was not observed in the Bhatia-Thornton number-number partial structure factor $S_{NN}(Q)$ obtained by DF-MD simulations. The formation of distorted ZrO_5 , ZrO_6 , and ZrO_7 polyhedra with a significant contribution of edge-sharing of oxygen was confirmed. The variety of large oxygen coordination and polyhedral connections with short Zr-O bond lifetimes, induced by the relatively large ionic radius of zirconium, disturbs the evolution of intermediate-range ordering, which leads to a reduced electronic band gap and increased delocalization in the ionic Zr-O bonding. The details of the chemical bonding explain the extremely low viscosity of the liquid and the absence of a FSDP. Reference: S. Kohara et al., Nat. Commun., 5, 5892 (2014).

522 First-principles local-energy and local-stress calculations of materials interfaces

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We present our recent development of computational techniques for local energy and local stress based on density-functional theory (DFT) [1-3]. In conventional plane-wave DFT methods, total energy and stress tensor are given as integrated or averaged quantities in a supercell, and local distributions of energy or stress are not obtained. Previously, the schemes

to calculate energy density and stress density were proposed, while the inherent gauge-dependent problem associated with the choice of symmetric or asymmetric forms of the kinetic terms prevented practical applications. Within the PAW-GGA framework, we have developed the computational techniques to obtain local energy and local stress as unique physical quantities via integrating the energy and stress densities inside local atomic or layer regions to satisfy the gauge-independent conditions [1, 2]. In this talk, we present our recent applications to i) grain boundaries (GBs) in Fe, Al and Cu, ii) impurity segregation to metallic GBs, iii) tensile tests of GBs, iv) local bulk moduli of Fe-Si alloys, and v) Fe/TiC interfaces. We discuss how the local energy and local stress can provide new insights into local bonding and properties of materials interfaces, and discuss remaining problems and future applications.

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523 Structures and properties of laser-assisted cold-sprayed metallic coatings

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In the cold spray process, solid particles impact with high kinetic energy, deform plastically and form a coating. This enables the formation of pure and dense coating structures. Even more, coating performance and deposition efficiency can be improved by assisting the process with a laser. Laser-assisted cold spraying (LACS) has shown its potential to improve coating properties compared with traditional cold spraying. In this study, coating quality improvement was gained by using a co-axial laser spray (COLA) process which offers a new, cost-effective laser-assisted cold spray technique, for high-quality deposition and repair. In the COLA process, the sprayed surface is laser heated while particles hit the surface. This assists the better bonding between particles and substrate and leads to the formation of tight coating structures. This study focuses on the evaluation of the microstructural characteristics and mechanical properties (e.g., hardness and bond strength) of LACS metallic coatings.

524 Formation of the C-type orbital-ordered state in the highly-correlated electronic system $\text{Ca}_{1-x}\text{Pr}_x\text{MnO}_3$

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The C-type orbital ordered (COO) state has been reported in the highly-correlated electronic system $\text{Ca}_{1-x}\text{Pr}_x\text{MnO}_3$ (CPMO) with $0.10 \leq x \leq 0.25$. The COO state is characterized by a spatial array of $(3z^2-r^2)$ -type orbitals for 3d electrons in Mn ions. The feature in CPMO is that the COO state appears by cooling from the disordered orthorhombic (DO) state, instead of a disordered cubic one in other manganites such as $\text{Sr}_{1-x}\text{Nd}_x\text{MnO}_3$. In spite of the difference, however, the formation of the COO state from the DO state has not been understood yet. We have examined the crystallographic features of the (DO→COO) transition in prepared CPMO

samples with $0.10 \leq x \leq 0.20$ by both x-ray powder diffraction and transmission electron microscopy. The experimental data obtained in this study first confirmed that the DO state had the orthorhombic Pnma structure for $0.10 \leq x \leq 0.20$. When the temperature was lowered from the DO state, the COO state with the space group of $P2_1/m$ was found to appear by the introduction of the e_{xz} strain, as a Jahn-Teller distortion for C-type orbital ordering, into the DO state. In the DO state, furthermore, we found both a slight increase in half-width values of x-ray powder diffraction profiles on cooling and the presence of characteristic diffuse scattering around each reflection in electron diffraction patterns. From these data, it is suggested that the formation of the COO state in CPMO accompanies remarkable fluctuations of orbital ordering in the DO state.

525 Hydrogen storage materials for hydrogen economy

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Hydrogen gas has poor volumetric energy density. Hydrogen storage technology is essentially necessary to realize the hydrogen economy. The hydrogen absorption and desorption capacities of alloys such as $Ti_{1.1}CrMn$ (AB2) and $Ti_{0.22}Cr_{0.39}V_{0.39}$ (BCC) were 1.8-2.2 wt % at 296 K. Pt-LiCoO₂ worked as an excellent catalyst for releasing hydrogen from NaBH₄-H₂O system. Nano-composite materials have been synthesized to modify the thermodynamic and kinetic properties of chemical hydrides. Superactivated carbon having specific surface area about 3000 m²/g can adsorb 5 wt % of hydrogen at liquid nitrogen temperature and 1.3 wt % of hydrogen at 296 K. It was confirmed that 10 wt% of hydrogen can be adsorbed on the activated carbon at 20 K. The density of adsorbed hydrogen was evaluated to be much larger than that of liquid hydrogen. Ammonia is a liquid chemical hydride and store hydrogen with high gravimetric density of 17.8 wt% and highest volumetric density of 10.7 kg H₂/100L as a liquid with mild pressure of 1.0 MPa at 298 K. The catalytic ammonia conversion to nitrogen and hydrogen is 99.8% at 0.1 MPa and 773K using active Ru-based catalyst. The remained ammonia concentration is 1000 ppm. The materials with ionic bonding will be useful for ammonia removal below the ammonia concentration of 0.1 ppm for fuel cell vehicles. Hydrogen storage materials will be applied for hydrogen energy carriers, emergency use, heat storage, hydrogen purification, next generation battery, on-board and on-site hydrogen storage systems.

526 A comparative study of CALPHAD and differential scanning calorimetry to optimize 7xxx aluminum alloys

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7xxx series aluminum alloys, especially the copper containing variants, are widely used for aircraft applications because of their superior mechanical properties and strength/weight ratios. Conventional 7xxx series alloys without lithium or scandium have been extensively investigated over last decades. However, well-considered thermodynamic calculations via the CALPHAD approach on a variation of the alloying elements can guide the fine-tuning of known

alloy systems and the development of optimized heat treatments. In this study, we compare results from thermodynamic predictions of the phase evolution and the solidus temperature with results from differential scanning calorimetry (DSC) measurements. The software algorithms implemented in FactSage, PandatTM, and MatCalc and corresponding commercially available databases were used. A systematic variation of the main alloying elements Mg, Zn and Cu, which are most important for precipitation hardening in 7xxx series alloys, resulted in more than 50 experimentally produced alloys. The work illustrates how thermodynamic calculated data can help to find optimized alloy compositions excluding prohibited areas with low melting phases in the temperature field of solution treatment procedures. The predicted fractions of phases prone to precipitation hardening are compared to hardness and strength values of the corresponding 7xxx series alloys. Moreover, the quality of the commercially available CALPHAD data is critically reviewed.

527 The effect of boron addition on precipitation and hot ductility of 1.5Mn-0.1Nb-Ti carbon steels in as-cast condition

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Twelve experimental steels with a base composition 1.5wt% Mn, 0.01wt% V and 0.1wt% Nb and varying C (0.05, 0.10 and 0.20wt%), Ti (20 – 250 ppm) and B (0 – 100 ppm) contents have been systematically examined to quantify the effects of composition on precipitation behaviour and hot ductility during simulated continuous casting. FEG SEM analysis showed that Nb-rich precipitates were present in the alloys with 0.10 wt-% C and 0.20 wt-% C. Additionally, all alloys with 0.05, 0.10 and 0.20wt% C showed the presence of Ti-Nb carbonitrides with size range 50 – 100 nm. Boron was bound to boronitrides in the size range 20 – 100 nm located in prior austenite grain boundaries.

Hot ductility- and relaxation tests were performed with a Gleeble 3800 to study hot ductility and strain induced precipitation processes in the alloys. Remelted and solidified tensile tests showed that the alloys without B or Ti additions exhibited poor hot ductility at 850°C and 950°C (reduction in area < 30%), whereas the 0.05 wt-% and 0.10 wt-% C alloys exhibited improved hot ductility (reduction in area 40-50%) by the addition of either >50ppm B or 250ppm Ti. The 0.2 wt-% C alloys showed no improvement from B or Ti additions. Examination of fracture surfaces of hot ductility specimens showed that boronitrides were present at prior austenite boundaries in all alloys that contained 80 – 100 ppm of B. Compression-relaxation tests showed that alloying with boron caused a noticeable decrease of the strain-induced precipitation start temperature in the alloys.

528 Mechanical properties of shape memory alloy fiber / aluminum composite fabricated by spark plasma sintering

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Aluminum was composited with iron-base shape memory alloy (SMA) fiber. Matrix metal can obtain compressive residual stress caused by shape memory effect of SMA fiber with strong interface between matrix and SMA fiber. It is important to join between matrix metal and reinforced SMA fiber successfully. In this study, aluminum matrix composite reinforced by iron-base SMA fiber was fabricated by Spark Plasma Sintering (SPS). The pure aluminum powder with iron-base SMA fiber was joined at 773K. As a result, intermetallic phase was consisted at the interface between aluminum and iron-base SMA fiber and it was clarified that the interfacial strength depends on kind and thickness of intermetallic phase. This strong interface gives beneficial residual stress into aluminum from SMA fiber. Mechanical properties of the SMA fiber / aluminum composite are improved with compressive residual stress by recovery process of SMA.

529 Nanoparticles-supported carbon nanowalls for green energy applications

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Carbon nanowalls (CNWs), self-aligned three-dimensional graphene materials, consist of graphene sheets standing vertically on substrates. Significant recent attention has been focused on the functionalities of the CNWs because of their unique morphologies and excellent electrical properties. For example, since they have large surface-to-volume ratios and very high aspect ratios, they are expected as catalyst supporting materials in fuel cells, field emitters, and various kinds of templates. In addition, high-density graphene edges on the top-region of CNWs promise expression of novel functionalities and applications.

In this study, we have realized ultrahigh-density nanoparticles, such as Pt, TiO₂ and so forth, supporting onto the whole surface area of CNWs employing a super critical fluid. In the case of Pt nanoparticles, a number density of supporting nanoparticles is found to be proportional to the density of surface defects on CNWs. Then, it reaches more than 10^{13} cm^{-2} at the optimized conditions. Furthermore, at catalytic performance tests, the Pt nanoparticles-supported CNWs show high-efficiency and highly-durable properties. Especially, at a potential cycle test simulating repeated start-stop operations of fuel cells, they hardly show drastic degradation at over 20,000 cycles, although a conventional carbon black usually show significant degradation around 2,000~4,000 cycles. These results indicate the high potential of CNWs as a highly-durable catalytic support for the future high-performance fuel cell.

530 Structure and microstructure of the phases involved in functional behavior in Co-Ni-Al and Ni-Mn-Ga systems

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The shape memory alloys (SMA) became widely applied mainly in medicine since its discovery in 1932. Except application based research, the investigation of SMA helps to understand various aspects of martensitic transformation. Early 90s brought (again) the attention to magnetically induced martensitic transformation in ferromagnetic SMAs and particularly Ni-Mn-Ga based alloys formed new topic, which originally contained Co-Ni-Al alloys too. Compared to Ni-Mn-Ga system with various modulations and extremely low energy of twinning boundaries, the ferromagnetic shape memory Co-Ni-Al systems exhibit just B2 to L1₀ transformation without modulation and the martensite transformation or the reorientation could not be induced by magnetic field. In contrast to Ni-Mn-Ga the wide variety of nanoprecipitate types are observed in Co-Ni-Al alloys similar to Ni-Ti alloys. These nanoparticles are thought to be responsible for deformation via twinning as the long-distance force fields of precipitates block the moves of dislocations. On the other hand, no such precipitates were found in Ni-Mn-Ga and no high dislocations density observed presumably due to high energy formation in ordered structure, which leaves the twinning as the favorite way of deformation also in Ni-Mn-Ga. In the presented lecture we try to compare functional properties of both systems with their microstructure. We expect here crucial role of twinning boundaries, high-temperature order and, if any, formation of nanoparticles. It seems that these systems can be understood as the model types of SMAs. Authors would like to acknowledge the financial support from the Czech Science Foundation projects 15-00262S.

531 New metallurgy of additive manufacturing in metal: experiences from the material and process development with electron beam melting technology (EBM)

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Additive manufacturing (AM) is becoming one of the most discussed modern technologies. Significant achievements of the AM in metals today are mainly related to the unprecedented freedom of component shapes this technology allows for. But full potential of these methods lies in the development of new materials designed to be used specifically with AM. Present paper discusses the issues related to the beam melting AM technologies allowing speaking of new direction in material science that can be termed “non-stationary metallurgy”, using the examples from material and process development for EBM. Many materials for AM like Ti6Al4V emerged from other industrial applications, where the processes are already well understood. But with the growing research into AM in metal the full extent of specifics of involved processes is starting to emerge. With the beam melting methods like EBM, temperature increase rates in the melt zone can exceed millions Kelvin per second. Though cooling rates are lower, they also can exceed tens thousands K/s. As energy deposition is

strongly localized, temperature gradients in and around melt zone can be extreme. This is leading to new multi-scale structures and new materials previously not possible with other manufacturing processes. Proper understanding of the AM process details will open up new possibilities, where material and component properties can be tailored for specific needs by controlling and adjusting the parameters through the manufacturing process. So the challenge for the material science is to define the relationships between the process parameters and the material properties in non-stationary metallurgy.

532 Thermo-mechanical treating of magnesium alloys and its influence on cold working plasticity

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The paper presents investigations conducted on AZ31 and AZ61 magnesium alloys. The aim was to improve their workability in tube bending process. Considering plastic elongation significant increase (from $A_{10}=10\%$ to more than $A_{10}=20\%$) was obtained. To achieve that, a number of tests were carried out, including extrusion process with several cooling velocities, heat treating, and additional plastic forming (like stretching process). On every stage of the trials both structural and mechanical test have been done. The final stage of investigations were tube bending trials where the maximum bending angle was tested.

533 Effect of temperature on shear localization in Cu-Ag nanocomposites

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Elemental Cu and Ag powders were mixed in certain compositions and subsequently compressed and deformed by high-pressure torsion. The disk-shaped samples (diameter 8 mm, thickness 0.5 mm) were characterized regarding microstructure and mechanical properties at different strain levels and varying processing temperature. At very high applied strains different nanocomposites are formed. In order to investigate the deformation mechanisms a special experimental setup was used. Samples were cut at a radius of 2mm and the disk cross-section was polished. In the center of the cross-section markers in form of grids (0.1 x 0.1mm) were cut by focused ion beam technique. Such prepared samples were put together with matching counterparts to obtain a full 8 mm disk again. Thereby additional shear deformation using the high-pressure torsion tool can be applied and after separating the two pieces again, changes on the grid markers can be analyzed. Up to four additional deformation steps were performed, the markers were characterized by SEM after each step. At room temperature processing a change in the deformation mechanism was observed. At low shear strains co-deformation of the powder particles leads to a fine lamellar microstructure of alternating Cu and Ag bands. When a critical strain level is reached, thus a critical lamella spacing, the deformation starts to localize in form of shear bands. At high strain levels, the entire deformation is realized by large shear bands propagating through the whole sample. Increasing the processing temperature leads to a combined deformation process via homogenous deformation and shear banding.

534 Texture characterization of stainless steel clad layers of process vessels

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In this study texture and residual stress of process vessels made of ferritic base alloys clad by protective layers of stainless steel were investigated by neutron diffraction using the diffractometer STRESS-SPEC at FRM 2 (Garching, Germany). Different samples were prepared: as welded and as welded plus relevant industrial heat treatment. Local texture measurements with a gauge volume of $2 \times 2 \times 2 \text{ mm}^3$ of the three cladding layers (at depths of 2 mm, 5 mm and 7.5 mm) for each sample were determined. Strain gradients in depth were carried out with the same gauge volume. Texture results indicated that there exists an annealed cube component in all the studied samples. Based on the measured pole figures at each depth the calculated orientation distribution functions data were used to calculate the Young's modulus with respect to each sample orientation. The calculated anisotropic Young's modulus in depth was then used as an input for the residual stress profile calculations. The residual stress distribution will then be discussed.

535 Multi-functionality of nanostructured silicon

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As the scaling of Si ULSI devices progresses deep, the design and fabrication rule approaches the size criterion at which the confinement effect becomes apparent. Actually, nanocrystalline Si (nc-Si) with a size smaller than 4 nm behaves as a wide-gap and functional material. It paves the way for researchers in the field of Si devices to explore post-scaling technology. Actually, emerging diverse studies have been conducted in photonics, acoustics, MEMS, sensors, biometric, and biomedical area. Among them, some topics on the functionality of nc-Si are discussed here in relation to emissive properties of photons, electrons, and acoustic wave. The luminescence from nc-Si can be tuned by structural and surface control from visible to uv band. The photovoltaic conversion in the short wavelength region has also been confirmed. The observed avalanche photoconduction and ballistic hot electron emission are due to a specific electron transport through chained nc-Si dots with quantized energy levels. The energetic and directional electron emission is useful in vacuum (parallel EB lithography under an active-matrix drive), in atmospheric-pressure gases (vacuum uv emission by discharge-free excitation of Xe molecules), and even in solutions (deposition of thin films by ballistic reduction). The thermally-induced ultrasound emission from nc-Si device shows no resonant peaks in the frequency response. The broad-band flat emissivity with no harmonic distortions makes it possible to use the emitter for reproducing both audible sound under a full-digital operation and complicated ultrasonic communication calls between mice.

536 Novel bioreactor-system for in-situ-cultivation of artificial tissue

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Novel micro-sized bioreactors are used to generate and maintain a physiological environment. Depending on the field of application physiological environments can comprise of different parameters. In the case of mammalian cell lines these parameters require a complex supply and monitoring system for the cultivation over a whole period. Thus we developed a bioreactor-system for long-term cultivation of different mammalian cell types imitating physiological conditions. The system included detection and control of the following parameters: temperature, pH-value, gas concentration and the continuous supply with nutrients. Enabling a high through-put cultivatingsystem, a micro fluidicnetwork was established as a bioreactor-system. The bioreactor-system consists ofseveral micro-sized chambers in a microliter scale. The chambers wereeach placed in a polymeric slidewithan individual medium supply and disposal. Every single chamber has a volume of approx. 190 μl and was perfusedby nutrients solution with a velocity of 100 $\mu\text{l/h}$.The pH-value is observed optically and controlled via CO_2 supply. All gas interchanges into every single chamber were realizedvia semi permeable membranes. The required temperature of 37°C was adjustedutilizing MOSFETsallocated on an aluminum board along the slides. Two slides each were housed in a PMMA case. Fluidic supply to all chambers was performed via appropriate tubes and syringe pumps. The bioreactor-system was put into operation successfully. All fluidic and gas interchanges worked properly. The temperature could be kept stable within a range of $\pm 0.5^\circ\text{C}$. Mouse fibroblast cells used for initial testscould be cultivated autarkical under physiological conditions for five days. Future experiments are striving for cultivation periods of some weeks.

537 Anisotropic defect recovery in HPT- and ECAP-processed ultrafine-grained Ni studied by difference dilatometry

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The attractive mechanical properties of ultrafine-grained metals prepared by severe plastic deformation are intimately related to the high fraction of lattice defects, such as grain boundaries, dislocations, and vacancies. Therefore, a thorough understanding of the thermal behavior of defects is of high importance. Difference dilatometry has proven as powerful tool to study the absolute concentration and kinetics of defects associated with open volume. This method allows the precise measurement of the volume change associated with the annealing out of these defects upon time-linear heating. The focus of our measurements is on high-purity Ni which is severely plastically deformed by high-pressure torsion (HPT) or by equal-channel angular pressing (ECAP). The annealing-induced length change of HPT- or ECAP-deformed Ni turns out to be highly anisotropic in the temperature regime between 350 K and 450 K. Normal to the shear direction, an irreversible length decrease is observed as expected due to the

annealing of defects and the associated decrease of excess volume. In contrast to that, an irreversible length increase is observed parallel to the shear direction. The aim of the present work is to connect this anisotropic behavior with the deformation parameters and the resulting microstructure, such as grain shape, grain size, dislocation structures and texture. Possible explanations for this anisotropic behavior include diffusion and anisotropic annihilation of vacancies at grain boundaries; relaxation of residual stresses; and recovery of mobile dislocations. Financial support by the FWF Austrian Science Fund is appreciated (project P25628-N20).

538 Material modelling and fracture behaviour of thin film systems

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The ongoing miniaturization of microelectronic devices requires a resizing of its integrated components. Consequently, the dimensions for each of the individual material layers forming the component are well below the micron scale by now. With a number of dissimilar materials packed into a tight assembly, the material properties will differ from those of single thin films. Additionally, nanoscale material layers are affected by the so-called size effect, leading to an increase of the yield strength with decreasing sample and/or grain size. Composite structures will always exhibit structural imperfections, which may act as crack initiation sites. The propagation of such cracks, and hence the fracture behaviour of thin film systems, is governed by the underlying material properties, including internal structural influences. An appropriate assessment of cracks is only possible if an adequate material model is established first. We numerically estimate the yield and hardening behaviour of the involved materials from spherical nanoindentations. Performing fracture experiments on multi-layered thin film systems and estimate the fracture properties is hard. With a combination of bending beam experiments and numerical investigations, we are able to determine these properties. Additionally, we are able to calculate local residual stresses, which will alter the driving forces at the crack tip. Different material combinations of copper and tungsten are investigated, and the resulting crack driving forces and fracture properties are determined. These findings provide deeper insight into the failure mechanisms of thin film systems at the nanoscale and may be readily used in the design of microelectronic devices.

539 Surface effects on L10 ordering processes in nanostructured intermetallics with magnetic anisotropy: Atomistic simulation

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Monte Carlo (MC) simulation studies of free-surface-induced selective destabilization of L1₀ superstructure variants in nanolayered FePt were extended upon nanowires and nanoparticles. The generated samples initially either perfectly ordered in the c-variant L1₀ superstructure, or

completely disordered in the fcc structure and containing 1 vacancy were relaxed with the Glauber MC algorithm implemented with vacancy mechanism for atomic migration. While the initial c-variant $L1_0$ superstructure of nanowires transformed totally to the $L1_0$ a-variant with monoatomic planes perpendicular to the wire axis, in the case of nanocubes the competition between the a- and b-variant $L1_0$ domains nucleating at the (100), (010) and (001) surfaces resulted in suppression of their growth. As a consequence, most of the cube volume remained untransformed and showed the c-variant $L1_0$ chemical long-range order (LRO) with a degree lowered by homogeneously creating antisite defects. The initially disordered samples were transformed by the creation of a specific $L1_0$ domain structure with a mosaic of particular $L1_0$ -variant domains at the surfaces and almost homogeneous long-range order in the inner volume. The analysis of correlation effects revealed that chemical ordering in the initially disordered nanosystems was initiated at the free surfaces.

540 Bulk metallic glasses composites produced via severe plastic deformation

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Bulk metallic glasses (BMGs) are a relatively new class of materials, which have been strongly investigated in the last years. An obstacle of this material class is the localized damage which reduces the ductility drastically. One idea to improve BMGs is to produce composites with two different MGs or one MG and a crystalline phase to avoid the formation of one fatal shear band. A promising route is Severe Plastic Deformation, e.g. High Pressure Torsion (HPT).

For this work specimen containing one MG (Zr-based or Ni-based) and composites with two MGs or one MG and a crystalline metal (Cu) were produced via HPT. For this process MG powders and crystalline Cu powder were mixed together and HPT was used to consolidate and deform the powders until fully dense bulk samples were achieved. The applied deformation was varied to achieve different microstructures for composites. These samples were investigated with different micromechanical testing methods, such as nanoindentation, in-situ SEM compression of micropillars, and finally in-situ TEM picoindentation to characterize the deformation behavior under ambient but also non-ambient conditions. Deformation behavior and so mechanical properties change due to the second phase. A change in hardness, Young's Modulus, fracture strength and fracture strain can be observed. Additional XRD, TEM, SEM and DSC investigation help to understand the structural evolution as the state of mixture or relaxation during the HPT-process and the formation of shear bands.

541 Influence of thermo- and HIP treatments on the microstructure and mechanical properties of IN625 alloy parts produced by selective laser melting: a comparative study

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Post-processing of strength-, corrosion- and temperature-resistant IN625 alloy parts manufactured by selective laser melting (SLM) is studied with the objective to determine the

conditions leading to an optimum combination of the alloy's mechanical properties at room and elevated temperatures. Rectangular prismatic IN625 coupons with four building orientations were SLM-built using an EOS280 system. The build platform with coupons was subjected to stress-relief annealing at 870°C during 1h under argon atmosphere. The coupons were next cut from the platform and machined to obtain dumbbell-shaped tensile testing specimens. The machined specimens were submitted to different heat treatments (HT) and hot isostatic pressing (HIP) and characterized from the microstructure and mechanical properties points of view. Mechanical testing was carried out at room temperature and at 600°C. The wrought IN625 alloy specimens subjected to the same processing and testing sequence as their SLM-built counterparts were used as a reference. It was hypothesized that low-solution annealing HT (980 and 1040°C) should reduce the anisotropy in mechanical properties inherent to the as-built alloy, whereas HIP treatment (1120°C at 100 MPa) should additionally strengthen the material by eliminating the microporosity generated during processing. It was shown that at room temperature, the as-built specimens exhibit YS ranging from 600 to 700 MPa, UTS from 900 to 1050 MPa, and elongation to failure, from 25 to 40%, depending on their orientation. The summary of the results obtained in the framework of this project will be presented in the conference.

542 Modelling yield strength in an A6061 aluminium alloy

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The yield strength of an A6061 Aluminium alloy for different artificial ageing times, temperatures and strain-rates is modelled taking into account precipitation, solid solution, grain boundary and dislocation forest strengthening. Precipitation kinetics during artificial aging and the individual strength contributions are simulated with the thermokinetic software package MatCalc. We introduce the model for the temperature and strain rate dependence of the yield-strength based on thermal activation theory. The experimental work presented here is performed on a Gleeble 1500 thermo-mechanical simulator, where the solution annealed and quenched samples are heat treated to produce samples in various microstructural conditions. We demonstrate that yield strength simulation is a powerful tool to reduce experimental effort and to cut down costs in the process of alloy engineering. This approach consistently represents the yielding behaviour of alloys in a variety of microstructural conditions with respect to the production history of the alloy and the testing conditions, i.e. temperature and strain rate.

543 Development of medium-Mn steels via batch and continuous annealing

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Medium-Mn steels exhibit an excellent combination of strength and elongation with $R_m \times A_{80}$ approaching the margin of 30'000 MPa% due to the transformation of a considerable amount of retained austenite primarily stabilized by Mn to strain-induced martensite by the TRIP effect. This steel grade can be manufactured via simple batch annealing or using stricter time-temperature schedules as required e.g. by continuous annealing. In the present contribution the

laboratory results for both processing routes with the final annealing applied to an initial non-deformed and deformed microstructure will be discussed in detail. The obtained mechanical properties, observed microstructures along with the amount and stability of retained austenite as a function of the most important annealing parameters will serve as the basis for the preparation of upcoming industrial trials at the production site of voestalpine Steel Division in Linz.

544 Microstructure evolution and creep properties of a directionally solidified ternary eutectic Mo-Si-B alloy

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The aim of the present study is to identify the ternary eutectic Mo-Si-B composition to produce directionally solidified materials, which are expected to have excellent high-temperature properties due to the well-defined microstructure. Different alloy compositions in the respective primary solidification areas of the phases were chosen to investigate the microstructural evolution. The results were compared to thermodynamic calculations of the liquidus projection and isopleth phase diagrams using the software FactSage™. By carrying out these experiments the eutectic point was found to have a nominal composition of Mo-17.5Si-8B (at.%). In the next step, the eutectic alloy was directionally solidified by a zone melting (ZM) process. The evolution of a typical eutectic structure due to the growth of lamellae is shown by microstructural investigations. Furthermore, we present a eutectic phase-field model for the Mo-Si-B alloy. The equilibrium interface geometries and interface mobility were calculated using an isotropic model. The results show that undercooling tends to affect the growing pattern and consequently the lamellar spacing significantly. High temperature mechanical properties, such as the compressive strength and creep strength at temperatures between 1100°C and 1400°C were evaluated and compared with a commonly used Ni-based superalloy and a powder metallurgically (PM) processed Mo-Si-B material. In comparison to the PM reference alloy, the creep resistance of the zone molten material was found to be substantially improved due to the relatively coarse directionally solidified microstructure. Thus, ZM alloys show great potential for applications at targeted application temperatures of around 1200 to 1300°C.

545 The 3D imaging and metrology of microstructural elements in innovative materials for clean energy systems and aeronautics

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The development of innovative materials for aeronautics and clean energy systems requires use of modern research techniques to characterize the structure on the level from micro- to nanoscale. Application of modern 3D imaging techniques such as HAADF-STEM, STEM-EDX and FIB-SEM tomography allows accurate qualitative and quantitative measurement of the structure elements in the micro- and nanoscale. The TEM tomography technique enables

obtaining 3D model of the investigated object(s) from the multiple 2D projection images, acquired over a range of viewing directions. FIB-SEM tomography is based on a serial slicing technique using a FIB-SEM dual beam workstation. Dual-beam SEM enables the acquisition of serial images with small (few nanometers) and reproducible spacing between the single imaging planes because no mechanical stage tilting is necessary between the FIB milling and the electron beam SEM imaging steps. The TEM and FIB-SEM tomography studies have been carried out for 3D visualization of morphology and spatial distribution of different structural elements in Ni-based superalloy and low-alloy creep-resistant steel. Electron tomography results provided quantitative data about shape, size and distribution of the particles, complementary to those obtained by means of quantitative TEM metallography.

546 Effect of layer-by-layer texture non-uniformity on the stress corrosion of gas steel tubes

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Steel tubes for trunk gas pipelines are obtained by hot rolling at the air, when interstitial impurities from the atmosphere have free access to the surface layer. Under such conditions the phenomenon of dynamic deformation ageing takes place; it consists in formation of interstitial atmospheres at dislocations of the surface layer. As a result these dislocations lose the ability to slip, so that the main deformation mechanism of surface layer proves the dislocation climb. Therefore the layer-by-layer texture non-uniformity develops in gas tubes under hot rolling: grains with rolling plane $\{001\}$ are dominating inside the rolled sheet, whereas grains with rolling plane $\{011\}$ – at its surface. The thickness of the surface layer depends on the temperature of rolling and its regime. Since inner layers of sheet have smaller interplanar spacing, than surface ones, the latter experiences the compressive stress due to interaction with the former. This stress prevents opening of surface cracks under conditions of stress corrosion in the soil. Layered textures of different gas tubes were compared. It was shown that in cases of defects, caused by stress corrosion, a thickness of the surface layer and the corresponding compressive stress deviate from some optimal values.

547 Effect of prior austenite grain size on yielding behavior of the low-C martensitic stainless steel

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The effect of the prior austenite grain size on the yielding behavior of as-quenched low-C martensitic stainless steel was investigated. Martensite (Fe-16%Cr-7%Ni-1.2%Mo-1.8%Cu-0.01%C alloy) samples with different prior austenite grain sizes were prepared by heat treatment for various times at 1323K. The microstructures of the martensite were observed by optical microscopy, scanning electron microscopy, electron backscatter diffraction and X-ray diffractometry. Tensile tests were undertaken at various test speeds. Also a relaxation test was performed before the tensile tests to determine the behavior of mobile dislocations. A lower

elastic limit was observed at lower test speeds and a larger prior austenite grain size. Moreover, a lower tensile strength was observed at a larger prior austenite grain size.

548 Analysis of recrystallization behavior of hot-deformed austenite reconstructed from EBSD orientation maps of lath martensite

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Ausforming generally utilizes work-hardened austenite prior to the martensitic or bainitic transformation. Therefore, it is important to control the state of work-hardened austenite to obtain the intended microstructures and properties when the ausforming process is applied. However, it is difficult to directly reveal how the recrystallization process of austenite progresses, because it is difficult to observe the high temperature austenite phase at room temperature. The recrystallization behavior of hot-deformed austenite of a 0.55% C steel at 800°C was investigated by a method of reconstructing the parent austenite orientation map from an EBSD (electron backscattering diffraction) orientation map of lath martensite. It becomes possible to evaluate various phenomena related to the hot-deformation of austenite, such as the heterogeneous deformation structure of work-hardened austenite and microstructural and textural changes with recrystallization. Recrystallized austenite grains were clearly distinguished from un-recrystallized austenite grains. Very good correlation was confirmed between the static recrystallization behavior investigated mechanically by double-hit compression tests and the change in austenite microstructure evaluated by the reconstruction method. By applying this method to double-hit compression tests at 800°C of SAE9254 with and without V addition, it was first revealed that the recrystallization proceeds along the austenite grain boundaries and in places with annealing twins, but the recrystallization is significantly retarded by the addition of 0.1% V. The strong compression texture of C.D.//<110> develops just after the hot-compression test, but this texture becomes considerably weaker as the recrystallization of austenite progresses.

549 The effects of Fe on the microstructure and the interface between hypereutectoid Cu-Al-Fe coatings and steel substrate

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Cu-Al-Fe aluminium bronzes with Al>14 wt.% are known for their wear resistance and are often employed in applications such as forming dies. However, production of these alloys using casting techniques is difficult due to the eutectoid reaction, which has been reported to cause embrittlement. In the present study, four Cu-Al-Fe coatings were produced from a novel Ce-deoxygenated gas atomized Cu-14Al-4.5Fe (all in wt.%) powders by deposition onto an E.N. 10503 steel substrate using plasma transferred arc technique. The microstructure and the interface between the coating and steel substrate were investigated using scanning electron

microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), electron backscattered diffraction (EBSD), X-ray diffraction and micro-hardness testing. The high temperature during deposition leads to dilution of the Fe into the coating by melting of the steel substrate and diffusion of Cu and Al into the Fe-rich interface layer. The results show that for a coating with high dilution of Fe, solid solution matrix microstructure was achieved with a decrease in matrix hardness. At the interface, large grains ($>40\mu\text{m}$) and protrusion of Fe-rich dendrites into the coating were observed. However, for the coating with low dilution of Fe, the interface microstructure consists of small grains ($<20\mu\text{m}$). The matrix has a martensitic microstructure with a finely distributed hard Fe_3Al intermetallic phase leading to enhanced wear resistance.

550 Direct imaging of mechanically or thermally induced grain structure changes in nanocrystalline metals

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Understanding the deformation processes in nanocrystalline metals during mechanical loading as well as during thermal annealing is a crucial step to optimize and develop these materials for practical applications. We have been developing automated crystal orientation mapping in STEM mode (ACOM-STEM) as a quantitative tool to directly image the grain structure of nanocrystalline metals including their local crystallographic orientation and optimized this approach to follow the structural changes during in-situ heating or straining. The structural evolution is analyzed to identify the active deformation processes and further compared to simulations based on the experimentally observed grain structure. We applied this approach to magnetron sputtered nanocrystalline Au and Pd films both during straining and thermal annealing observing the structural evolution in real space.

551 Effect of rare earth additions on the deformation behavior of magnesium

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Plastic deformation and work-hardening behavior of Mg-Gd and Mg-Y alloys have been studied under conditions of uniaxial tension and compression between 4K and 298K. The yield stress show strong temperature dependence and it decreases as temperature increases between 298K and 4K. The mechanisms of plastic flow are determined by the basal texture inherited during recrystallization of the cold rolled sheet. Profuse mechanical twinning has been observed under compression, resulting in abrupt texture change with strong influence on subsequent flow stress and work hardening. Under tension the plastic flow occurs predominantly by slip. The alloys exhibit pronounced asymmetry of the yield stress and work-hardening, attributed to different mechanisms operating under tension and compression. Increasing Gd and Y content results in reduction of tension-compression asymmetry. Strain Rate Sensitivity measurements reveal that at room temperature SRS parameter decreases with increasing concentration of Y and Gd, whereas deformation at 78K shows opposite trend. The presentation will provide framework for discussion of the influence of RE elements on strength and ductility in Mg-based alloys.

552 Sheet forming processes for AW-7xxx alloys: Relevant process parameters

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Medium strength aluminium alloys such as AW-5xxx and 6xxx alloys are common and proven alloys for automotive structural and body-in-white (BIW) parts in European cars. For crash relevant parts including A-pillars, B-pillars and side impact beams, a further increase in the strength to weight ratio is required to enhance the safety for passengers. Therefore, high strength AW-7xxx alloys have attracted the attention of material suppliers and part manufacturers. In the last five years, European aluminium sheet suppliers along with part manufacturers have made significant investigations into the feasibility of using AW-7xxx alloys for manufacturing crash relevant parts. In this paper a literature study into potential AW-7xxx alloys and process chains for such an application has been presented. In reviewing the various existing forming technologies an emphasis is placed on how the forming parameters (e.g. temper, temperature, forming speed etc.) influence the phase transformations and mechanical properties through the forming process chain. Finally, the important factors for selecting the most appropriate forming technology will be discussed.

553 Resolving the strength

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Ultrafine-grained (UFG) materials produced by severe plastic deformation (SPD) may show both enhanced ductility and strength and hence resolve the so called strength-ductility paradox. To gain mechanistic insights into such resolution, the effect of high-pressure torsion (HPT) on the microstructure and mechanical behavior was studied using a cast Al-7 wt. % Si alloy. As expected, the grain size decreased while the fraction of high-angle grain boundaries and microhardness increased due to HPT processing. However, tensile testing at room temperature revealed a simultaneous increase in strength and ductility compared to the as-cast sample. The samples showing simultaneous increase in strength and ductility also showed an increased contribution from grain boundary sliding (GBS), even at room temperature, which is attributed to the existence of a high fraction of high-angle and high-energy grain boundaries. It is proposed that the occurrence of moderate GBS, providing ductility, in very small size grains provides Hall-Petch strengthening and this suggests a potential combination for simultaneously achieving high strength and high ductility in SPD-processed UFG materials.

554 Influence of peak temperature during weld simulation thermal cycle on microstructure and mechanical properties in weld HAZ of a low carbon quenched and tempered steel

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This study investigates the effect of peak temperature during weld thermal cycles on the microstructure and mechanical properties of the coarse grained weld heat affected zone (CGHAZ) in a low carbon quenched and tempered steel, using Gleeble simulation technique. Different peak temperatures have been selected between 1300-600°C with heat input of 12kJ/cm. This heat input is in line with shielded metal arc welding (SMAW) process extensively used in the fabrication of engineering structure. The specimens were heated in single pass weld thermal cycle to peak temperatures (T_p) of 1300°C, 1150°C, 1000°C, 900°C, 800°C, 700°C, and 600°C and cooled to room temperature. Microstructural characterization was done with the help of light microscope and scanning electron microscope (SEM). Mechanical properties like impact toughness, notched tensile strength and hardness measurements were performed. Impact toughness of the simulated weld HAZ regions were assessed using Charpy impact testing at minus 50°C. An attempt has been made to correlate mechanical properties with microstructure. It is found in this study that the impact toughness 234J and hardness 280VHN are the best for the simulated weld HAZ specimen at the peak temperature 900°C and worst at the peak temperature 1300°C (the impact toughness 81J and hardness 362VHN). The poor impact toughness has been attributed to the presence of the blocky and connected martensite-austenite (MA) constituents along prior-austenite grain boundaries and at the interface of bainitic ferrite laths which have been found to act as a brittle phase for the initiation of the brittle fracture in weld CGHAZ.

555 Osteoconductivity of protein adsorbed titanium implants using hydrothermal treatment

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Protein adsorption is considered to have a strong influence on the biological reactions of bone-substituting materials. However, the osteoconductivity of protein adsorbed Ti surface is not completely clear. In this study, we produced the protein adsorbed Ti implants using hydro-processing. The hydrothermal treatment in the distilled water gave rise to the super-hydrophilic Ti surface and they had much protein adsorbability. Fibronectin and albumin were picked up as a protein, which was cell adhesive protein and not cell adhesive, respectively. And also, the content of the adsorbed protein was analyzed by FT-IR (ATR). The water contact angle influenced the amount of the adsorption of the protein. The osteoconductivity of the samples were evaluated by in vivo testing.

556 Observation of local structures in Mg-Zn-Y LPSO structures by scanning tunneling microscopy

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Scanning Tunneling Microscopy (STM) is a powerful surface microscopy technique for investigation of atomic and electronic structures of surfaces. So far, applications of STM to the bulk materials have been limited, because STM needs very, almost atomically, flat surface for successful observations. However, the ability of STM to image only atoms in the surface layer with very high spatial resolution makes it a unique tool to analyze the materials that have very fine and disordered structures. In this study, we have prepared atomically flat surfaces that retain the atomic arrangements found inside the LPSO structures by cleaving the samples and observed local arrangements and electronic properties of Y-Zn clusters in closed-packed layer of LPSO. We have investigated 18R type Mg₈₅Zn₆Y₉ and 10H type Mg₇₅Zn₁₀Y₁₅ alloys. To prevent oxidation, cleavage of the sample was carried out in ultra-high vacuum (better than 1×10^{-8} Pa) condition. Samples were cooled down to Liq. N₂ temperature before cleavage and STM observation. We found that the observed distribution of Y-Zn clusters in 18R type Mg₈₅Zn₆Y₉ alloy can be explained by “coarse graining model” in which each cluster is treated as a particle [1]. Mg₇₅Zn₁₀Y₁₅ alloy shows well-ordered Y-Zn clusters. We have carried out STS (Scanning Tunneling Spectroscopy) and found that there are a few types of cluster. We will give an interpretation of the results. [1] H. Kimizuka, S. Kurokawa, A. Yamaguchi, A. Sakai & S. Ogata, Scientific Reports 4, Art. 7318 (2014).

557 Effect of prior strain on damping capacity and mechanical property during heat treatment

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Many researchers have consistently studied for texture control and grain refinement in order to improve room temperature formability, however the study on specific properties of magnesium is still inadequate. Therefore, it is necessary to study on specific properties of magnesium. In previous study, relationship between damping capacity, mechanical property and texture in 30% rolled magnesium alloy AZ31 was studied. It was found that damping capacity has been affected by grain size, crystal orientation and so on. It is necessary to study damping capacity under various deformation conditions in order to investigate damping capacity in detail. Therefore, in this study, damping capacity is investigated in different prior strain and heat treatment condition. Magnesium alloy AZ31 was rolled at 673K with rolling reduction of 10%, 30% and 50%, respectively. Damping capacity test specimens were machined out parallel to the rolled direction. These specimens were annealed at 573K for 10-120 minutes. Then, damping capacity was measured by using internal friction measurement machine, and microstructure was examined by using optical microscopy. Hardness was measured by Vickers hardness tester under a condition of 0.3N. In all kinds of specimens, similar tendency is shown. With increasing of heat treatment time, grain size increases, whereas hardness decreases. Also, in the case of damping capacity, 10% rolled specimen shows the largest damping capacity than 30% and 50% rolled specimens. This means that prior strain is affected on damping capacity.

558 Micro-tensile testing of single block structures of lath martensitic steel

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Micro-tensile testing was employed to elucidate the anisotropic plasticity of the single block structures in a lath martensitic steel. The material used was an as-quenched low-alloy steel. Micrometre-sized specimens with gauge section dimensions of $\sim 14\mu\text{m} \times 14\mu\text{m} \times 35\mu\text{m}$ were fabricated by focused ion beam. The lath martensite variants were determined using electron backscatter diffraction pattern analysis. Two specimens were prepared: the habit plane of the lath martensite was arranged for the maximum shear stress plane for specimen A and parallel to the loading direction for specimen B. Micro-tensile tests were performed at a loading rate of $0.1 \mu\text{m/s}$ at room temperature in laboratory atmospheric air. In the specimen A, yielding occurred at a stress of $\sim 810 \text{ MPa}$ through the activation of the in-habit-plane slip system. The yield stress for the specimen B was $\sim 940 \text{ MPa}$. The out-of-habit-plane (112) slip system with the highest Schmid factor was operative in the specimen B. The critical resolved shear stress (CRSS) values for the in-habit-plane and out-of-habit-plane slip systems were determined to be ~ 380 and $\sim 460 \text{ MPa}$, respectively. This indicates that the dependence of plasticity on the habit plane orientation stems from microstructural elements within the block structure. Also, the apparent CRSS value for the out-of-habit-plane slip system including the block boundary effect was determined to be $\sim 530 \text{ MPa}$ using a single packet specimen. Therefore, the contribution of the block boundary to the strengthening of the lath martensite is minor when compared to the microstructural anisotropy underlying in the block structure.

559 Effect of cavity volume on deformation behavior of tailored step cast Al ingot

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Recently, tailored step cast method was invented, in which melt would be supplied periodically at the solid-liquid interface just after solidification like in continuous casting. This process allowed the hard to cast wrought Al alloys to be cast in a given shape like cast Al ones. However, cavity seemed to be often left liquid-solid interface was controlled improperly. In the present study, deformation behavior of tailored step cast Al alloys has been investigated with the variation of cavity volume. For this purpose, several ingots with different cavity volume were cast. And a series of hot forging tests was carried out to close cavities and tensile tested. The results showed that the initial cavity volume was the most important feature among the other variables affecting the tensile properties of tailored step cast Al alloys.

560 Formability enhancement of Al sheets with two step forming

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In the present study, two step forming comprised of mechanical and gas blow forming was investigated to fabricate automotive part having a complicated shape using Al sheets such as 5052 and 7075 Al with a conventional formability which has been known to be quite lower compared to deep drawing steels. Usual Al sheet forming consists of several steps of forming, sometimes with the use of heated dies. Also, superplastic gas blow forming has been applied to form complicated shape with fine grained sheet. However, superplastic blow forming with slow production cycle cannot be an adequate solution for large volume productions like automotive industry. Also, conventional Al alloy sheets with large and pan-caked grain structure cannot be a solution for gas blow forming that requires low flow stress level at the elevated temperatures. We have designed the two step forming in which Al sheet was drawn to a kind of preform step following gas blow forming for accurate geometry. In order to judge a formability enhancement of Al sheet in terms of forming process, model geometry came from a practical automotive part which had quite depth with complicated curvatures. The optimum forming conditions for respective forming steps were considered most important technical features of this process and would be discussed in details. Also, the effort to avoid detrimental microstructure evolutions was given and discussed for a practical application.

561 The role of grain boundary character on the hydrogen embrittlement of high-Mn TWIP steels

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In this research, sensitivity to intergranular hydrogen embrittlement (HE) depending on a character of grain boundary was studied in austenitic twinning-induced plasticity (TWIP) steels. To characterize the grain boundary structure, the coincidence site lattice (CSL) concept was employed. Each grain boundary was categorized into special boundaries sharing a high fraction of lattice sites between two grains in a grain boundary or random boundaries. Hydrogen was introduced into the samples with an electro-chemical method, and then a uniaxial tensile test was conducted to a strain of 0.1 and 0.2. After the tensile test, the microstructure was observed with electron back scattered diffraction (EBSD) technique to investigate the relationship between failure of grain boundaries during the deformation and their interfacial characters, orientations, and deformation modes. Therefore, this study can propose the optimum boundary structures presenting a high resistance to HE and be applied in grain boundary engineering process, in which the grain boundary character was controlled by special thermo-mechanical processes.

562 Synthesis kinetics, stability and local order of amorphous $\text{La}_2\text{Mo}_2\text{O}_7$ -d, a potential SOFC anode material

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A reduced amorphous derivative of oxide-ion conductor $\text{La}_2\text{Mo}_2\text{O}_9$ has been proposed as efficient, sulfur-tolerant anode material for Solid Oxide Fuel Cell devices [1], and its stability tested. This amorphous oxide is usually obtained by annealing, at or below 800°C , a powder sample of the parent compound in a flow of dilute hydrogen. The reduction kinetics has been studied, by thermogravimetric analyses (TGA) at different temperatures and X-ray diffraction (XRD), on samples with different specific surface areas prepared by solid state reaction and soft chemistry routes. Reduction takes place in two stages with distinct reduction rates; it has been successfully modeled using a shrinking core type law previously proposed by Bessières et al. [2] to model iron oxide reduction. When reduction goes on, molybdenum exsolution is observed. The stability of amorphous $\text{La}_2\text{Mo}_2\text{O}_7$ -d seems to be different under flowing reducing gas and under fuel cell operating conditions. The oxygen stoichiometry of the amorphous phase may vary in a relatively wide range, which has been studied by TGA in flowing reducing and oxidizing conditions up to various temperatures, and by XRD. Attempts to determine the electronic conductivity of amorphous $\text{La}_2\text{Mo}_2\text{O}_7$ -d have been made by four-probe measurements, and also to characterize its local order using XRD, X-ray absorption spectroscopy and electron paramagnetic resonance. [1] X.C. Lu, J.H. Zhu, J. Electrochem. Soc. 155 (2008) B1053. [2] J. Bessières, A. Bessières, J.J. Heizmann, Int. J. Hydrogen Energy 5 (1980) 585.

563 Decoration and doping of graphene by RF sputtering and atomic layer deposition processes

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Recently graphene and graphene-based hybrid materials have attracted much attention due to unique chemical and physical properties and a wide range of applications such as photovoltaics, catalysis, optoelectronics, electronics, energy and hydrogen storage. To develop an appropriate understanding of the properties of these hybrid materials, it is necessary to address the material preparation, structural and defect issues as well as their influence on the performance. This work will provide a review on the several methods which have been used for the production of hybrid materials based on graphene. In particular, it will deal with graphene-based materials doping and intercalation processes by low pressure plasma route and atomic layer deposition, the materials chemical and electronic properties as well as their defect control. Transition metals and their oxides will be considered for graphene-based materials decoration. The possibility of a combination of electronic properties and structural disorder in doped-graphene for application in the energy sector will be presented. The effect of the deposition process parameters on the material electron properties will be investigated as well as correlation with the hybrid materials performance in energy applications like hydrogen storage and catalysis.

564 Welding of automotive aluminium alloys by laser wobbling process

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Laser welding is the automotive production technology that promises better features in terms of quality and productivity in many instances. Some of the challenges in laser welding of the aluminium alloys are associated with reflectivity, hot cracking and porosity susceptibility, which can be overcome by laser welding processes deploying innovative beam wobbling heads. In fact, the key hole rotation with the consequent mixing of the molten bath, can deliver a reduction of the porosity. Furthermore, the addition of the right amount of filler wire allows for a Mg content out of hot cracking range in fused zone. Both these features significantly improve welds quality. The scope of this paper is to examine the improvement that this new welding method brings in welding tailored blanks parts, widely used in automotive to develop different stiffness aluminium components. For this purpose, butt joints and overlapping joints were produced with sheets made out of two industrial grades, i.e. AA 6082 T6 and AA 5754 H111. The technique was evaluated with or without the use of filler wire (AA 5556). The qualification of the welding process encompassed Non Destructive Testing (NDT) and mechanical testing. Results indicate that butt joints fail in the base material of the minor thickness sheet. Instead, for the shear test of the lap joint the rupture occurs in the ZTA of the thin sheet. The wobbling combined with other optimized welding parameters allows avoiding porosity. For lap joints a residual porosity, dependant on the fused zone dimensions, was always detected.

565 Continuous high pressure tube shearing process

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Thin walled metallic tubes are used in several medical device applications. These devices require the specific mechanical properties which could vary from inside of the tube to the outer surface. The process of High Pressure Tube Shearing (HPTS) developed by the authors is relevant to the group of severe plastic deformation techniques. It enhances the strength of metallic tube walls (i) in bulk, i.e. throughout the wall thickness, or (ii) only in near-surface regions, with possibility to regulate the thickness of the layer or to produce the specific gradient in microstructure. The severe shear strain is imposed within the thickness of the tube wall due to the difference in magnitude of the material flow velocities at the inner and outer surfaces of the tube, which results from the difference in rotational speed of the mandrel and the confining die. Concurrently, a high hydrostatic pressure is produced by means of a special geometry of the mandrel designed to reduce the tube wall thickness. The combined severe shear strain and high hydrostatic pressure are applied within a localized zone, which moves progressively along the tube. This type of deformation processing results in a significant grain refinement of the tube material leading to a pronounced increase in strength. It was demonstrated on three types of tube material.

566 Laser machining of ceramic electrolytes for solid oxide fuel cell applications

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Laser engraving is being reported as a very suitable technique for achieving cost-efficient production of high-quality ceramic parts. Equipments consisting of infrared, green or ultraviolet laser sources, together with a galvanometric computer-controlled head, are now affordable and more frequently used in industry. Therefore, we have used laser machining to modify the shape and microstructure of ceramic plates for solid oxide electrolytes or gas separation membranes. On the one hand, we have prepared self-supporting thin stabilized zirconia (SZ) ceramic electrolytes. They are carved from a sintered SZ plate to shape a 20 μm thick and 8 mm in diameter central region, surrounded by an unprocessed 150 μm thick supporting zone. These self-supporting ceramic membranes can be used to prepare electrode-supported solid oxide fuel cells that can operate at low temperature, due to the reduced thickness of the electrolyte. On the other hand, we have used pulsed laser ablation to imprint in ceramic electrolytes surface roughness on the level of some microns. In this way, we can increase the electrolyte-electrode contact area and, as a result, to decrease the polarization resistance of the electrochemical cells. In this communication we describe the material preparation procedure by laser machining, as well as the structural, microstructural and mechanical characterization by several physical, spectroscopic and diffraction techniques. Finally, single cells have been also studied by electrochemical impedance spectroscopy to ascertain the improvement in electric resistance caused by the laser machining.

567 High strength - high conductivity carbon nanotube - copper composite wires prepared by spark plasma sintering and room-temperature wire drawing

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Conducting copper wires are ubiquitous in today's world and there is a demand for stronger yet lighter ones, in fields such as aeronautics, space and power transportation as well as in niche applications such as materials for high-field magnets. Strengthening copper is achieved by alloying, by introducing another phase or through grain refinement, but these methods also usually increase scattering of the conducting electrons, i.e. they decrease the electrical conductivity. Tensile tests performed on macro- or microscopic dog-bone carbon nanotube - copper (CNT-Cu) composites showed that the presence of CNTs strengthens copper, but strongly decrease the electrical conductivity, which could in part reflect excessive carbon contents (typically 5-20 vol.%) in the samples. Here, CNT-Cu wires with very low carbon contents (≤ 1 vol.%) are prepared by a combination of spark plasma sintering (SPS) and room-temperature wire-drawing. The microstructure, microhardness, tensile strength at 293K and 77K, electrical resistivity at 293K and 77K of the CNT-Cu wire will be described and compared to those of pure Cu wires prepared by the same route.

568 Effect of grain refinement and nanosized precipitates on the self-organized dynamics of dislocations in Al-Mg alloys

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Plastic deformation of alloys often displays the Portevin-Le Chatelier (PLC) effect – a plastic instability which manifests itself through stress serrations and traces on the specimen surface caused by strain-rate bursts within localized deformation bands. While the investigation of the PLC effect is important for practical application of alloys because of its detrimental effects on their formability, it also presents a striking example of self-organization of dislocations, displaying a vast spectrum of behaviors, e.g., a transition from static deformation bands to band propagation, dynamical chaos, synchronization, or avalanche-like behavior. In spite of this complexity, most investigations revealed some similar features which allowed for a qualitative classification of observed behaviors. The transition to a submicrometer or nanometer scale structure through grain refinement or addition of nanosized particles gave rise to new kinds of behavior which are not yet understood. In particular, contradictory results have been obtained in the recent studies on ultra-fine grained materials. Both intensification and suppression of the PLC effect was observed depending on the composition and microstructure of the material. At the same time, nanoscale precipitation in coarse-grained Al-Mg alloy may results in a qualitative change in the kinematics of the deformation bands. In the present work, the PLC effect is studied in an Al-Mg alloy containing $\text{Al}_3(\text{Sc}, \text{Zr})$ nanoscale particles, both in the coarse-grained state and after ECAP. The talk will briefly outline some milestones of the studies of self-organization in plasticity and present the new results obtained for the materials containing nanostructural elements.

569 Multiscale study of heterogeneity and intermittence of plastic deformation of commercially pure titanium

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Investigations of crystal plasticity during last two decades proved that plastic deformation is intrinsically heterogeneous on a mesoscopic scale controlled by self-organization phenomena in the dislocation system. It is natural to suggest that such heterogeneity may be rather strong in hexagonal materials because of their strong anisotropy and combination of different microscopic mechanisms such as dislocation glide and twinning. For example, the macroscopic heterogeneity of plastic flow of hexagonal materials manifests itself through non monotonic three-stage evolution of the work-hardening rate. At the same time, recent local extensometry studies showed relatively uniform local strain distributions during tension of a commercially pure α -Ti [1]. These results require a development of multiscale approaches to the problem of heterogeneity of plastic flow. In the present work, such a multiscale approach to investigation of plastic deformation of polycrystalline Ti was based on the combination of several experimental techniques which provide simultaneous recording of conventional tensile

deformation curves, local strains along the specimen axis, and acoustic emission. The studied samples were cut along and normal to the rolling direction, taking into account the anisotropic texture resulting from rolling of titanium. The measured signals were processed using methods of the theory of nonlinear dynamical systems, widely applied to characterize the complexity resulting from self-organization. 1. A. Roth, M.A. Lebydokin, T.A. Lebedkina, J.S. Lecomte, T. Richeton, K.E.K. Amouzou, Mater. Sci. Eng. A 596 (2014) 236–243.

570 Corrosion of MgCa alloys with conversion coatings

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Magnesium plays the fundamental role in the biological system as a component of bone tissue. The yield module and the compressive strength of magnesium with calcium is the closest to the properties of natural bone as compared to other commonly used implants. These alloys are biodegradable in aqueous solutions containing in particular chloride ions.

Conversion coatings are used to slow the degradation of the magnesium alloys and to enhance the adhesion of the final polymer coating to metallic substrate. Studied conversion coating contains the elements, such as fluorine and potassium, which may have a positive effect on the regeneration of damaged bones. The corrosion process of magnesium alloys which containing 1% wt., 2.5% wt. and 5% wt. Ca was examined in as-cast condition and with conversion coatings. This material was subjected to the corrosion test in Ringer solution in order to determine the dissolution kinetics of the alloys at 37°C. The progress of corrosion was investigated in the structure observations and as mass loss. The increase of Ca up to 5% in Mg alloys leads to a very rapid biodegradation of the alloy which may have an impact on the process will be very sensitive to small changes in conditions such example as: pH or temperature. Conversion coatings (thickness at 13 µm) have been produced from a KOH + KF solution of at 5 ° C. Then, a similar study in the reagent Ringers "and in order to determine the corrosion rate of the coating material were carried out.

571 Deformation anisotropy and associated mechanisms in rolling textured high purity titanium

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Deformation anisotropy and associated mechanisms in rolling textured high purity titanium

Jong Woo Won ^a, Seong-Gu Hong ^b, Chong Soo Lee ^{a, c},

^a Department of Materials Science and Engineering, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea. ^b Division of Industrial Metrology, Korea Research Institute of Standards and Science, Daejeon 305-340, Republic of Korea. ^c Graduate Institute of Ferrous Technology, Pohang University of Science and Technology, Pohang 790-784, Republic of Korea. Plastic anisotropy and associated deformation mechanisms of rolling textured alpha phase titanium were investigated. Uniaxial compression tests were conducted along the three featured directions, the rolling direction (RD), normal direction (ND) and transverse direction (TD), in combination with an EBSD measurement and a Schmid factor analysis. The results revealed that the specific crystallographic feature of the material, caused by rolling texture, influences the activities of dislocation slips and twinning, and this

significantly varies with the loading direction, consequently leading to anisotropic deformation. The material yielding was dominated by dislocation slips, and the change of slip system with the loading direction (prismatic $\langle a \rangle$ slip for the RD and TD, and basal $\langle a \rangle$ slip for the ND) gave rise to yielding anisotropy. As the material deformed beyond the yielding point, deformation twins took place and played a decisive role in the deformation. The twinning characteristics significantly varied with the loading direction, and this had a completely different effect on strain hardening behavior, thereby causing deformation anisotropy.
Keywords: Titanium; Anisotropy; Texture; Sslip; Twinning; Schmid factor

572 Precipitation and recrystallization interaction in Nb microalloyed steels

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It is well known that softening stasis is often observed in microalloyed steels. The precipitation and coarsening behaviors are deeply involved in the softening stasis. However, the determination of start and finish of stasis is far from satisfactory since the precise quantitative analysis of the size and the distribution of nano-size precipitates is quite difficult. Lots of models such as Avrami type equation, classical nucleation and growth model, and mesoscale model are reported for nucleation and precipitation kinetics, which are needed to be more self-consistency. In this study, models as well as experimental evidence were reviewed in order to clarify the softening stasis. The precipitation and recrystallization interaction and parameters used in models are reevaluated. The results are compared with the measured.

573 Evolution of recrystallization textures in cold-rolled commercially pure aluminum

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The evolution of recrystallization texture in commercially pure aluminum sheet cold-rolled by 90% reduction in thickness was measured by Juul Jensen et al. The cold-rolling texture consisted of the Goss $\{110\}\langle 001 \rangle$, brass $\{110\}\langle 112 \rangle$, S $\{123\}\langle 634 \rangle$, and copper $\{112\}\langle 111 \rangle$ components. When the cold-rolled aluminum sheet was annealed at temperatures between 253 and 341°C for times between 5 min and 20 h., the cube $\{001\}\langle 110 \rangle$ component evolved. The evolution of the cube texture cannot be explained by either the oriented nucleation theory by Burgers and Louwerse or the oriented growth theory by Barrett. The cube texture evolution originates from the copper component by the strain-energy-release-maximization (SERM) theory by Lee. Once the cube oriented, dislocation free nuclei evolve, they are in the best position to grow at the expense of neighboring deformed high energy grains of the Goss, brass, S, and copper orientations, and the volume fractions of the Goss, brass, S, and copper components would decrease. However, the volume fraction of the Goss component increased a little at annealing temperatures of 253 and 278°C, at variance with expectation. Low stacking-fault-energy alloys with the brass $\{110\}\langle 112 \rangle$ rolling texture evolve the $\{236\}\langle 385 \rangle$ texture after recrystallization, whereas high stacking- fault-energy alloys with the brass rolling texture evolve the Goss texture after recrystallization by the SERM theory, resulting in the increase of

the volume fraction of the Goss texture.

574 Synthesis of a metal matrix nanocomposite through high-pressure torsion

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High-pressure torsion (HPT) is one of the major severe plastic deformation (SPD) procedures where disk metals generally achieve exceptional grain refinement at ambient temperatures. To date, HPT has been applied for the consolidation of metallic powders and bonding of machining chips whereas only very limited reports have examined the application of HPT for the fabrication of nanocomposites. An investigation was initiated to evaluate the potential for the formation of a metal matrix nanocomposite by processing two commercial metal disks of Al-1050 and ZK60 magnesium alloy through HPT at room temperature. Examinations were conducted to evaluate the evolution of microstructure and mechanical properties with increasing numbers of HPT turns up to 20. This study demonstrates the possibility for using HPT to fabricate a wide range of hybrid nanocomposites.

575 Effect of CH₄ content on the characteristics of surface layers of low temperature plasma nitrided 2205 duplex stainless steel

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Plasma nitriding was performed on the 2205 duplex stainless steel samples at 400 V with a gas mixture of H₂ and N₂ for 15 hrs with changing N₂ percent, temperature and adding various amounts of CH₄. After treatment the behavior of the surface layer was investigated by optical microscopy, X-ray diffraction, GDOES analysis and micro-hardness testing. Potentiodynamic polarization test was also used to evaluate the corrosion resistance of the samples. With increasing both N₂ percentage from 10% to 25% and nitriding temperature from 370°C to 430°C, the thickness of nitrogen expanded austenite (S-phase) layer and surface hardness increase up to 16 µm and 1200 HV_{0.1} at the treatment temperature of 430°C with 25% N₂, but decreases the corrosion resistance due to the formation of Cr₂N and (Fe, Cr)₄N. Thus in order to further increase the thickness of S-phase layer and the corrosion resistance, the influence of adding various amount of CH₄ (1% to 5%) in the nitriding atmosphere was investigated. Adding CH₄ in the nitriding atmosphere increases the layer thickness compared with that of nitrided sample. The highest thickness can be obtained at 1 % CH₄, but addition of CH₄ beyond 1 % slightly decreases the layer thickness. Moreover, when nitrided at 400°C with 10% N₂ and 5% CH₄ content, best corrosion behavior is obtained which also have around 10 µm layer thickness and about 870 HV_{0.1} surface hardness.

576 Texture modification of warm-deformed Mg-Zn based alloy by micro-alloying

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Although it was revealed that Mg-Zn based alloy is a promising magnesium alloy to weaken deformation texture by solute drag effect, a methodical study of the effect of alloying addition on texture modification/randomization in Mg-Zn alloy has been not performed yet. In this study, the recrystallization behavior and resultant texture evolution of micro-alloyed Mg-Zn based alloy were investigated during warm-deformation and subsequent annealing by in situ scanning electron microscopy and electron backscatter diffraction observations. Based on analysis results, attractive alloying elements effective for randomizing texture of magnesium alloys were suggested.

577 A Study on the Metallurgical Factors for Development of Creep Resistance of Alloy 617 at 950 °C

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In this paper, the heat-treatment for grain boundary serration in Alloy 617 was introduced as one of the effective method to strengthen the GBs during creep at very high temperature. The proprietary direct aging combined with controlled cooling rate led successfully to the transition of serrated GBs in Alloy 617. The lower final aging temperature produced higher amplitude and higher fraction of serrated GBs. After the creep test under 950°C/30MPa condition, the serrated specimen had about 2 times longer creep life than standard specimen. Moreover, when the serrated specimen was subjected to 5% cold deformation before creep (referred to as 'pre-strained/serrated specimen', hereinafter), the creep life could be extended to be 2.8 times longer as compared with standard specimen. The longitudinal section of the standard specimen after creep rupture showed a typical sharp crack running and propagating along GB perpendicular to the loading axis in standard specimen. However, the serrated GBs (both serrated and pre-strained/serrated specimens) were highly resistant to GB cracking, resulting in an isolated GB crack with low frequency. It should be also noted that the pre-strained/serrated specimen contained not only coarsened primary carbides but also fine secondary carbides distributed uniformly in γ matrix. The evolution of the nano-sized MC carbides as well as $M_{23}C_6$ carbides along the serrated GBs has been investigated during creep, which was discussed with their dislocation interaction and GB sliding to correlate with creep life.

578 Effect of cryomilling on the microstructure, high temperature compression and creep properties of oxide dispersion strengthened steel

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This study investigated the effect of cryomilling on the microstructures and high temperature mechanical properties of oxide dispersion-strengthened steel. Cryomilling was newly tried on this ODS steel to control grains, oxides, and dislocation microstructures. Fe-14Cr-3W-0.4Ti (wt.%) alloy powder and 0.3wt.% Y₂O₃ powder were mixed and were mechanically alloyed (MA) through ball milling at each of room temperature (RT) and cryogenic temperature (-150°C) and then hot isostatic pressing (HIP), hot rolling, and annealing processes were implemented to manufacture two types of ODS ferritic steel, K1 (RT) and K4 (-150°C). The grain size of K1 was 0.5~1.5µm and K4 was 300nm on average. Oxide particles were shown to be finer and more uniformly distributed in K4 (5~10nm size distribution) than in K1 (average size 30nm). The two alloys were subjected to high temperature compression (RT ~ 900°C) and creep (650°C) tests. K4 represented higher yield strength under all temperature conditions. However, K4 showed rapid strength decreases at high temperatures exceeding 700°C and showed similar levels of strengths to K1 at 900°C. In the case of creep properties, steady-state creep rates of K4 were lower than K1 in the same stress range. However, the degree of improvement of creep properties by cryomilling was not great compared with the improved strength levels. Stress exponent was almost similar values K1 (n=4.15) and K4 (n=4). [supported by the Program for the Development of Core Defense Materials, Korea]

579 An approach to the deformation of thick steel plates by high-frequency induction heating

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In this study, automatic HF induction-based line and triangular heating processes were introduced in order to investigate the influence of process parameters on the three-dimensional temperature distribution and subsequent deformation behaviour of an SS400 thick plate. First both three-dimensional electromagnetic-thermal and thermal-structural finite element simulations were carried out by using a commercial software package program of ANSYS. Actual experimental line heating was carried out by applying newly designed laboratory-scale HF IH quipment capable of bending thick plate with the moving accuracy of ±0.1 mm in heating coil position. For the case of line heating, the drastic increase of temperature gradient upon increasing input power could mainly be predicted, which attributes to the amount of permanent bending deformation of the thick plate. Furthermore, both the depth and width of heat affected zone tend to increase with the increase in the input power. After plotting the amount of vertical deformation as a function of various positions from top surface of the plate, we can deduce following conclusion; the higher input power, the more thermomechanical deformation can be generated regardless of both concave- and saddle-type doubly curved plates. Grain structure within the heat-affected zone became equiaxed and finer, implying that the amount of heat input onto the upper surface of the workpiece was large enough to cause recrystallization. For the

case of triangular heating, there is another important factor to determine final curvature of the plate: proper design of triangular heating pattern.

580 High temperature deformation and dynamic recrystallization behaviour of AlCoCrFeNiTi_x high entropy alloys

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Recently, alloy design strategy of high entropy alloys (HEAs) starting from equimolar quinary AlCoCrFeNi has attracted great attention due to their excellent high compressive strength at room temperature together with corrosion and wear resistance at high temperature. Especially, the effect of Ti alloying with AlCoCrFeNi HEA on the microstructure and mechanical properties has been studied. This alloy was easily fabricated by simple copper mold casting as a bulk-type specimen. On the other hand, as-cast HEAs typically show an easy tendency to brittle shear, columnar or cone and split fracture with less plasticity at ambient temperature. This implies that it is essential to improve the mechanical performance by deducing feasible thermomechanically controlled process (TMCP) conditions. We have, therefore, attempted in this study to investigate on the high temperature deformation behavior of AlCoCrFeNiTi_x HEAs ($x=0, 0.025$) by the aid of a series of compression tests with high-speed heating apparatus. As-cast rods are composed mainly of BCC solid solution. By means of high-temperature compression between 700 °C and 1100 °C, on the other hand, more than two-phase structure could be observed and then partial recrystallization proceeded with increasing the compressive temperature. The influence of multi-phase structure and recrystallization on the variation of mechanical performance such as microhardness and room-temperature strength was also discussed.

581 A study on the microstructural evolution of low and medium Mn TRIP steel during annealing process

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The regulation of CO₂ emission to preserve or sustain the global environment and the safety regulations for improving passenger safety have been more and more strengthened. These enhanced regulations lead to the wide adoption of AHSS(Advanced High Strength Steel) and light weight materials to meet the regulations. In particular, Hot Press Forming steel is expected to be adopted more and more despite the higher cost. To satisfy the demand on the cost-effective and eco-friendly steel sheets, cold formable next generation AHSS such as eXtra-AHSS displaying the enhanced formability comparing with conventional advanced high strength steels has been developed in order to replace or compete with hot press formed parts with higher cost. To control the stability and the volume fraction of retained austenite is significant for realizing eXtra-AHSS property. Ultra-fine grained microstructure was obtained for low and medium Mn TRIP steel, which led to the enhanced yield strength and elongation. Understanding and controlling microstructural evolution during annealing process is of importance since the stability depends on chemical composition and grain size as well.

582 Deep ultraviolet photodetector based on sulphur-doped cubic boron nitride thin film

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Cubic boron nitride (c-BN) is a wide bandgap III-V compound semiconductor potentially useful for solar-blind photodetectors. This paper describes work on the use of Sulphur doping to adjust the bandgap of c-BN films prepared by plasma-enhanced chemical vapor deposition (PECVD). An S-doped c-BN film based metal-semiconductor-metal (MSM) solar-blind ultraviolet (SBUV) photodetector was successfully fabricated and its electro-optical properties were characterized. The photocurrent shows peak responsivity at 254nm with sharp cutoff wavelengths at 220 and 300 nm, respectively, which is appropriate for use in solar-blind detection. The maximum response reached 1.55×10^{-7} A/W/cm² with a rejection ratio of more than three orders of magnitude. The high solar-blind region UV response could be attributed to the successful substitution of boron by Sulphur and the suppression of B vacancies. The experimental results show the same peak in response at around 254nm as is found in the theoretical analysis.

583 Near-threshold fatigue crack growth behavior of ultrafine-grained metals

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Severe plastic deformation (SPD) techniques allow the production of ultrafine-grained metals, which exhibit a strong increase in strength. However, the influence of grain refinement on the crack propagation behavior is still not fully understood. Therefore the fatigue crack growth characteristics of various metals with grain sizes ranging from the micro- down to the nanocrystalline regime were examined. Fatigue crack growth measurements at different load-ratios and extensive fracture surface analyses were carried out in order to gain further knowledge about crack closure and damage mechanisms. Different metals were compared to see the influence of crystal structure and Young's modulus. Along with the grain size variation the influence of the grain aspect ratio was in focus, which can cause a strong orientation dependence of the fatigue crack growth behavior. Elongated microstructures aligned perpendicular to the crack growth direction can considerably improve the overall resistance against fatigue crack growth.

584 Phosphorus at grain boundaries of iron and steels: An overview

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Presence of trace elements in metallic materials may significantly affect their macroscopic properties. The trace elements segregate at grain boundaries and change local bonding there

which further results e.g. in reduction of the cohesion of the grain boundaries and suppression of their migration: the latter effect is often utilized in grain size stabilization in nanocrystalline materials. The effect of phosphorus segregation in iron and steels is probably the most frequently studied problem in this respect.

In this contribution, available literature data on thermodynamic parameters of phosphorus segregation at grain boundaries obtained both theoretically and experimentally, are summarized. Their comparison is done and discussed from the point of view of solid solubility, anisotropy of interfacial segregation, and position of phosphorus atom at the grain boundary. The change of the grain boundary cohesion induced by interfacial segregation is also mentioned. This comparison results in new aspects of grain boundary segregation of phosphorus which can also be generalized for other impurities.

585 Release of polymer additives from pharmaceutical packaging studied by an original UHPLC-ESI-MS/MS and ToF-SIMS approach

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Polymers are increasingly used for packaging of pharmaceutical products in relation with interesting functional properties as lightweight, recyclable and processable materials. However, this packaging should not be a source of contamination to the content. It is then important to study extractables and leachables from the polymer packaging. In this context, an original approach is proposed by taking advantage of combining a highly sensitive surface analysis technique (Time-of-Flight Secondary Ion Mass Spectrometry - ToF-SIMS) with a multiresidue trace analysis method based on Ultra-High Performance Liquid Chromatography Tandem Mass Spectrometry (UHPLC-MS/MS). It is also proposed to take into account a very high number of possible detected molecules, i.e. authorized polymer additives European Pharmacopoeia but also some frequently used other polymer additives and some of degradation products, corresponding to a total set of 25 molecules. Development methodologies including optimization of the extraction conditions by solid phase extraction (SPE) and comparison of two ionization interfaces for UHPLC-MS/MS as well as ToF-SIMS data treatment will be discussed. It will be in particular illustrated that UHPLC-MS/MS data quantification was obtained when the concentration was superior to the method quantification limits of each substance and that ToF-SIMS statistical data treatment made possible to define two levels of identification (additive considered as intrinsic to the polymer vs additive considered as a contamination possibly related to the fabrication process). To further substantiate the validation of this original approach, data acquired on six different polymers used for pharmaceutical packaging applications will be compared, highlighting potential artifacts of the approach.

586 Atomic layer deposited protective layers

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Atomic Layer Deposition is a chemical vapor thin film deposition technique where precursors are pulsed one at the time onto the substrate surface. The precursors react saturatively on the surface and the film grows layer by layer. Because of the growth principle ALD allows the growth of dense conformal films on substrates of different size and shape. ALD is industrially used in microelectronics but emerging application areas are energy technology and protective coatings. The thicknesses of ALD films are usually from few nm to few hundred of nm. In protective coatings oxides are the most common materials and in ALD especially Al, Ti, Ta and Zn oxides have been applied. Since ALD films are pinhole-free and often thin they are used to protect against moisture, radiation, out-gassing but not often against corrosion. Very good moisture barriers are obtained with thin ALD oxide layers on polymers and cardboard. This property is very attractive in encapsulation of OLEDs. In energy technology sector protection of electrodes in Li-ion batteries, fuel cells and supercapacitors by ALD has been reported. Yet another area is protection of silver jewelry from tarnishing by a thin oxide layer. In traditional corrosion protection ALD films have proven to be useful in tailoring of interfaces and sealing of defects in coatings made by other techniques. In the presentation the use of ALD in the above mentioned protection applications is highlighted.

587 The study of the microstructure of the metal after rolling thick workpieces of non-ferrous metals and alloys in relief and smooth rolls

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One of the ways to improve the quality of steel production by rolling is the creation of additional streams of the metal flow, i.e. intensification of shear deformation not only in longitudinal but also in transverse direction. From the review of scientific-technical and patent literature it is known that to improve the quality of products when rolling usually use a system of alternating projections and depressions, which is created on the surface of the wide faces of slab or on work rolls. In this case, along with deformation in altitudinal and longitudinal directions, the conditions for deformation of the metal in the transverse direction, which has a positive effect on reducing the anisotropy of properties. Knowing the advantages of rolling with additional shear deformations, we have developed energy-saving technology of plate billets rolling of non-ferrous metals and alloys, allowing to obtain a uniform fine-grained structure throughout the volume of the workpiece, and designed two new constructions of the pair of work rolls with embossed surface as an annular groove, forming a trapezoidal protrusions and depressions alternating each other along the entire length of the barrel (the first pair to unequal treatment of the ledge to the depression and the second pair with equal respect to the ledge to the depression) to implement this technology in practice. In this paper were described results of investigations of microstructure evolution of copper alloys by rolling on the proposed technology in the relief rolls and according to the current technology in smooth rolls.

588 Glass-forming ability and crystallization behavior of Al-Ni-La alloys with other element additions

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A series of Al-Ni-La alloys with different contents of Gd and Si added were suction cast under identical conditions into a wedge-shaped copper mold for investigating the compositional dependence of glass-forming ability (GFA). The GFA of the ternary alloys, except for the optimal glass former ($\text{Al}_{85.5}\text{Ni}_{9.5}\text{La}_5$), is enhanced when proper content of Gd or Si is added. The largest content of Gd or Si up to which GFA can be enhanced increases with a decrease in Ni content. The alloying induced change in GFA was interpreted based on Miracle's model of atom packing efficiency and the chemical action between unlike elements. Furthermore, an abnormal crystallization phenomenon, indicated by an enlargement in the temperature interval between two exothermal peaks with increasing heating rate, was observed during anisothermal annealing of the Al-Ni-La-Si amorphous alloy. The reasons for this special phenomenon are discussed on the basis of thermal kinetic analysis.

589 The effect of rare earth Er on the microstructure and mechanical properties in high temperature titanium alloys

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In this paper, the high temperature titanium alloy containing Er, i.e. $\text{Ti-6Al-2.5Sn-4Zr-0.3Mo-1Nb-0.35Si-xEr}$ ($x=0, 0.1, 0.3\text{wt\%}$) were prepared by using Vacuum Arc Remelting. It is indicates that the microstructure of cast alloys was typical lamellar microstructure. Both the grain size and lamellar structure inside grains were significantly refined with addition of the Er element. The casting billet was forged in beta phase, and then forged in alpha and beta two phase region. After been forged, the alloys were heat treated at 1020 degree, 1h AC and 750 degree, 2h AC. All alloys have two type of microstructure after heat treatment, i.e. lamellar and duplex microstructure, respectively. There were no silicide precipitate after solution treatment, but the silicide increased after aging treatment. Less silicide precipitated in the original beta layer, but in the boundary of lamellar alpha, and less α_2 phase precipitated in the alloys after aging treatment. Subjected to 600 degree 100h thermal exposure, the α_2 phase with size of 5nm to 10nm was precipitated homogeneously in alpha lamellar. The spacing between the α_2 particles was small, about 10nm grade. Specimen containing Er shows excellent creep properties at 600 degree 100h and 150MPa. It is suggests that the silicide and α_2 phase which pin up dislocations strongly were precipitated in the alloys during creep procedure, and the second phase containing Er reduced the amount of beta-stabilizing solution element in surrounding matrix, thus it increased the content of Si solution in the matrix.

590 Gas nitriding of high vanadium alloy steel

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Gas nitriding is a thermochemical treatment widely used to form surface nitrides. This technique is of great industrial interest as it forms a unique composite structure with a hard surface and a tough interior, so that nitriding of alloys and steels can further enhance its surface mechanical performance and tribological properties. HVAS is an air-hardening alloy steel and due to its high vanadium, molybdenum and chromium content provides very good protection against and high strength. The service lives of the hammer and crushing jaw, which were made of HVAS to be used in the crushing industry. The formation of nitride is advantageous for the performance of HVAS surface due to a large number of nitrogen forming elements in the presence of HVAS, but it is rarely reported in the field of nitriding of HVAS. In this paper, A composite surface layer was fabricated on a HVAS plate by means of a surface gas nitriding at 550°C for 70h. The microstructural characterization and phase analysis of resultant nitride layers were performed using optical, scanning electron microscopy, electron probe microanalyzer, X-ray diffraction methods and hardness measurements. The results of the investigation showed that a composite layer consisting of $\text{Fe}_3\text{N}+\text{Fe}_2\text{N}$ is feasible on the surface of HVAS. Vickers hardness test indicate that the hardness value of the nitrided sample is about 1100 HV at the top surface, and decreases gradually to about 700 HV in the matrix. The depth of hardened layer after surface gas nitriding was about 200 μm .

591 Morphology evolution of grain boundary carbide in Inconel alloy 690 after grain boundary engineering

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The chromium riched carbides precipitated at grain boundaries have strongly effects on the resistance of intergranular attacks of nickel based superalloy and austenitic stainless steel. In this paper, the effects of grain boundary characters on the morphology evolution of grain boundary carbides in highly twinned Inconel 690 nickel based superalloy aged at 715 °C-1100 °C for 1-100 h were investigated by TEM, SEM and EBSD. During aging at 715 °C, all the carbides grow dendritically, and the growth of carbides is controlled by the diffusion of Cr atoms. The morphology of carbide precipitated at coherent $\Sigma 3$ boundary is finest within all the aging time. Bar-like carbide precipitated near both sides of incoherent $\Sigma 3$ boundary, and bigger carbide than that of coherent $\Sigma 3$ boundary has been found on the incoherent $\Sigma 3$ boundary. Bar-like carbide precipitated near only one side of $\Sigma 9$ boundary, and much bigger carbide than that of $\Sigma 3$ boundary has been found on the $\Sigma 9$ boundary. The morphology of carbides precipitated at $\Sigma 27$ and random grain boundaries are similar, and the sizes are the biggest among all the types of grain boundaries. The carbides precipitated at grain boundaries with all types grow bigger with the aging time prolonging, but their growth rates are different. During aging at 800 °C or higher temperature, the growth of carbide is controlled by the phase interface energy. The morphologies of carbides precipitated at grain boundaries with various characters are similar. The shape of most of carbides is cubic with straight phase interfaces.

592 Atomistic experimental and simulation investigation on the modification of Al-Si alloys

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Modifying the eutectic Si from flake-like to fibrous is a key factor to improve the properties of Al-Si alloys. The impurity-induced twinning (IIT) mechanism and the twin plane re-entrant edge (TPRE) mechanism as well as the poisoning of the TPRE mechanism are generally accepted to be valid under certain conditions. However, IIT, TPRE or poisoning of TPRE mechanism cannot be used to interpret all the observations accompanying modification, indicating that other factors may be also valid. In this contribution, high resolution scanning transmission electron microscopy high angle annular dark field imaging and electron energy loss spectroscopy as well as atom probe tomography was used to elucidate the distribution of modifying elements within eutectic Si and at the interface between eutectic Al and eutectic Si. Density functional theory calculation was also performed to elucidate the bonding behaviour of modifying elements within Si twins and its effect on the Si twinning. Both experimental and simulation investigation reveals that modifying elements have three different roles: (i) the adsorption at the intersection of Si facets, inducing IIT growth mechanism, (ii) the adsorption at the twin plane re-entrant edge, inducing TPRE growth mechanism, and (iii) the adsorption ahead of the growing Si twins, inducing a solute entrapment within eutectic Si. This investigation not only demonstrates, for the first time, a direct experimental support to the well-accepted poisoning of the TPRE and IIT growth mechanisms, but also provides a full picture about the behaviour of modifying elements.

593 Local atomic symmetry in metallic liquids and glasses

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With sufficient high cooling rates, a variety of liquids, including metallic melts, will cross a glass transition temperature and solidify into glass accompanying a dramatic increase of the shear viscosity in approximate 17 orders of magnitude. Due to the intricate atomic structure and dynamic behaviors of liquid, it is yet difficult to capture the underlying structural mechanism responsible for the dramatic slowing down during glass transition, which impedes deep understanding of the formation and nature of glasses. Here, we report that a universal structural indicator, the average degree of five-fold local symmetry, can well describe the slowdown dynamics during glass transition. A straightforward relationship between structural parameter and viscosity (or alpha-relaxation time) is deduced, which establishes an explicit relation between the dramatic dynamic arrest and the underlying structural evolution. This finding would be helpful in understanding the long-standing challenges of glass transition mechanism in the structural perspective.

594 Surface modification of interfacial structure of the novel solar cells

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The carrier lifetime and the doping property of the photoabsorption materials are essential factors determining their application in solar cells. Surface modification is a practical approach to get unique contact of two different materials and realize special interfacial structure and status between two buildingblock layers. Here, we talked about the surface passivation of silicon nanostructure using for hybrid solar cells, and even the carrier lifetime and the doping property of the lead halide perovskites in solar cells. On the one hand, a promising passivation scheme in ultrathin silicon hybrid solar cells is presented. We demonstrate that this passivation scheme employing different silicon layers at both interfaces should be a promising method in the ultrathin silicon/polymer hybrid solar cells. On the other hand, the synchronous surface passivation of the trap states and interfacial doping of the perovskite are achieved by its surface modification with a functional material. Here, an intelligent approach to reduce carrier recombination both at the surface and the subsurface is proposed by synchronous surface passivation and interfacial doping of the perovskite adjacent to hole transport material.

595 Toughen and harden metallic glass by designing statistical and spatial heterogeneities

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Although having remarkable mechanical properties, metallic glass is severely limited by the lack of toughness and hardening ability which are among the basic requirements for structural and functional applications. Various attempts have been made experimentally to address these issues but with very limited improvement. Here we report a series of attempts via material design approach using finite element modeling. We deliberately introduce statistical and spatial heterogeneities into the metallic glass samples, i.e. free volume dispersity and spatial variation. We show that the plasticity and toughness can be enhanced systematically with the increase of the heterogeneity and in certain circumstance even apparent hardening can be achieved. We will discuss the underlying mechanisms and also possible design of new metallic glass composites using the concept of heterogeneity dispersion.

596 Microstructure analysis of magnesium-based foams through X-ray micro-computed tomography

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X-ray micro-computed tomography technique was utilized to investigate 3D microstructure of carbon nanotube reinforced magnesium composite foams with different porosities and compositions. The data were analyzed to explore the effect of overall porosity and carbon

composition on 3D pore microstructure such as the number of large pores, pore connectivity, pore size, pore size distribution, pore shape distribution, and specific surface area. The increase of overall porosity resulted in more large and connected pores, and a larger specific surface area. For all studied composite foams, pore size varies in the range of several microns to hundreds of microns; and over 80% of the pores have the aspect ratio less than and equal to 2. The introduction of pores significantly increases the specific surface area. The results show that the specific surface area of magnesium composite foams with 49% overall porosity is at least 41 times of the external surface area of the same sample. These microstructure features make the material appealing for hydrogen storage and biomedical applications.

597 Ultrahigh-strength bulk metallic glasses

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Compared with crystalline alloy materials, bulk metallic glasses (BMGs) exhibit higher strength, specific strength and hardness due to their disordered structures and structural uniformity. The development of ultrahigh-strength (more than 5 GPa) bulk metallic materials is significant for urgent applications for spaceflight, micro-electromechanical systems, industrial manufacture, etc. Recently, we reported a family of novel B-rich Co-based BMGs. The influences of the similar element substitution on the glass-forming ability, the thermal stability, the elastic moduli, and the mechanical properties were evaluated. These BMGs exhibit high thermal stability, high elastic moduli, ultrahigh compressive strength and high Vickers hardness, which are highest values among all of reported BMGs. The correlations among these properties were clarified. These kinds of ultrahigh-strength BMGs with high glass-forming ability, high thermal stability and extraordinary mechanical properties could be applied as high wear-resistance bearings, high-temperature structural parts and luxury materials (jewelry, watch, ultraportable electronic devices and etc.).

598 Strain induced martensitic transformation in Austempered Ductile Iron (ADI)

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Austempered ductile iron (ADI) has undergone a special heat treatment to greatly enhance mechanical properties. The heat treatment process of ADI consists of austenitization, quenching to a temperature between 250°C and 450°C and isothermal austempering [1]. After such a heat treatment, the microstructure consists of acicular ferrite and high carbon enriched retained austenite. The high carbon enriched retained austenite can transform to martensite during plastic deformation. The treatment parameters (austenitization temperature, austempering temperature, austempering time and alloying composition) can influence the retained austenite fraction, grain size and its stabilisation [2], which in turn will influence the following martensitic transformation. The influence of different treatment and composition parameters on the martensitic transformation and texture formation during plastic deformation has been investigated using neutron diffraction. The combination of texture analysis and in-situ deformation tests allowed quantitative phase analysis and extraction of martensite phase

fractions as a function of strain level. The experiments allowed us to determine the influence of austempering temperature, Nickel content and plastic strain on the martensitic transformation kinetic in ADI. References: [1] Leopold Meier, Michael Hofmann, Patrick Saal, Wolfram Volk, Hartmut Hoffmann, Materials Characterization 85 (2013) 124-133 [2] Srinivasmurthy Daber et al, J.Mater.Sci. 43 (2008):4929-4937

599 Metal-diamond composites processed by selective laser melting

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Beam based additive manufacturing (AM) processes such as selective laser melting (SLM) provide many possibilities for fabrication of novel metal matrix composites (MMC) with tailored properties. The very rapid consolidation of the base material in a small material volume and the achieved high solidification rates allow for the manufacture of components containing meta-stable materials like diamond which tend to decompose in conventional casting or sintering processes. At the same time, these conditions may lead to complex out-of-equilibrium microstructures, pronounced element segregation and crack formation in the bulk alloy. In the present work, the feasibility of processing CuSnTi-based alloys including diamond particles by SLM is studied. The microstructure evolved during processing was characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD) techniques. It revealed that under appropriate processing parameters the majority of the diamonds survived without graphitization, and the diamond particles were well bonded with the matrix. SLM processing leads to metal-diamond composites with good thermal conductivity and wear resistance, making them promising candidates for applications in the abrasive tooling industry. Additionally, the attempt to further optimize the matrix alloy in terms of chemical composition, microstructure, etc. for a better processability is presented and discussed.

600 Fatigue deformation and damage behavior of Fe-18Cr-18Mn-0.63N high nitrogen austenitic stainless steel

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The fatigue behavior of Fe-18Cr-18Mn-0.63N high nitrogen austenitic stainless steel was examined under strain or stress amplitude control. Under tension-compression fatigue tests, immediate cyclic softening takes place and subsequently a saturation or quasi-saturation state occupies the main part of the whole fatigue life. At lower strain amplitudes, the variation in effective stress, which is associated with the destruction of short range ordering, is mainly responsible for cyclic softening. In contrast, the imposed high strain amplitudes also encourage the reduction in internal stress to contribute to cyclic softening. The relationship of fatigue life vs. plastic strain amplitude follows a bilinear Coffin-Manson rule, resulting from the variation in slip deformation mode with the strain amplitude. At low strain amplitudes, cracks initiate

along slip bands, and planar slip dislocation configurations dominate the major characteristic of microstructures. At high strain amplitudes, intergranular cracks (mostly along GBs and few along TBs) are formed, and the microstructures consist of dislocation cells, stacking faults, a small amount of DTs, besides planar slip dislocations. Under tension-tension fatigue tests, with increasing stress amplitude, the deformation damage features change from slip cracking into intergranular cracking. At low stress amplitudes, deformation microstructures comprise planar slip dislocations, stacking faults, and a few DTs. At high stress amplitudes, planar- and wavy-slip dislocations, DTs and twin-like bands are observed. The difference in deformation mechanisms at low and high stress amplitudes are the major reason for the presence of a bilinear Basquin relationship in the curve of fatigue life vs. stress amplitude.

601 Enhanced corrosion resistance and cell behavior of NiTi shape memory alloy by titanium ion implantation

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The near equiatomic TiNi alloy has been widely used in biomedical applications because it provides a combination of corrosion resistance, biocompatibility, and low elastic modulus. Moreover, this alloy currently plays important role in many clinic implants such as vena cava filters, vascular stents, and wires for orthodontic and dental applications. In the long-term in vivo application of TiNi, however, corrosion unavoidably occurs, leading to a loss of strength and the release of Ni ions, which are considered potential risks, causing allergic responses, cytotoxicity, and carcinogenic effects. In the present work, the Ti-49.8Ni alloy was modified by Ti ion implantation to improve its corrosion resistance and biocompatibility. The chemical composition and morphologies of the TiNi alloy surface were measured using atomic force microscopy (AFM), auger electron spectroscopy (AES), and X-ray photoelectron spectroscopy (XPS). The results revealed that Ti ion implantation caused the reduction of Ni concentration and the formation of a TiO₂ nano-film on the TiNi alloy. The phase transformation temperatures of the Ti-TiNi alloy kept almost invariable after Ti ion implantation. Electrochemical tests indicated that the corrosion resistance of TiNi was enhanced by Ti ion implantation. Moreover, the Ni ion release rate in 0.9% NaCl solution for the alloy remarkably decreased due to the effect of newly formed TiO₂ nano-film. The experiments on cell proliferation show better cyto-compatibility for the TiNi alloy after Ti ion implantation.

602 Special twinning behaviors induced by dynamic plastic deformation

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With the nature of high strain rate impact loading, dynamic plastic deformation (DPD) has been proved an efficient method to produce nano-twin structures in low stacking fault energy metals and alloys with fcc crystal structure. Our research work shows that DPD can also change the deformation behavior of hcp metals and high stacking fault energy Al alloys. It is found that a

large fraction of kink bands could form in Al-7Mg alloy and commercial purity (CP) Ti during DPD process at low deformation strains. With increasing deformation strain, the kink bands gradually evolved into in-coherent twins in Al-7Mg alloy, while in CP-Ti the kink bands could evolve into {11-21} twins. The twinning mechanisms in the two alloys are discussed.

603 Interfacial strength characterization in a high-modulus low-density steel-based Fe-TiB₂ composite

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The titanium diboride (TiB₂)-reinforced steel composite, designed for automotive applications, exhibits a combination of high isotropic Young's modulus and low density as compared to existing advanced high strength steels (AHSS). The steel-based composite is produced by in-situ precipitation of TiB₂ particles during eutectic solidification followed by hot rolling, and its microstructure displays a homogeneous distribution of both large primary TiB₂ and small eutectic TiB₂ particles in the ferrite matrix. Instead of interfacial debonding, particle cracking is the primary mode of damage, revealing very high interfacial strength. To investigate the intrinsic interfacial strength, a hybrid method combining both nanoindentation and finite element analysis (FEA) was used. A micron-sized sample containing a single crystal TiB₂ attached to the ferrite matrix was fabricated by focused ion beam (FIB), and was compressed using nanoindentation with a flat indentation tip. By combining the compression and FEA results, the interfacial strength was therefore determined.

604 Understanding and controlling the microscale silicon distribution for microstructure optimization of Q&P steels

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Q&P steels are alloyed with a medium carbon content, and a small amount of silicon and manganese. Their microstructure is composed of tempered martensite, fresh martensite and retained austenite which gives them a good balance of strength and ductility. It is created by a thermal treatment as following: firstly, fully austenitization; then an interrupted quenching to a temperature which is in between the Ms and the Mf temperature in order to create a mixed microstructure with quenched martensite and retained austenite; and a final partitioning process which holds the sample at a temperature normally higher than Ms. During heating, the originally homogeneously distributed silicon segregates into the residual ferrite and stabilizes it against the desired austenite transformation. As a result one may obtain structures with residual ferrite or with high Si-containing austenite areas at the end of the austenitization treatment. During quenching this leads to big and undesired ferrite or to relatively soft upper bainite, which doesn't participate at the subsequent partitioning process. In our research we characterize the partitioning of silicon during heating and cooling and investigate the possibilities to prevent strong silicon micro-segregation. This is done by controlled cooling after hot rolling, by modifying the heating process, or by alloying with gamma-stabilizing micro-alloying elements.

605 Influence of Cu doping on martensitic and magnetic transitions in Ni-Mn-Sn alloys

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The martensitic transformation features and the magnetic properties of Ni-Mn-Sn magnetic shape memory alloys are strongly dependent on the composition. The structural and magnetic transition behaviors, and consequently the associated physical properties can be modified through the doping with a fourth element. In this work, the fourth element Cu was introduced to gradually substitute Ni in polycrystalline Ni-Mn-Sn alloys. It was shown that the martensitic transformation temperatures of $\text{Ni}_{50-x}\text{Cu}_x\text{Mn}_{39}\text{Sn}_{11}$ ($x=0, 1, \dots, 7$) alloys decreased with the increase of Cu content. Accordingly, the room temperature phase exhibited a gradual transition from four layered 4O martensite to cubic austenite. When x was between 0 and 5, $\text{Ni}_{50-x}\text{Cu}_x\text{Mn}_{39}\text{Sn}_{11}$ alloys were in paramagnetic state at room temperature. When x is between 6 and 7, the alloy was in ferromagnetic state at room temperature, where the martensitic transformation is coupled with the magnetic transition from ferromagnetic to paramagnetic.

606 Revealing the individual hardening effects of twins, dislocations, grain boundaries and solid solution in a twinning-induced plasticity steel

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Manganese-rich austenitic twinning-induced plasticity (TWIP) steels with high strength and superior ductility have received much attention in the past two decades. Tremendous efforts have been made to explore their unusual hardening behaviour which includes contributions from twins, dislocations, grain boundaries and solid solution. Nevertheless, the individual hardening effects of twins, dislocations, grain boundaries and solid solution to the high strength of TWIP steels are still unclear. In the present work, the flow stress of a TWIP steel was experimentally decomposed into the respective contributions of twins, dislocations, grain boundaries and solid solution. For the determination of forest hardening, synchrotron X-ray diffraction experiments with line profile analysis were carried out to measure the dislocation density. It is found that the yield stress of the present TWIP steel is controlled by solid solution and grain boundary hardening, which contribute to 238.3 and 238.5 MPa of the flow stress, respectively. After yielding, the work-hardening rate is dominated by dislocation multiplication which accounts for up to 922 MPa at a true strain of 0.4, equal to ~60% of the flow stress. In comparison, twins contribute to only 118 MPa at the same true strain, equal to ~8% of the flow stress. In other words, twins have minor effect on the flow stress, in contrast to the current understandings in the literature.

607 Affinity viscosimetry sensor for enzyme free detection of glucose in a micro-bioreaction chamber

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With the growing demand of miniaturization of cell cultivation a new approach towards measuring and sensing bio-analytes need to be made due to the problem of small volumes containing small amounts of analytes. We developed a 150µl micro-bioreaction chamber, which is able to grow many different cell types ranging from HeLa-Cell to Lymph-Cells. Inside the reaction chamber an online measurement needs to be achieved to closely monitor and control the process of cell growth and interaction. Important parameters to monitor and control are pH-value, oxygen and carbon dioxide levels along with lactose amount. One of the most important analyte to monitor is the concentration of glucose inside the reaction chamber, because it determines how vital any kind of cell is. Most of the available glucose sensors monitor the glucose concentration with the help of GOD (glucose oxidase), which becomes very inaccurate in terms of long time measurement and uses (i.e. consumes) glucose during the measurement, which is not anymore available for the cells. Therefore, we focused on developing an enzyme-free glucose sensor based on a microelectromechanical system (MEMS). The magnitude of the sensor is 8mm x 1,6mm x 1mm and it is directly positioned into the reaction chamber. For online measurements, we show how the measurement curve is performing in terms of sensitivity, lower and upper limit of detection (bioreactor cells, medical application), response time and figure deviation. Moreover, we would like to show how reliably the sensor can detect the glucose concentration over a defined period of time.

608 Experimental bio-ESPI for validation of magnetic induced deformation on HeLa cells

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The introduction of gas lasers in 1960 revealed an unknown phenomenon. When high coherent light is used to illuminate an optical rough surface or if this light propagates through material with a different refractive index, the surface seems covered with bright and dark spots. This "speckle pattern" is the result of surface variations and is unique for each state of the surface. Since its discovery, several methods have been established using this phenomenon. One of them is the Electronic Speckle Pattern Interferometry (ESPI). In its typical field of application, the ESPI is used for the analysis of macroscopic samples and focuses on mechanical stress as well cyclic deformations. However in terms of this work a different approach is presented.

It is focused on inducing a cellular deformation and monitoring this displacement with an ESPI setup. For doing so, it is crucial that cell and their environment stay close to their idle-state. Therefore the behavior of the cell should only depend from the cellular state and the applied force. To induce the deformation magnetic particles have been placed in the selected HeLa specimen. Afterwards these cells have been exposed to an electro magnet. During this work different modes of excitation have been performed. These first results have been quite promising and revealed, that the detection of cellular displacements is possible. Therefore, it is

intended to apply this method on other specimen and link the measured response with the individual biological backgrounds.

609 Modeling molten Ni-based superalloy properties

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Optimization and control of casting process-parameters can significantly improve the properties of directionally-solidified turbine airfoils. The casting process window is often limited by alloy chemistry, component geometry and possible formation of casting defects. An ab-initio molecular dynamics approach is used to predict fundamental parameters of molten Ni-based superalloys with the goal of informing models of defect formation during solidification process. The Density Functional Theory calculations are applied to molten Ni-based superalloys in order to predict density and diffusion rates in alloys ranging from: model alloys, Rene-N4, Rene-N5, CMSX-4, 1484, and 1487. Calculated densities are consistent with densities interpolated from binary alloy measurements and limited measurements of commercial alloys. Density inversion is also predicted for some of the molten alloys for compositions and temperatures expected in the mushy zone. Progress toward predicting local ordering and shear viscosity will be reviewed. Finally, a new dimensionless parameter is proposed for the temperature dependent materials properties that control defect formation during casting.

610 Development of high-performing extruded magnesium alloy

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Although magnesium alloys have attracted attention as light-weight structural materials due to the low density, their low absolute strengths and low ignition-resistance should be improved to apply to aerospace and military industries. Therefore, the most researches on the development of magnesium alloys have been focused to find the solutions for these drawbacks. However, up to now many researches have been concentrated on improving only one property which is the strength or the ignition-resistance. So, it should be needed study on the enhancement of multi-properties. According to our previous study, the as-extruded Mg-5Gd-2Y-1Zn-0.7Ca alloy specimen, which was extruded at 350 °C with the extrusion ratio of 20, exhibited relatively high UTS of 380 MPa, elongation of 8.9 %, and ignition temperature of 910.3 °C. In this study, effects of Zn addition on the mechanical properties and the ignition-resistance of Mg-5Gd-2Y-1Zn-0.7Ca alloy to develop the high-performing extruded Mg alloys. The test specimens were prepared by indirect extruder with billets of diameter of 40 mm. The extrusion temperature was 350 °C and the extrusion ratio was 20. DTA analyses were performed under dry air atmospheres to measure the ignition temperatures of tested alloys. Also, we discussed the effect of heat treatment on mechanical properties and the effect of Zn addition on corrosion properties of Mg-5Gd-2Y-xZn-0.7Ca alloys.

611 Enhancement of toughness by cooling rate control in bulk metallic glasses

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Bulk metallic glasses (BMGs) are typically considered to be brittle materials due to their highly localized deformation mechanism. Recently, there have been great efforts to control the crystalline phase in the bulk metallic glass matrix composites (BMGMCs) to enhance work-hardening capability. Unlike dendritic growth, the formation of spherulites such as B2 phase does not make severe chemical composition change in the amorphous matrix, which makes tuning the alloy composition much easier. However, fast growth of spherulites results in some defects such as micro segregation and grain boundaries.

In the present study, we demonstrate that single crystal spherulites with B2 structure can be obtained in the (Ti,Zr)₅₀-(Cu,Pd)₅₀ pseudo binary system, which has potential for biomedical use. There is little difference in chemical compositions between spherulites and the amorphous matrix. In addition, there are no high angle grain boundaries and chemical segregation inside the spherulites. Ti-Zr-Cu-Pd alloys were prepared using rapid cooling – suction casting system in vacuum chamber. The volume fraction of B2 phase can be tailored by controlling the cooling rate. The effect of cooling rate on the microstructure and mechanical properties will be discussed.

612 Dependence of film thickness and laser annealing parameters on the optical and electrical properties of ITO/metallic glass alloy bi-layer films

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Ag₄₀Mg₄₀Al₂₀ metallic glass alloy composite, namely, AMA was used in this study. ITO/AMA bi-layer films as an electrode were prepared using RF sputtering and co-sputtering process. The optical and electrical properties of the bi-layers as functions of both ITO and metallic glass alloy thickness are reported. Meanwhile, the bi-layer films are annealed using different laser parameters. For as-deposited bi-layer electrode, the optimal ITO and metallic glass alloy thickness are 30 nm and 9 nm, respectively. The bi-layer films have the transmittance and the sheet resistance values of 77% and 91 ohm/sq. At optimal pulse energy of 1 μJ, the properties of the 30 nm ITO/ 12 nm AMA bi-layer films were improved up to 81% and 36 ohm/sq. due to its crystalline. Therefore, the bi-layer structure has a potential candidate to be as a transparent electrode.

613 Morphological evolution of carbides in DZ125 superalloy during heat treatment

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Morphological evolution of carbides in DZ125 superalloy during heat treatment was investigated by using SEM and EDS. The results show that in DZ125 directionally solidified superalloy, the MC carbide is either smooth platelet or bone-shaped structure with nodes in morphology in as-cast state, which is rich in Ta Ti and Hf. During solid solution treatment, parts of primary MC carbide dissolve, so that the morphology of MC carbide changes from smooth and regular surface to scattered configuration. During aging heat treatment, the secondary MC carbides enriched in Hf element precipitated from the matrix. With the process of heat treatment, the content of Hf element in primary MC carbides increases obviously. Moreover, there is some M_6C carbide precipitated on the grain boundary. The morphology evolution of carbides will be beneficial to the mechanical properties.

614 Evaluation of the hardening capacity of low-alloyed steels quenched by HPGQ in vacuum furnaces

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The technology of High Pressure Gas Quenching (HPGQ) in modern vacuum furnaces has already reached the level of 20 to 25 bar pressure and upto 80m/s gas flow velocity. By using primarily nitrogen (N_2) it is possible to quench also lowalloyed steels, depending of course on the cross-section thickness of the workpiece, and to compete in many cases successfully with oil quenching. For liquid quenchants the hardenability of lowalloyed steels is tested worldwide by standardized Jominy test, but their hardenability and their hardening capacity, when gas quenched, is unknown. The Quenching Research Centre (QRC) at the Faculty for Mech. Engineering, University of Zagreb, Croatia owns unique facilities developed at the Institut für Werkstofftechnik (IWT), University of Bremen, Germany, which is able to experimentally determine the hardenability of lowalloyed steels quenched by gas upto 20 bar pressure and unlimited flow velocity. A computer-aided method has been developed for axially symmetric workpieces of any complex shape to show at a glance the complete hardness distribution on axial cross-section. An included database of relevant steel grades and possible quenching parameters enables to choose other steel and/or quenching parameters and display other relevant hardness distributions, without performing experiments.

615 Effect of nickel content on structure and scratch and wear resistances of nickel doped diamond-like carbon thin films

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Nickel doped diamond-like carbon (DLC:Ni) thin films were deposited on p-Si (100) substrates with DC magnetron co-sputtering deposition by varying DC power applied to a Ni target. The bonding structure, surface morphology, scratch resistance and wear resistance of the DLC:Ni films were studied using X-ray photoelectron spectroscopy (XPS), micro-Raman spectroscopy, atomic force microscopy (AFM), microscratch test, and ball-on-disc micro-tribological test. The Raman results showed that the I_D/I_G ratio increased with increased DC power applied to the Ni target. The increased Ni content in the DLC:Ni films apparently increased the surface roughness of the films. The scratch results indicated that the high Ni content in the DLC:Ni films abruptly affected the scratch resistance of the films via the degraded cohesive strength of the films. The tribological results showed that increasing the DC power applied to the Ni target significantly increased the friction and wear of the Ni-DLC films due to the degraded sp^3 -bonded cross-linking structures of the films. However, the much lower friction and wear of the DLC:Ni coated Si samples than those of the uncoated Si sample implied that the DLC:Ni films could effectively prevent their Si substrates from wear.

616 Stress-induced hardening in a Zr-based bulk metallic glass under elastostatic compression

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Elastostatic compression test on $Zr_{60.34}Cu_{22.32}Fe_{4.85}Al_{9.7}Ag_3$ bulk metallic glass (BMG) were conducted at 90% of yielding strength at room temperature. Homogeneous deformation with 0.48% plastic strain and without shear banding was achieved after holding for 5 days. The deformed BMG shows decreased relaxation enthalpy and increased maximum shear stress and density, demonstrating a hardening behavior, ie., mechanical annealing effect, of BMG after Elastostatic compression. This phenomenon was discussed in terms of the atomic level stress theory and the coalescence of negative and positive free volume. Keywords: Bulk metallic glass; Creep; Relaxation

617 Real-time STEM imaging of nucleation, growth and transformation of the precipitates in age-hardening Al-Cu-(Mg) alloys during in-situ heating

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Age-hardening in Al alloys has been used for more than a century. However, the lack of direct observation limits our understanding of the dynamic nature of the evolution of nano-precipitates during age-hardening. Though extensive microstructure characterizations have been done to understand the transformation process induced by heating, the underlying mechanism pertaining to this well-known phenomenon remains ambiguous. One major reason is that the microstructure observation didn't synchronize with heat treatment in most previous literatures. We will report atomic-scale real-time scanning transmission electron microscopy (STEM) investigations on the micro-structural evolution in Al-Cu-(Mg) alloys using micro-electro-mechanical systems (MEMS) based in situ heating holder. Transformation kinetics and pathway during precipitation in these alloys are revealed in great detail. This study provides quantitative data for temperature-dependent kinetics of nucleation, growth and coarsening of individual strengthening precipitate. The observed local changes associated with a group of precipitates are of fundamental importance in uncovering the interaction of growing precipitate with matrix, dislocations and proximal precipitates due to the overlap of diffusion field. The direct atomic-scale imaging of the thickening and lengthening of the plate-like phases, theta-prime (Al_2Cu) and S (Al_2CuMg), generates new mechanistic insights into their growth process. The evolution of crystal structure in an individual precipitate during heat-treatment is also elucidated by these in-situ TEM studies, as is crucial for linking numerous meta-stable phases.

618 Hierarchical nano-structural design for property enhancement in Al-Mg-Si-(Cu) alloys

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Merits of light-weight and high specific strength of Al alloys have stimulated their wide-spread use in automotive and aerospace industries. Al alloys can also be made as excellent conductors, but their applications to electrical and electronic industries are often limited because of their relatively low strength. We report a promising strategy of nano-structural design for property improvement in Al alloy based on modification of the sequence of conventional cold-deformation and artificial ageing. Through controlling the aggregation states of alloying elements before cold-deformation, we can tune the nano-precipitation in Al alloys directly aged after cold-deformation. A considerable improvement in strength and good ductility is achieved in Al-Mg-Si-(Cu) alloy by our approach. Upon ageing, the dislocations in the as-deformed alloy can act as preferential sites for the solutes precipitation and reduce the tendency of the solutes diffusion toward grain boundary to form grain-boundary segregates, resulting in a considerable prevention on the occurrence of inter-granular corrosion. For post-ageing at elevated temperature, the proposed thermo-mechanical process could enhance the removal of solutes from the Al matrix and properly utilize the work-hardening effect to compensate the loss of

age-hardening effect due to the coarsening of the hardening precipitates in the material, leading to an excellent combination of enhanced strength and good electrical conductivity. Electron microscopy reveals the hierarchical nanostructures consisted of nano-precipitates, nano-lamellar sub-grains and dislocations formed by concurrent precipitation and defects annihilation. Possible mechanisms contributing to the comprehensive property improvement will be discussed.

619 Simultaneous increase in strength and ductility of an Al-Si-based casting aluminum alloy

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A356 casting aluminium alloy (a Al-Si-Mg based aluminium alloy) has a great potential in applications in complex-shape components in the aerospace and automobile industries because of their excellent castability, low density, corrosion resistant, high strength to weight ratio, and low coefficient of thermal expansion, etc. However, A356 alloy normally suffers from low strength and low ductility. Common strategies in improving the strength of A356 alloy are unavoidably accompanied by the scarification of ductility. In this work, we presented a simple route by combining rapid solidification with a post-solidification heat treatment to simultaneously increase strength and ductility of A356 alloy systematically. It is interesting that the hierarchical microstructure originated from non-equilibrium solidification which lead to the concomitant increase in strength and ductility is not influenced by the subsequent long-term solid-solution treatment and artificial aging (T6 heat treatment). The currently applied treatment can effectively enhance the yield strength, ultimate tensile strength, and elongation to failure from 190 MPa, 251 MPa, and 6.2 % to 265 MPa, 353 MPa, and 22.3 %, respectively. The most important significance is that the trend in enhancing the strength and ductility can be systematically enhanced by enhancing one processing parameter, i.e. solidification rate.

620 Interfacial microstructure of 3A21 aluminum alloy/mild carbon joint by magnetic pulse welding

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Magnetic pulse welding is a solid state impact welding process, similar to explosive welding, which produces metallurgical bond by oblique high-speed impact between two metal parts. In this work, dissimilar joining of 3A21 Al alloy/ mild carbon steel (with 0.20 wt% C) was carried out using the magnetic pulse welding method. Microstructure evaluations at the welding interface between the two base metals were precisely examined using scanning electron microscopy (SEM) and high resolution transmission electron microscopy (HR-TEM) coupled with energy dispersive x-ray spectrum (EDX). It was shown that the welding interface exhibits gradient structures composed of dynamic recrystallization zone, nanograined solid solution zone and amorphous zone from Al-alloy side to steel side. It is believed that the formation of the gradient structures at the welding interface was driven by the melting and subsequent rapid solidification of the Al alloy and the diffusion of Fe atoms. The formation of the amorphous zone near the steel side was theoretically explained in terms of Miedema model which indicates

that there is a great driven force for the transformation from Al(Fe) solution to amorphous phase. It is proposed that the amorphous phase formed at the interface plays a crucial role in the wedding of Al alloy and steel by magnetic pulse welding.*Corresponding and presenting author. E-mail address:lliu2000@mail.hust.edu.cn (L. Liu).

621 On interface boundary structures and compositions in aluminum alloys

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The interface boundaries in aluminum alloys often play crucial roles in determining properties of the material. They include twin boundary, grain boundary, and various precipitate-matrix interfaces/ boundaries. Therefore, it is interesting to investigate their structures and compositions. In this work, combining computational and theoretical approaches with deliberated experiments, the structures and compositional changes of a few typical interfaces/boundaries were systematically studied. The major goal of the study is to explore new processes and methods for obtaining high performance aluminum alloy materials.

Firstly, the possibilities to introduce twins in various aluminum solid solutions were investigated against temperature and solute concentration using first-principles energy calculations. Three types of impurities, including intrinsic defects, substitutional solute atoms and interstitial solutes were considered to study their effects on the SFEs in corresponding aluminum solid solutions. It is shown that the intrinsic defects hardly change the SFEs of aluminum, whereas substitutional solutes such as Ge, Y, Sc, Sr and interstitial solutes such as C, N, H, can drastically lower the SFEs of aluminum at low temperatures. Our study suggests that at circumstances the possibilities to introduce twins in aluminum materials can be increased significantly, since their SFEs can be lowered to the values smaller than 45mJ/m^2 . The physical mechanism of lowering the SFEs in those aluminum solid solutions were interpreted by their electronic structures around the SFs and the solutes or impurities. Secondly, various precipitate-matrix interfaces/ boundaries in a AlZnLiMg alloy were studied in details.

622 Quasi-static and dynamic properties of Ti-3.5Al-2.5V-1.5Fe-0.25O titanium alloy plates

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This paper aimed to study the quasi-static and dynamic properties of a titanium alloy plate with nominal composition Ti-3.5Al-2.5V-1.5Fe-0.25O in thickness of 8 mm. The quasi-static tensile properties were investigated by MTS testing system at strain rate of 10^{-3} s^{-1} , while dynamic compression properties by Split Hopkinson Pressure Bar system at strain rate of $3000\pm 200\text{ s}^{-1}$. The results show that the quasi-static properties of the Ti-3.5Al-2.5V-1.5Fe-0.25O plate are in the same level to the commercial Ti-6Al-4V plate in 8mm-thick. The Ti-3.5Al-2.5V-1.5Fe-0.25O plate exhibits good dynamic strength. The average dynamic flow stress is 100MPa higher than that of Ti-6Al-4V plate. However, the maximum strain during homogeneous plastic deformation of Ti-3.5Al-2.5V-1.5Fe-0.25O plate is only 50~60% of that of Ti-6Al-4V plate.

623 Nano-sized surface structures and bubbles in W exposed to high flux D plasma

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International thermal experiment reactor (ITER) project is aimed to build stable Tokamak fusion reactor. Due to its favorable physical properties, tungsten (W) is foreseen as plasma-facing material in ITER. During the operation, the W materials will be exposed to high fluxes and fluences of hydrogen isotope plasmas. It was found that high flux deuterium (D) plasma will cause nano-sized surface morphology on W, such as surface nanostructures and pinholes [1]. However, the mechanism for D bubble in W is far from understood, and no experimental evidence for D bubbles formation in W was reported before. This study is intended to investigate the D bubbles in W and the relationship between D bubbles and nano-sized surface structures. Polycrystalline tungsten samples were exposed to a low energy (~ 38 eV) and high flux ($\sim 10^{24}$ m $^{-2}$ s $^{-1}$) D plasma beam in the Pilot-PSI linear plasma device at 500 K and 1000 K. Surface nanostructures and pinholes were observed on W surface as reported before. Beneath the W surface exposed to D plasma, D bubbles were observed by TEM. The depth of D bubbles was about 15 nm from the surface. For both grains with and without surface nanostructures, bubbles were observed in the near surface region. Therefore, the surface nanostructures were not directly related to the bubbles inside. In addition, it was found that the pinholes were formed on the surface at 1000 K due to the intersection between bubbles and the surface. [1] Y.Z. Jia, et al., J. Nucl. Mater. 463 (2015) 312.

624 Recent progress in X-ray laue diffraction 3D microscopy

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Significant progress has been made at the Advanced Photon Source over the past ten years in developing and applying focused x-ray diffraction techniques in spatially-resolved structural studies at sub-micron level for material science and condensed matter physics issues involving the local crystal structure, orientation, grain morphology, elastic strain, and plastic deformation. A description of the technique will be presented with illustrations of highlighted recent applications. Discussions will include the ongoing upgrade plan of pushing microdiffraction techniques towards nanodiffraction with significant improvements in smaller beam sizes and higher focusing flux, based on the new X-ray sources with diffraction limited emittance and high brightness provided by the multi-bend achromat (MBA) lattice.

625 Study of the precipitation of secondary phases in a duplex and superduplex stainless steel

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The aim of this work is the study of the mechanism of precipitation of the intermetallic phases presents in duplex stainless steels (UNS S32205 and UNS S32750), moreover to find out the most suitable method for detecting and analyzing accurately these secondary phases, particularly Sigma-phase, Chi-phase, nitrides and carbides. The samples were characterized after a solution annealing at 1080°C followed by an isothermal heating at 830°C for different time ranges, ranging from 1 minute to 9 hours, with the porpoise of concerning the mechanism of chi-phase nucleation and nitrides formation in relation with the sigma-phase. The work has two principal objectives, in the first part it has been studied the proper technique for the detection, identification and quantification of the secondary phases, having the best results with the scanning electron microscopy (FESEM) with a backscattered electron detector (BSE) in comparison with the optical microscopy (MO). On the other part, it was studied the composition and mechanism of formation of the intermetallic phases concluding that chi-phase almost doubles the content of molybdenum in sigma-phase, and as a result the kinetics of nucleation and growth were also found to be remarkably faster when the alloy content in the steel is higher. In addition, chromium nitrides and carbides were also observed to precipitate as a result of the heat treatments and, in the case of the chromium nitrides, they act as a favorable site for the nucleation of sigma-phase and chi-phase.

626 Analysis of the static recrystallization behaviour of Nb-Ti microalloyed steels including low strain levels

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As useful tools to optimize rolling schedules, during the last decades a big effort has been put into the development of semi-empirical models describing the mechanisms involved in the austenite microstructural evolution during hot deformation. However, these models present some limitations. Recent works have shown that some of the equations proposed to predict the recrystallized grain size and recrystallization kinetics fail in the case of low strains. However, low strains are of interest in some industrial processes, such as the first passes of plate rolling or for through-thickness microstructural evolution simulation of heavy gauge plates or coils. In this work, the static softening behavior of two low C steels microalloyed with Ti (0.01%) and different Nb levels (0.04 and 0.11%) has been investigated. Double and single hit torsion tests were carried out at deformation temperatures from 1150 to 850 degrees centigrade and strain levels ranging from 0.1 to 0.3. For both steels, the experimental recrystallization kinetics at $\epsilon=0.2$ and $\epsilon=0.3$ correlated well with the theoretical predictions of a previously developed equation. However, when reducing the strain to $\epsilon=0.1$ the experimental softening was significantly retarded compared to the model predictions. Moreover, a dependence of the n Avrami exponent both on temperature and applied strain was found. Current equations proposed for the prediction of the recrystallized grain size also tend to overestimate the measured values. A strain exponent of $m=0.46$ was determined experimentally for the calculation of the recrystallized grain size, this value being lower than those usually proposed

in the literature.

627 Nanostructured Cu-based shape memory alloys obtained by high-pressure torsion

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Shape memory alloys (SMAs), thanks to a characteristic martensitic transformation, present unique functional properties that make them very attractive for technological applications. The most widespread SMAs are Ni–Ti binary alloys, however, in the last years Cu-based alloys are attracting more and more attention, because they can exhibit better performances under the continuously increasing demanding conditions. In polycrystalline state Cu-based SMAs are brittle and mainly two approaches were used to overcome this problem, i.e. use of single crystals or grain size reduction. Concerning the latter, high-pressure torsion (HPT) is a well-established procedure for obtaining nanometer-sized grains that was already applied in Ni–Ti SMAs, but not so much for Cu-based ones. In the present contribution the whole process, starting from elemental powders, to produce nanostructure Cu–Al–Ni SMAs by HPT will be introduced. A complete microstructural characterization of the final material done by electron microscopy and X-ray diffraction together with some functionality tests will also be presented. It is proposed that the current approach will enable a deep understanding of the superelastic and shape memory effects in nanostructured Cu-based SMAs, what was unfeasible with the up-to-now applied production technologies.

628 The possible role of grain boundary complexions on the diffusion of silver through SiC in TRISO nuclear fuel particles

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The TRISO (Tristructural Isotropic) coated fuel particle is made of a uranium kernel coated with three layers of PyC and one of SiC. The purpose of these coatings is to work as a miniature fission product containment vessel, retaining all-important radionuclides even under off-normal conditions. However, among all the wide range of fission products, silver, a strong gamma-ray emitter, can diffuse through SiC even in what appears to be intact fuel. Despite more than forty years of work on this topic, no experimental or modeling work has been able to fully explain the origin of the exacerbated diffusion of silver. Although it is now accepted that silver diffuses through grain boundary diffusion, little is known on the characteristic of the grain boundaries of SiC in this type of fuel and how these variations could affect the release of Ag. In the present work we show the existence of different grain boundary complexions in SiC and relate these variations with the existence of different diffusion rates by combining molecular dynamics modelling and experimental work.

629 Low temperature impact on glass and carbon composite laminates

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Glass and carbon fibre composite laminates in different thicknesses were dynamically loaded at a velocity of about 4 m/sec by a falling weight machine using a cylindrical impactor with hemispherical nose 19,8 mm in diameter. The updating of the impact machine with a thermal chamber allowed to test the rectangular specimens at temperatures under 0°C. An environmental condition critical for structures made of composite material is, in fact, related to low temperatures especially in presence of dynamic loads. This problem is really important for the navigation in the Arctic Ocean.

Impact tests were carried out at complete penetration and at different energy levels. The load curves obtained in the different test conditions were compared. In general, a more brittle behaviour was noted under extreme temperature conditions respect to the room one leading to higher impact forces and absorbed energies necessary to create a larger number of matrix cracks.

630 Macrosegregation of alloying elements in hot-top of large size high strength steel ingot

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Macrosegregation, which refers to the chemical heterogeneities, is one of the important casting defects that should be kept as low as possible. For this, experimental investigations must be conducted to understand the mechanisms governing this phenomenon. However, characterizing macrosegregation in large ingot steel is very expensive and time consuming. Moreover, in this kind of casting, hot-top plays an important role in decreasing the extent of this defect. It contains high levels of macrosegregation due to the directional solidification. Thus, the analysis of macrosegregation in this zone could improve the comprehension of its formation mechanisms and the alloying element behaviours. In this research, the hot-top plus 30 cm below were cut from 40 ton ingot of medium carbon low alloy steel. Then, specimens of $1.5 \times 4.5 \times 6.5 \text{ cm}^3$ were prepared from slice cut along the longitudinal axis of the hot-top. The chemical compositions of these samples were obtained using mass spectroscopy which allows the construction of the whole slice's chemical map. Macrosegregation analyses of the different elements (C, Mn, Cr, Mo and Ni) were performed by the examination of alloying element distributions and their degrees of segregation. The important findings were the revelation of positive macrosegregation and the strong dependence of macrosegregation degree on the initial content of the element. Results are discussed in the framework of the theory of solute rejections during solidification, the primary solidification mode and the nature of alloying elements (alphagene and gammagene).

631 Understanding glass formation from the atomic structure perspective in metallic glasses

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Up to date, atomic packing nature of metallic glasses (MGs) which lack periodic translational ordering remains mysterious, and consequently, the structural mechanism of glass formation in these disordered solids is still unclear, particularly the atomic structure response for the dynamic arrest during glass transition and the relationship between the atomic structure and glass-forming ability. Our current talk include three aspects; first, a universal structural description on atomic packing nature in MGs, i.e., the combination of spherical periodic order (SPO) and local translational symmetry (LTS), will be briefly mentioned. Second, we will discuss the structural origins responsible for the frozen-in dynamics accompanying the glass transition. We found that glass formation is virtually the development process of one-dimension LTS ordering. The LTS ordering clusters form a percolating network that serves as the “backbone” of the rigid glass solid when the temperature approaches glass transition temperature. The percolation occurred via pinning different LTS clusters together, which only needs to adjust a small amount of “joint” atoms in-between them and therefore the thermal energy consumption is very low. Thirdly, investigation of the dynamic evolution of structural heterogeneity will be presented. Our analysis indicates that good glass formers have not only the predominant structure with a large packing density at its liquid state but also excellent heritability of structural heterogeneity during cooling. There exists a solid correlation between the heritability of the structural heterogeneity and glass-forming ability in MGs.

632 Air-based sputtering deposition of nitride, oxynitride, and N-doped oxide thin films

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Conventionally, sputtering deposition of nitride, oxynitride, and oxide thin films was performed by using pure nitrogen/oxygen as reactive gases. Here, air was employed as a reactive gas and argon was used as a plasma gas to produce the films. Thus, the deposition could be conducted at low vacuum, which could reduce significantly the overall processing time and cost. Titanium nitride, titanium oxynitride, and N-doped titanium oxides were selected as a model system for the air-based sputtering deposition. When tuning the key parameters, especially the different air/Ar flow ratios, the obtained films exhibited nitride, oxynitride, and N-doped oxide characteristics, respectively. In principle, kinetically favorable reaction predominated at low air contents, leading to the formation of titanium nitride and oxynitride films. On the other hand, the formation of titanium oxide films at higher air contents was mainly governed by thermodynamics. Processing windows of the resultant films were revealed and discussed.

633 Preparation and squeeze casting of nano-SiC/A356 composite assisted with ultrasonic vibration process

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Metal matrix nano-composites (MMNCs) have excited great interest in recent years, due to their excellent properties. In this work, an efficient process by combining high-energy ball milling with ultrasonic vibration (UV) was employed to prepare MMNCs. The composite granules with 20 wt.% nano-SiC particles were firstly prepared by milling the nano-SiC and Al particles. Then these preformed composite granules were remelt in the matrix melt and treated by UV to prepare the slurry of nano-SiC/A356 composite. The slurry was finally formed by squeeze casting under different pressures. The effects of UV and squeeze casting process on the microstructure and mechanical properties of the MMNCs with different SiC contents were studied. The results indicate that uniform slurry could be obtained within 100s under UV treatment. The nano-SiC particles were uniformly dispersed in the matrix after solidification, and no agglomerations were observed. Compared to the matrix A356 alloy, the tensile strength of the MMNCs with 1~2 wt.% nano-SiC was increased significantly, and the elongation was not reduced.

634 Complementary study of specific volume equilibration in Zr-based bulk metallic glasses using positron annihilation and laser-dilatometry

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Isothermal sub- T_g volume kinetics of bulk metallic glasses upon annealing are readily accessible by dilatometric techniques. Especially by employing the method of high-resolution laser-dilatometry, it is possible to study the equilibration kinetics on very long time scales of up to 10^6 seconds with an unprecedented accuracy. In such measurements it was found that there are at least two, seemingly independent, volume-affecting processes: The well-known irreversible annealing-out of quenched-in excess volume and a superimposed reversible process comparable to very slow vacancy equilibration in a crystalline material. The time-volume evolution of these phenomena can be further analyzed in terms of its functional behavior, e.g., by fitting stretched exponential functions to the measurement data. However, it is not possible to directly deduce the atomistic origin of the volume equilibration process solely from dilatometric measurements. In order to investigate the kinetics on a local atomic scale, techniques based on positron electron annihilation in solids, especially positron lifetime spectroscopy and the Doppler broadening of the annihilation quanta, serve as very powerful tools. These methods can deliver insights into the nature and size of volume-afflicted defects. Moreover, the annihilation of positrons with core electrons contains information about the chemical environment of the annihilation site. In this talk a comparison of measurement results for Zr-based bulk metallic glasses obtained by positron annihilation and high resolution laser dilatometry techniques is given. The results are discussed in terms of possible changes in the short range order of the glass and the atomistic origins of the macroscopic volume equilibration.

635 Enabling diamond deposition with Cold Spray through the coated particle method

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This paper describes the application of cold spray to the deposition of a diamond grade pre-coated with Cu and Ni. This is the first time that pre-coated diamond powders are used as the sole feedstock without the addition of binders (ductile phases) in cold spraying. The experimental results showed that it was possible to manufacture thick metal-diamond composite coatings with high diamond fraction in the coating but without phase change or graphitization. The particle speed velocity achieved in the nozzle was analysed using Computational Fluid Dynamics (CFD). Results from this paper also have demonstrated a new methodology for the deposition of metal-diamond/ceramic composite coating with the cold spray technique. The bonding mechanism is also proposed and analysed at theoretical level using Finite Element Analysis (FEA), to include experimental evidence of key findings.

636 Experimental investigation of welding parameters on automatic TIG welding of aluminium 5083 plate

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Aluminium 5083 is a widely used alloy in marine industries, pressure vessels, rail cars, vehicle bodies, tip truck bodies, etc. due to its exceptional performance in the extreme environment. It offers high resistance to attack by both sea water and industrial chemical environment. It also possesses good weld strength when welded with suitable filler materials. The weld quality depends up on the parameters such as welding current, voltage, welding speed, gas flow rate, etc. To enhance the weld quality of Al 5083 alloy plate using Al 5356 alloy filler rod, an automatic TIG welding system is developed, by which welding speed can be controlled and a uniform speed is obtained. Welding of Al 5083 plate is carried out using Al 5356 filler wire for different welding currents and welding speeds. The effect of welding speed and current, on the tensile strength of the weld, hardness at three different zones viz. weld metal, heat affected zone and base metal are investigated. The hardness and temperature on the weld zone and the base metal are greatly influenced by the welding speed and current. It is observed that, the weld strength is influenced by different values of welding current and speed. The effect of material deposition rate on the weld strength in uniform welding is also studied. Taguchi based design of experiments is used for the evaluation and the optimal welding parameters are determined using the utility theory based multi objective optimization technique.

637 Identification of oxide nano-octahedron and its chemical behaviours in stainless steels

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We find that a “single-grained” MnS inclusion in the steel is compositionally and structurally inhomogeneous. Fine octahedral precipitates of spinel MnCr_2O_4 with dimensions of several tens of nanometers, are dispersedly distributed in the MnS inclusions. In-situ TEM studies indicate that the MnS initially dissolves at the $\text{MnCr}_2\text{O}_4/\text{MnS}$ interface in the presence of salt water, and the dissolution gradually spreads outwards leaving a pit around the MnCr_2O_4 octahedron. However, the reactivity of these octahedra is various. First-principles calculations indicate that the dynamics of MnS dissolution is the function of the species of terminal ions enclosing the nano-octahedron catalyst. The MnCr_2O_4 nano-octahedron with metal terminations is more reactive in catalyzing the MnS dissolution than O-terminated ones. This work not only sets up a new basis for understanding the initiation of pitting corrosion, but also presents a novel example of how an inorganic nano-inclusion undermines its metallic matrix in an electrochemical manner which may occur in a wide range of engineering alloys and biomedical materials serving in wet environments.

638 The effect of thermo mechanical coupling on microstructure and properties in Al-Li alloy

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The microstructures and properties of heat-treatable 2198 Al-Li alloys were studied under the condition of thermal mechanical Coupling. The non-uniform with precipitates and grain size distribution in 2198 Al-Li alloy was processed by friction stir spot weld (FSSW) technology. The microstructure of FSSW 2198 alloys was divided into four zones: thermal-mechanical affected zone (TMAZ), heat affected zone (HAZ), and base material (BM) according to thermodynamics and dynamics during process of FSSW. The microstructure was analyzed by means of SEM, XRD and TEM. It was revealed that the grain size of FSSW 2198 alloys can be remarkably refined to $5\mu\text{m}$. However, the precipitates fully dissolved in the TMAZ and decreased in HAZ. The correspondences of mechanical properties to the non-uniform microstructure in FSSW 2198 alloys were indicated. Meanwhile, the mechanical properties of different regions with various microstructures in FSSW 2198 alloys were studied using small samples. The study results showed that the tensile strength of FSSW 2198 Al-Li alloys decreased according to the distribution of T1 precipitates. The further research have known that the ultimate tensile strength of the FSSW 2198 Al-Li alloy can increased from 373MPa to 423MPa via aging treatment at 175°C for 36h, and due to T1 phase precipitating again in 2198 Al-Li alloy.

639 Effect of CNT distribution on mechanical and physical properties of CNT reinforced aluminum matrix composites

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In this study, carbon nanotubes (CNTs) reinforced 6061Al alloy matrix composites with three different CNT distribution types were fabricated by dry mixing-powder metallurgy (D-PM), wet mixing-powder metallurgy (W-PM) and friction stir processing (FSP), respectively. Microstructural observations indicated that CNTs were distributed as clusters and approximate networks in the composites fabricated by the D-PM and W-PM methods, respectively, whereas CNTs were individually dispersed in the aluminum matrix in the FSP composites. The D-PM composites show decreased strength compared with the matrix alloy as a result of the clustering. The strengths were increased a little for the W-PM composites and the maximum strengths were achieved for the FSP composites due to the uniformly dispersed CNTs and good interface bonding. The best electrical conductivity was obtained for the W-PM composites, even a little higher than the FSP composites, which could be attributed to its network structure.

640 Evolution of plastic zone size at a crack tip with ultra-fine grains in metastable austenite

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One way to reduce the cost of hydrogen infrastructure would be to increase the strength of austenitic steels, in order to reduce the size of components. A laboratory-made austenitic steel, Fe-16Cr-10Ni, was produced with several grain sizes, ranging from 21 micrometer down to 1 micrometer. Small CT specimens were cut out of 1.5-mm-thick sheets. Hydrogen was introduced by high pressure, high temperature hydrogen gas charging. Fatigue cracks were then introduced. The resulting crack paths were compared for coarse and ultra-fine grains (21 and 1 micrometer respectively). The microstructure and the plastic zone size were also analysed by EBSD, to investigate the influence of grain size on the distribution of plastically-deformed area.

641 Metamodel of a thermodynamic simulation applied to multiscale modelling

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One of important disadvantages of heterogeneous multiscale modelling is high computing costs. A typical multiscale simulation consists of numerous fine scale submodels, usually one for each computational point of coarse scale model. If computational requirements of fine scale submodel is not negligible and a number of coarse scale computational points is large, effecting computing time is rather high, what is sometimes unacceptable. One of possible solution is limiting computing power requirements of submodels, replacing them with some simplified and

speeded up submodels. However the speed-up could be significant, risk of rapid decrease of results quality appears when the state of computational domain exceeds narrowed applicability range. In this paper, the Authors address two issues. The first one is an attempt to develop a metamodel, replacing direct thermodynamic computations of precipitation kinetic with approximating model. The second one is developing of adaptive multiscale model able to use both, thermodynamic and approximating submodels interchangeably. MatCalc simulator has been used for thermodynamic modelling of precipitation kinetic. Simple heat treatment of P91 steel grade was examined. Since the output from MatCalc is very reach, only selected variables were chosen to be modelled with approximating submodels. Several attempts with various approximation variants (interpolation algorithms and Neural Networks has been investigated and its comparison is included in this paper. Both MatCalc and approximating models were integrated with multiscale modelling framework AM3. The rules for switching between them were implemented. Some testing multiscale simulations were computed and the analysis of computation process is included in the paper.

642 Influence of the composition and sintering conditions on the thermomechanical properties of SPSed carbides

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Spark Plasma Sintering (SPS) has proved his efficiency on the sintering for various materials which were known to be difficult to sinter without additives. In this presentation, the investigation of some thermomechanical properties will be illustrated on two different carbides (zirconium carbide and boron carbide). Zirconium carbide is interesting for high temperature application. Zirconium carbide (ZrC_x) and oxycarbide (ZrC_xO_y) specimens were synthesized by carboreduction of zirconia and carbon and was then sintered by SPS. A complete study including chemical composition, particle size evolution was carried out. The kinetics of densification of spark plasma sintered ceramics have been also investigated. From these results, it appears that densification mechanism can change between the intermediate and final sintering stage. During this last stage, the deformation mechanism is similar to the one involved during creep of dense ceramics. The comparison of densification and creep strain rates seems to show that no specific effects strongly enhance strain rate during the final densification stage of Spark Plasma Sintering. [1]

Boron carbide is a promising material for moderator in nuclear industry. The influence of preparation before SPS was studied: a liquid way was studied and compared. Different solvent were tested and green bodies were obtained. During sintering, different chemical reactions were observed: oxides present in the native powder react and the composition of the carbide evolves. In a second step, thermomechanical properties were determined.

References: [1] G. Antou, N. Pradeilles, M. Gendre, A. Maître, Scripta Materialia, (2015) 101 pp 103-106

643 Non conventional mechanical testing of thick Al-Li alloys sheets welded by Solid State Capacitor Discharge (SS-CDW) for aeronautical applications

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Al-Li alloys are promising lightweight materials for aeronautical applications, however, their current use is limited due to their poor weldability by conventional liquid phase welding methods. Among the solid state welding techniques, capacitor discharge welding is very attractive since the typical adverse properties of solidified microstructures are avoided. Moreover, the process is clean, energetically efficient and fast. SS-CDW is characterized by a large current intensity (340 kA peak), low voltage (30 V peak) in secondary circuit and welding times in the range of 5-30 ms. The SS-CDW machine essentially includes two Cu electrodes, a capacitor bank, which stores the needed welding energy, and a high-voltage to low-voltage transformer. Due to the highly localized heating at the joint interface, a recrystallized structure of about 500 microns width around the joint interface is usually formed which generally exhibits a dramatic drop of the mechanical properties. As the copper electrodes are connected with a massive copper tooling, the subsequent conduction cooling after welding is also extremely fast. Consequently, this kind of alloy undergoes a kind of natural aging with progressive increasing of local mechanical properties in time. Instrumented Vickers/Brinell indentation test, in the macro hardness range, is suitable to assess the change of local properties over a sufficient volume of a representative microstructure. A correlation between recrystallized microstructure and mechanical properties will be worked out after a sufficient aging time for the Al-Li alloy joint welded by SS-CDW.

644 A novel facility to investigate dust mobilization in confined environments with applications of the safety of the medical industry

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Many medical industry all around the world are faced with the problem of dust mobilization during the productive process of medicines. This mobilization can be dangerous for the safety of the operators working in the factory and for the safety of the factory. It is therefore necessary develop predictive models to simulate and forecast dust mobilization. The Quantum Electronics and Plasma Physics Research Group of University of Rome Tor Vergata has developed a facility to experimentally replicate dust mobilization in different critical conditions in an enclosed environment. The measurements available in the facility provide the boundary conditions to run numerical simulations and to validate the mobilization models used. Even if the initial field of application of this novel facility is dust mobilization is nuclear fusion, the methodology developed can be used for the industry of medicine, the agribusiness and others. The authors will present the experimental and numerical results discussing new applications.

645 Superplastic behavior of hot rolled Al-Mg-Sc sheets joined by friction stir welding

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The high-strength sheets of Al-5.4Mg-0.2Sc-0.1Zr alloy were produced by equal-channel angular pressing (ECAP) to 12 passes via route B_C at 573 K followed by isothermal rolling at similar temperature to a total thickness reduction of 80%. The final sheets with ultra-fine grained (UFG) structure were joined by friction stir welding (FSW). The tensile samples from all of the characteristic FSW microstructural zones were machined perpendicular to welding direction. The alloy demonstrated excellent superplastic properties in the range of temperatures of 673 to 737 K and strain rates of $2.8 \times 10^{-4} \text{ s}^{-1}$ to $2.8 \times 10^{-1} \text{ s}^{-1}$. During superplastic flow, no localization of plastic deformation in the stir zone was found, and, therefore, the sheets joined by FSW showed uniform deformation in whole material body. The relationship between superplastic ductility and microstructure and the use of this technique for the fabrication of large-scale superplastic sheets are discussed.

646 Thermal desorption spectroscopy study on the hydrogen behavior in a plasma charged aluminum

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Hydrogen in aluminum has been known to be the cause of blister and pore. Some aluminum alloy is susceptible to stress corrosion cracking, which is based on intergranular cracking arisen from hydrogen embrittlement. The behavior of hydrogen in aluminum has not been fully understood yet. Hydrogen gas plasma enables to introduce high hydrogen concentrations into aluminum without Al(OH)₃ layer on the surface of specimen. In this paper, we have investigated the behavior of hydrogen in a plasma charged aluminum by means of thermal desorption spectroscopy, a method to evaluate the amount and trap states of hydrogen. Cold-rolled pure aluminum foils of 0.2mm in thickness were annealed, mechanically polished and charged with hydrogen gas plasma. Immediately after hydrogen gas plasma charging, TDS tests were performed under ultra-high vacuum. The hydrogen desorption spectrums obtained by TDS tests had three peaks corresponding to desorption from the vacancy, dislocation and pore. Compared to as-annealed sample, in a plasma charged one, the amount of hydrogen trapped by vacancies especially increased. This result suggests that hydrogen uptake occurs with the formation of hydrogen-vacancy complex on aluminum surface during hydrogen gas plasma charging.

647 Reactive diffusion for contact in advanced MOS devices

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Metallic silicides are widely used as contact materials in MOS structure and NiSi is actually used due to low resistivity, low thermal budget and low Si consumption. Ni(Pt)Si with 10 at% Pt is currently employed in recent technologies since Pt allows stabilizing NiSi at high temperature [1]. The presence of Pt and the very low thickness (< 10 nm) needed for the contact in the devices bring new concerns for actual devices. Indeed phenomena like nucleation, lateral growth, interfacial reaction, stress, texture, and transient phase formation [2] can play an important role. The presence of alloy elements (Pt, Ge...) as well as stress and defects induced by the confinement in devices may have also an effect on the contact properties. In this work, in situ techniques (XRD, sheet resistance, DSC) were combined with atom probe tomography (APT) to study the formation mechanisms as well as the redistribution of dopants and alloy elements (Pt, Ge...) during the silicide formation [3]. In particular, the analysis by APT in 3D and at the atomic scale of MOSFET allows to link redistribution to process steps [2, 4]. [1] Mangelinck et al, APL, 75 (1999) 1736 [2] Mangelinck et al, Phys. Stat. Sol. A, 211 (2014) 152 [3] El Kousseifi et al, Acta Mater., 83 (2014) 488 [4] Panciera et al, APL, 100, (2012) 201909

648 Recent advancements towards industrialization of magnetic pulse welding

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In an innovation economy, industries need to innovate to make their product more efficient and economical. One way of increasing the product performance is by the application of multi-material combinations. However the welding of bimetallic joints still remains a challenging task and conventional methods quickly reach their technology limits, emphasizing on need for new methods. To achieve this target, today industries look for new technology which will permit bimetallic joining while meeting other requirements such as low initial and running cost, eco-friendly, processing time, and automatable. Magnetic pulse welding is one such promising solid state welding technology fulfilling all the required attributes and it has attracted considerable interest from industries. In MPW, welding is achieved by the effect of Lorentz force i.e. by making the parts to be welded are made to impact at very high velocity between each other. The Lorentz force is induced by the creation of a time varying high density magnetic field around the parts with help of an inductor. The process physics revolves around the effective management of the generated magnetic field to achieve the desired impact velocity allowing metallurgical bonding between the parts. The characteristics of the magnetic pulse generator and the tool limit the possible part configurations that could be welded using this technology. Through rigorous control of processing conditions, solid state welding between dissimilar metals, such as Al-Cu, Al-Fe, Fe-Ti has been successfully implemented. This article will deal with recent technological advancement, which enhances its application potential in an industrial environment.

649 Friction stir welding on light-weight metal – aluminum alloy Al6061

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Friction stir welding (FSW) is a solid state joining process in which metals are joined together using frictional heat and severe plastic deformation. The heating and the mixing of the metals is performed using a hardened tool with a shoulder and pin. FSW of lightweight metal alloy Al6061 has been carried out in the present study. For welding aluminum the parameters used were a constant tool rotation speed of 1600 rpm and varying tool translation speeds of 250, 500, 750 and 1000 mm/min. The welded coupons were characterized for microstructural observations and mechanical properties such as tensile and Charpy impact properties. The tensile and impact properties were studied at two different temperature namely, room temperature (RT) and 300°C. The FS welded aluminum specimens showed 86% – 98% tensile yield strength, in comparison to the base material at RT. At 300°C, the yield strength was observed to be 85% to 93% of the base material value. For the impact properties, the Al specimens showed 60% – 140% specific impact energy, in comparison to their respective base materials. Based on the mechanical properties and microstructural examination, the optimal weld parameter was identified as 1600 rpm and 250 mm/min which is dependent on the tool pin and shoulder design utilized in the study.

650 Susceptor design and in-situ shrinkage-temperature measurement during microwave sintering of oxides

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Microwave energy is a very efficient way for the synthesis of new materials or the sintering of ceramics. In the last years, many improvements have been realized, including the design of original susceptors and the discovery of the most suitable thermal insulator among others. All these were carried out to optimize the conversion of electromagnetic energy into heat. However, most of the performed developments result by trial and error methods until now. This strategy can be time consuming and is not the most suitable way to optimize any processes. To produce high quality sintered oxides parts, we have designed specific assemblies including thermal insulating materials and eventually SiC or ZrO₂ based susceptors. The design of the best assembly was obtained by modelling using a multi-physics software. The relations between the experimental results like temperature distribution within the sample and homogeneity of the microstructure, and the modelling results have been discussed. In addition, it has been developed a specific instrumentation to measure the shrinkage and the sample temperature during microwave sintering, in working carefully on the temperature calibration. It is shown how those developments allow to significantly improve the overall control of the microwave process, in either 2.45 GHz or 915 MHz. Some examples will be presented showing successful sintering experiments on different materials (nano-powders Al₂O₃), with different sizes and geometries. The microstructure is also discussed as a function of the thermal history of the microwave heated samples, and the advantages of the microwave process over conventional are discussed.

651 Characterization and modelling the microstructure and texture evolution in AlMgSi-extrusions

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The mechanical properties and surface appearance of aluminium extrusions are critically dependent on the microstructure and texture of the extruded profiles, and the requirements with respect to these aspects may vary with applications. Moreover it is often a challenge to produce extrusions with a consistent and homogenous grain structure and texture along as well as through the thickness of the profiles. It is thus vital to understand and be able to model how different microstructures and textures are formed and how they evolve during and after extrusion. In the present work a modelling framework has been implemented which includes a Finite Element model (HyperXtrude) to account for the strain, strain rate and temperature along a set of particle paths during extrusion. From these the deformation texture and grain structure are calculated with an appropriate deformation texture model (ALAMEL) and sub-structure evolution model (ALFLOW), respectively. The sub-structure model have been coupled to a crystal plasticity model to provide an orientation dependent subgrain size and dislocation density during deformation which provides the driving force for the post-extrusion recovery and possible recrystallization behaviour. The post-extrusion microstructure and texture evolution are calculated with the recovery and recrystallization model ALSOFT, which is accompanied by a recrystallization texture model, capable of predicting the recrystallization texture in axi-symmetric as well as flat extruded profiles. The predictive power of the modelling framework has been tested and compared with experimental results both for the lab-scale extrusion of round bars as well as flat profiles of an AlMgSi-alloy.

652 Effects of powders on the EBM process and on as-built materials

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Additive manufacturing technologies have expanded over the last decade, the most developed techniques being those classified as powder bed (Laser Beam Melting, Electron Beam Melting). Those techniques use powders as raw materials to manufacture metallic 3D parts. One question which is often raised deals with the role of the powders on the process and on the parts. The objective of this talk is to illustrate how important the starting materials can be. The influence of powders on the Electron Beam Melting process itself, particularly on the pre-heating stage will be discussed. The influence of the powders used on the dimensional and microstructural characteristics as well as the final properties of produced parts will be illustrated through a few examples drawn from different raw materials.

653 The wet and hot corrosion behavior of iron aluminides

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Iron rich Fe-Al based alloys show various enhanced properties compared to stainless steels, such as a high corrosion resistance, high wear resistance as well as low costs of the raw materials. Therefore, Fe-Al based alloys are of main interest for industry to replace stainless steels for the fabrication of different components. As these components are often used within corrosive environments, e.g. oxidizing atmospheres, acids or molten salts, it is important to study the respective corrosion resistance of these alloys.

In this study, two different iron aluminide based alloys were investigated: Fe-Al-Ti-B and Fe-Al-Ti-B with 2 at% Mo. A good balance between strength at high temperature and sufficient ductility at ambient temperature characterizes these alloys. Their oxidation resistance was established in air using a thermobalance. Hot corrosion was tested by heating to 900 °C for 100 h in Na₂SO₄ salt. For wet corrosion, the electrochemical potential was measured in sulphuric acid with a pH of 1.6 at 97 °C. Additionally, NaCl, formic acid as well as nitric acid (all pH = 2) were added as pollutants.

654 Challenges of material science in additive manufacturing. Some case studies with CLAD process

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Additive manufacturing of metallic parts has taken long strides in its path to marketplace. Technological innovations combined with optimization of feedstock materials and on going standardization have improved the process viability. The layer-by-layer deposition of feed materials (wires, powders, ribbons.), molten after interactions with a finely tuned heat source (Electron beam, Laser) still remains a dominant characteristic. Amongst a multitude of commercial machine systems, powder bed and powder fed are the prevalent technologies in vogue. In CLAD process, powders are coaxially fed into the laser beam focal point where they are fused and deposited in tracked layers. Notwithstanding the simplicity of the process, the fundamentals of material science that control the product quality and reliability are extremely complex. To illustrate problems facing the technology of metal prints, Laser based CLAD process has been applied for the fabrication and repair of parts. The paper will discuss structural aspects in relation with process parameters and underline challenges still facing additive manufacturing from industrial and basic science perspectives.

655 Mechanical stability of austenite in carbon- and nitrogen-added metastable austenitic stainless steel

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The effect of carbon and nitrogen on the mechanical stability of austenite was evaluated with Md_{30} temperature by using type-304 metastable austenitic stainless steels containing different amount of carbon or nitrogen ranging from 0.05 to 0.2 mass%. The Md_{30} temperature was significantly decreased with increasing both of carbon and nitrogen, and no difference was found in the effect on Md_{30} temperature between carbon and nitrogen for the 0.1 to 0.2%C and N steels. However, as a result of tensile testing at ambient temperature (293 K), the austenite structure was more stabilized by carbon compared with nitrogen. That means the effect of carbon and nitrogen on mechanical stability greatly depends on temperature. The reason of their different effect was discussed in terms of the deformation structure developed in the austenite before starting deformation-induced martensitic transformation, with consideration of stacking fault energy. By tensile testing at 293 K, deformation bands consisting of deformation twins and epsilon-martensite plates were formed in the 0.1%C steel, which leads to a formation of fine martensite grains along the deformation bands. In that case, the growth of martensite is effectively suppressed by the boundaries of deformation bands. On the other hand, dislocation cell structure was formed in the 0.1%N steel in which blocky martensite grains are coarsely distributed in the vicinity of austenite grain boundary, where the martensite could grow rapidly. The difference of the deformation structure of austenite is related with stacking fault energy, which is larger in nitrogen-added steels than in carbon-added steels.

656 EBSD study of delta-processed Ni-base superalloy

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Ni-base superalloys are extensively used in aerospace and power generation industries because of their excellent mechanical properties at elevated temperatures and good corrosion resistance. Typically, these alloys require accomplish critical standards in manufacturing process. In this study, Inconel 718 Ni-base superalloy was subjected to delta-processing technique (DP718), regions with partially recrystallized grains were selected for microstructure analysis. The samples were compressed below an above d solvus temperature at two different strain rates 0.001 s^{-1} and 0.01 s^{-1} . Microstructural characterization was developed using electrons backscattered diffraction technique (EBSD). Kikuchi patterns and orientation relationship of d phase were identified. For samples deformed above d solvus at 0.01 s^{-1} it was observed an increase in the percentage of low angle boundaries (LAB) within deformed grains and a decrease in high angle boundaries (HAB). The CSL grain boundaries preference formation increases with the amount of recrystallization (high temperatures and low strain rates). In addition, it was possible observed the microstructure evolution of Inconel 718 subjected to a delta-processing technique using orientation map images.

657 Influence of niobium content on the hot mechanical behavior of nickel alloys

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Nickel-base superalloys are usually employed for large forged parts in aerospace industry. A comprehensive understanding of their mechanical behavior during hot working is required, especially for manufacturers in order to enhance the in-service properties.

In this context, the first part of the work aims at investigating the mechanical behavior of nickel during hot deformation, with particular emphasis on the influence of niobium additions in solid solution. For this purpose, a series of wrought model alloys including pure nickel and Ni-Nb alloys (Ni-0.01, 0.1, 1, 2, 5 and 10 wt. % Nb) were prepared and deformed by hot torsion at temperatures ranging from 800 to 1000 Celsius degrees and at three (von Mises equivalent) strain rates of 0.03, 0.1 and 0.3 s⁻¹. Afterwards, the key rheological parameters that characterize strain hardening and dynamic recovery were determined through a simple analytical method based on the classical Laasraoui-Jonas constitutive equation, allowing reasonable fit for the flow curves for all studied Ni-Nb alloys. In this way, the effect of niobium solutes on the fundamental mechanisms of deformation was well highlighted.

In the second part, three usual models describing strain hardening and dynamic recovery, referred to as the Laasraoui-Jonas (LJ), Kocks-Mecking (KM), and power law (PW) equations are compared within the range of moderate strains. Transformation formulae are derived, allowing the parameters of one law to be computed from the parameters of any of the two others. The theoretical derivations are illustrated by the specific case of a Ni-Nb alloy in the solid solution domain.

658 Microstructure observation of Al-Mg-Ge alloy aged at 423K and 473K using TEM

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Al-Mg-Ge (0.43at.%Mg-0.2at.%Ge balanced in Al) alloy was fabricated using casting and samples were subjected to homogenization treatment at 623K for 21.6 ks. Solution treatment was conducted at 873K for 3.6 ks and quenched in ice water. Ageing treatment was conducted at the temperatures of 423K, 473K. Hardness was measured after ageing treatment. Microstructure structure of Al-Mg-Ge alloys was observed by Transmission electron microscopy (TEM) / high resolution transmission electron microscopy (HRTEM) with difference ageing times, under-aged, peak-aged and over-aged. In under-aged and peak-aged, it was possible to observe that G.P. zones, random type precipitates, parallelogram type precipitate and β'' phase in Al-Mg-Ge alloy. And β' phase, type-A precipitate and equilibrium phase β were observed in over-aged. The precipitation sequence in Al-Mg-Ge alloy can be described as: S.S.S.S. \rightarrow G.P. zones \rightarrow random type precipitate \rightarrow parallelogram type precipitate, β'' phase \rightarrow β' phase, type-A precipitate \rightarrow equilibrium phase β -Mg₂Ge.

659 Precipitation process of silver nanowire in borosilicate glass by solid-state ion exchange assisted with forward and reverse electric fields

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Silver nanowire (~ 300 nm in diameter) was formed in borosilicate glass by electric field-assisted solid-state ion exchange. In this procedure, there are two stages; a silver doping stage by a voltage application with silver as the anode (referred to as forward) and a silver precipitation stage by a voltage application of opposite direction (referred to as reverse). In the present study, the silver precipitation process was experimentally investigated for glass samples prepared in different application time of reverse voltage. As a result, the crystal growth from silver nuclei on ion-exchanged surface to depth direction in silver doped area was observed. Grown precipitates with dendritic structures formed nanowire networks. According as the application time of reverse voltage increased, the length of nanowire became longer. However, when the tip of the nanowire reached the interface between silver doped and un-doped areas, the nanowire continued to extend along the interface (not to depth direction). After the long application time of reverse voltage, the interface between silver-doped and un-doped areas was filled by silver nanowire networks and they formed a thin silver layer (~ 20 μm in thickness). The embedded silver layer showed low electrical resistance, and therefore, this layer is applicable to a lateral-directional electrical circuit in a glass matrix.

660 Effects of hybrid structures on the stress reduction and thermal properties of the joints in electronics devices

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Electronics devices consist of silicon chips, copper leads, substrates and so on which are jointed to each other with solder, conductive adhesive or other materials. Each coefficient of thermal expansion is different and it causes the strain concentrations and cracks. The solder easily deformed by the difference of the thermal expansion and it relieve the stress on the devices however the epoxy resin of the conductive adhesives are harder. So we suggested the composed joint including the relaxation layers of low elastic material. We compared the share strength and elongation of the epoxy resin joint, silicone rubber joint and the composite joint of the two materials. We also analytically investigated the stress reduction effect of stress reduction varied according to the design of the relaxation layer in the composite joints. The parameters such as the width, height, pitch and the distance of the relaxation layer from the joint edge are investigated. The high relaxation layer close to the joint edge effectively reduced the stress of the joint. The stress reduction effect appeared in the different pitch of the layers. We also measured the shear strength with the composite joint with silicone rubber layer whose size, pitch and height are controlled.

661 Formation of hydrogen by ball milling of Mg and Mg alloy in sea water

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Mg alloy chips coming from machining process are fine and active, and then difficult to recycle by melting. The treatment of Mg chips are one of concerns for expanding the application of Mg alloy, therefore a suitable processing for the Mg chips is demanded. Mg alloys has the disadvantage of poor corrosion resistivity, and easily reacts with water, causing the hydrogen and Mg hydroxide. This hydrolysis reaction is enhanced by the existence of NaCl in water. It is expected that hydrogen as clean energy is produced by the hydrolysis reaction of the Mg chips. In addition, the Mg chips are converted to eco-friendly and safety Mg hydroxide. In this paper, hydrogen is produced by the hydrolysis reaction of the Mg chips generating by machining and sea water with a salt concentration of 0.5M. Furthermore, ball milling is carried out to enhance the formation of hydrogen. The hydrolysis reaction combined with ball milling causes 800ml/g of hydrogen at 2h, which is 8 times larger compared to the reaction without ball milling. A similar result is obtained for the AZ91 chips. It is believed that the combined process of hydrolysis reaction and ball milling is useful for the production of hydrogen with the disposal of Mg chips.

662 Photonic application of diatom frustules

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Diatoms are unicellular aquatic microalgae possessing amazing self-assembled ordered micro- and nano-porous hierarchical silica cell walls called frustules. The quasi-periodic and highly regular pore patterns on the diatom surface are very attractive for applications based on optical and photonic properties of materials. The present contribution reports on pioneering research aimed at explore the multiple scattering and localization of light shown by diatom frustules in order to amplify their photoluminescence in a random laser (RL), as this technology is highly attractive for medical diagnostics and other advanced applications. RL is a special type of laser in which the optical feedback is due to light scattering in an amplifying medium instead of a conventional optical cavity. We have studied a set of selected frustules with different shapes and pore patterns, obtained from diatom cultivation in large scale photobioreactors, for comparative analysis of their random lasing effect in the bioscaffold soaked with organic dyes having luminescence in the visible range. Taking advantage from a multidisciplinary approach combining expertise from biology, physics and materials sciences, relying on high-resolution instrumentation and advanced algal cultivation equipment the results about random laser emission in the composite material were obtained. This will allow going ahead in the research aimed to the application to photonic devices in the field of medicine and medical diagnostic.

663 Novel elastic properties of iron carbide at extreme conditions

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Iron carbide is well known in metallurgy and indeed cast iron has been used for centuries for warfare, agriculture, and architecture. It occurs naturally in iron meteorites and may be an important component of planetary cores. Its elastic properties at ambient conditions give no clue to its behaviour under extreme conditions, where several electronic transitions in iron lead to unusual elastic properties. The presentation will demonstrate how application of novel methodologies to the study of iron carbide under extreme conditions provides new information about the origin of our planet.

664 Design of third generation advanced high strength steels for processing in the continuous galvanizing line

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Third Generation Advanced High Strength Steels (3G-AHSS) are highly promising candidates for automotive weight reduction, thereby improving environmental performance, while maintaining or enhancing passenger safety. However, corrosion protection for these materials are of paramount importance to maintain vehicle structural integrity and meet consumer durability expectations, for which the continuous galvanizing process is amongst the most economical choices. The present contribution will discuss the alloy design philosophy, thermal processing routes and mechanical property results for a series of medium-Mn TRIP steels based on the C-6Mn-Si-Al system such the thermal processing routes to attain target 3G-AHSS properties are compatible with the continuous galvanizing process. In particular, the paper will focus on the microstructural evolution (emphasizing retained austenite (γ_{ret}) volume fraction), the resultant alloy tensile properties and γ_{ret} stability as a function of starting microstructure, intercritical annealing time/temperature, isothermal bainitic transformation time at 460°C and applied strain. These finding will be placed within the context of fundamental studies on phase transformation kinetics, microstructural-property relationships and multi-length scale microstructural investigations.

665 Phase field modelling of bainite formation in low carbon steels

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The formation mechanism of bainite has been object of controversy in the past decades and even today there are two conflicting schools of thought concerning the role of carbon in the

formation of this phase. A considerable number of models in literature are based on the assumption that bainite forms by a rapid process without the need of carbon diffusion and the mechanism of bainite formation is therefore diffusionless-displacive, similar to that of the martensitic transformation. However, recently metallographic evidence of the occurrence of carbon diffusion during bainite formation has been reported, supporting the hypothesis that bainite formation is diffusional-displacive, similar to the formation of Widmanstätten ferrite.

So far the prediction of diffusion-controlled lengthening rate of Widmanstätten plates has been subjected to several uncertainties due to difficulties in modelling the anisotropy of the interface mobility and interface energy in connection with the degree of coherence of the interface. In the proposed models local-equilibrium interface concentrations were adjusted in order to take the effect of the interface kinetics and capillarity into account.

In this work a multiphase-field model was applied to study diffusion-controlled bainite formation kinetics. The model is used in combination with a thermodynamic and diffusivity database, which accounts for the concentration dependence of the carbon diffusivity. The effect of anisotropic interfacial properties on the developed microstructural morphology was analysed in relation to temperature and carbon supersaturation.

666 Exploring the interplay between grain boundary facet junctions and dislocations

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Faceting of grain boundaries is an important manifestation of anisotropy in the dependence of excess interfacial free energy on boundary inclination. It is interesting to consider the interaction of the junctions between adjacent facets and other microstructural elements since this can have a strong influence on the interfacial behavior and properties and on the overall morphological evolution of the boundary. In this presentation, we discuss electron microscopic observations and atomistic simulations illustrating the interplay between facet junctions, dislocations, and interfacial disconnections at metallic grain boundaries, focusing in particular on faceted FCC $\Sigma=3$ and BCC $\Sigma=5$ boundaries. Junctions themselves can exhibit intrinsic dislocation character that arises from translational incompatibilities between the intersecting boundaries. The impact of such effects is well illustrated at $\Sigma=3$ $\{111\}/\{112\}$ facet junctions, where we have shown how the dislocation character of the junctions can strongly influence the core-relaxations of $(1/3)\langle 111 \rangle$ interfacial disconnections. The overall facet junction distribution can also be closely linked to the interfacial dislocation arrangement, particularly if there is an attractive interaction between the junctions and the interfacial dislocations. Since the arrangement and density of interfacial dislocations depends on the deviation from an ideal low-energy reference configuration, such interactions introduce a coupling between the facet length-scale and the misorientation and inclination of the boundary. We explore this linkage through atomistic-scale observations and modeling of $\{310\}/\{210\}$ facet junctions at a $\Sigma=5$ boundary in Fe and through in situ TEM observations of evolving $\{112\}$ facets at a $\Sigma=3$ boundary in Au.

667 Thermomechanical modelling of dissimilar friction melt bonding of AA6061 to dual-phase steel: Prediction of solidification cracking and residual stresses

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Friction Melt Bonding (FMB) is a novel technique to weld aluminium to steel in lap-joint configuration. To carry out the weld, a rotating cylindrical tool showing no pin is pressed against the surface of the steel sheet which is placed over the aluminium one. Heat is generated from the friction and plastic dissipation induced by the tool in the steel plate. This heat does not melt the steel, but locally melts the aluminium in contact underneath owing to the large difference in melting temperatures. The molten aluminium reacts with the solid steel and forms a thin Fe/Al intermetallic layer, responsible for the bonding. In the case of AA6061, of wide use in transport industry, the solidification of the alloy can result in the formation of solidification cracks leading to low joining strengths. In this paper, the RDG criterion developed by Rappaz et al. is used to predict the initiation of the cracks. The prediction requires knowing the thermomechanical loads in the form of thermal gradient and strain rates. Both are estimated from a thermomechanical finite element modelling. The comparison with the experiments shows that it is possible to avoid the formation of hot tears by controlling the process parameters such as the thermal environment. In addition to solidification cracking, residual stresses arising from the high thermal gradients might have a significant impact in the lap-shear strength of the welds. To measure the impact of residual stresses, as-welded and annealed samples are mechanically tested and results are compared to the simulations.

668 Self-consistent thermomechanical model of friction stir welding

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A self-consistent coupled model of thermomechanical processing in Friction Stir Welding (FSW) is presented. The approach is similar to the boundary layer theory in fluid mechanics, and relies on the asymptotic scaling techniques. The results are a set of general closed-form expressions for the maximum temperature reached in the process, the thickness of the shear layer, the shear stress around the pin, the torque and the thermal effect of the shoulder. These expressions are useful to understand FSW in well known alloys such as aluminum-base ones and extrapolate that understanding to other alloys. The methodology consists of determining asymptotic regimes from which to generate scaling laws, and focuses on the most common conditions encountered in practice in FSW, which involve relatively slow translation velocities, relatively high rotation velocities and thin shear layers. In the analysis of this problem, a threadless pin is considered and the effects of the pin and shoulder are separated. The model presented helps understand the torque, temperatures, and deformation history of the material near the rotating pin, but it does not address the mechanical mixing occurring behind the pin. Predictions of torque, temperature, and shear region thickness agree with experiments.

669 B effect on hardenability of high thickness forged steel materials

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To fulfill the industrial demand of forged steels with high mechanical and microstructural requirements coupled with reduced cost, the possibility to reduce the Mo and other elements content has been evaluated. In order to do that the effect of boron addition has been studied (up to 30 ppm) on steel hardenability. The effect of B addition has been investigated on two steel with different chemical composition at laboratory scale. In particular the steel chemical composition has been designed in order to make effective the B addition in terms of hardenability. Two 80Kg ingots have been cast at vacuum induction melting plant and then hot rolled at a pilot mill. The effect of B steel addition on hardenability has been evaluated and compared to that of steel for same application but without B. Results show an improvement of hardenability if 30ppm B are added even if a Mo reduction is performed.

670 Combination of techniques for microstructure characterization as a tool for optimized and new pipe products

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Over the past decades, the complexity of requirements regarding the application of pipe products has steadily increased. This tendency is observed in a wide range of fields, e.g. automotive, energy, and industry. Operating pressure, more hostile environmental conditions (higher or lower temperatures, corrosive-erosive conditions), and lightweight design (i.e. high-strength materials) are some of the challenges that have to be addressed by future products. Steel producers all over the world are therefore developing new products along the entire processing route from steelmaking to pipe production and possible final heat treatment. The microstructure is a key element of advanced steels as it is directly linked to the production parameters and the product properties. For this reason, advanced and combined techniques of microstructure investigation are essential, e.g. high-resolution microscopy, electron backscatter diffraction and nano-indentation. Various approaches of microstructure characterization for the development of optimized and new pipe products in different fields of application will be presented. In the case of high-alloyed steels, light-optical microscopy indicated the existence of small precipitates that have been found to affect corrosion and toughness properties. High resolution electron microscopy showed that these precipitates are on the nm-scale. The quantification of small microstructural features within the heat affected zone of longitudinal welds makes the correlation with the resulting toughness properties possible. The low temperature toughness of the base material for large diameter line pipes is linked to the crystallographic texture via the thermo-mechanical rolling parameters.

671 Microstructural evolution during the heat treatment of laser beam melted AlSi10Mg

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Al alloy AlSi10Mg processed by Laser Beam Melting (LBM) exhibits a much finer microstructure than its cast counterpart due to the ultra-fast cooling rates imposed in the LBM process. One important consequence of this microstructural refinement is that a classical T6 age hardening heat treatment will not have the same effect on LBM AlSi10Mg when compared with cast AlSi10Mg¹. Indeed, a previous study² by the present authors has shown that heat treating LBM AlSi10Mg at 510°C for 6 hours followed by a second isothermal hold at 170°C for 4 hours brought a marked improvement of the yield stress by 30% and of the elongation at break by 220%. However, this was achieved at the expense of a decrease in both hardness and ultimate tensile strength. A better understanding of the underlying phenomena is needed in order to optimize the heat treatment of LBM AlSi10Mg. The present work hence aims at investigating in more depth the microstructural evolution induced upon heat treating LBM AlSi10Mg. Changes in texture as well as in the distribution of Si-rich precipitates (size, morphology...) have been studied, with a particular attention to those changes taking place during the first step of the heat treatment at the higher temperature of 510°C.

1. N.T. Aboulkhair, C. Tuck et al., Metall. Mater. Trans. A, vol. 46 (2015), pp. 3337-3341
2. A. Mertens, O. Dedry et al., Proc. of the 26th international Solid Freeform Fabrication Symposium, Austin (TX), August 10-12 (2015), in press

672 Impact of the confinement on the in-cage dynamics of molecular hydrogen in clathrates hydrates

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We have studied the diffusion dynamics of confined molecular hydrogen as a function of confinement size using nanoporous clathrate hydrates with hydrophobic nanocages of two different dimensions. Clathrates hydrates are nanostructured porous materials, where hydrogen is stored in cages of two different sizes of 0.75 and 0.946 nm, resulting in storage capacity of up to 5.3 wt.% We have found that by varying the size of the pore by only 20 % in the effective radius we can modify the diffusive mobility of confined hydrogen in both directions, i.e. both reducing and enhancing mobility compared to the bulk. In the smaller cages of clathrate hydrates with a mean size of 0.795 nm hydrogen is localized in the center of the cages even at temperatures up to 200K. Moderate increase of the confinement to 0.946 nm leads to the onset of jump diffusion between tetrahedrally shaped sorption sites in large cages with separation length of 2.79 ± 0.23 Å already at $T=10$ K, where bulk hydrogen is frozen at ambient pressure. The differences in microscopic dynamics in cages of two different sizes can help to explain the differences in the macroscopic parameters: trapping of hydrogen in smaller pores matching the molecule size leads to higher temperature stability and reduction of sorption pressure. Similar mechanism could play a role in the processes of electrochemical storage: better storage capacities are observed for smaller cavities, while better ionic conduction has been facilitated by larger pores.

673 Recent advances in real-time studies of metal solidification under external fields

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Fundamental solidification research has been vigorously pursued for over sixty years since Turnbull's classical nucleation theory published in early 1950s. Every fundamental breakthrough since then has underpinned successful development of applied technologies that have provided either step-change or innovation in manufacturing novel materials and goods for industry and society. The remaining scientific and technological challenges in metal solidification processes and many other materials synthesis processes involving phase changes are to understand precisely and quantitatively the real-time evolution of phases at multi-length and time scale, especially for those in a highly dynamic and non-equilibrium conditions, such as solidification under the influence of an ultrasound or electromagnetic field.

Recently, we used the speciality synchrotron X-ray beamlines housed at the Advanced Photon Source, Argonne National Laboratory, USA, the Diamond Light Source, UK and Swiss Light Source, Switzerland to study in-situ the highly transient phase changes phenomena occurred during the metal solidification processes under either an ultrasound field or an electromagnetic field. The ultrafast synchrotron X-ray imaging and tomography techniques were used to reveal how exactly the ultrasonic bubbles break up growing crystals and liquid-solid interface; and how electromagnetic pulses alter the growing directions of primary intermetallic phases and the eutectic phases. The real-time synchrotron X-ray experiments allow the time-evolved 2D and 3D phases to be studied from nanosecond to second time scale, revealing many highly dynamic phenomena in metal solidification processes that cannot be obtained by the traditional experimental methods, and opening a new era for quantitative solidification and materials processing research.

674 Multi-scale modelling of advanced steel processing

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Microstructure evolution during materials processing assumes a critical role in tailoring material properties, e.g. the austenite-ferrite transformations are a key metallurgical tool to improve properties of advanced low-carbon steels. Computational materials science offers now tremendous opportunities to formulate next generation process models which are informed on the atomistic mechanisms of microstructure evolution thereby reducing the number of empirical parameters that are typically used in current process models. The challenges and opportunities of implementing advanced computational materials science tools into process models will be critically reviewed. Microstructure evolution kinetics depends on interface migration rates that can be significantly affected by alloying elements, e.g. Mn, Mo and Nb in steels. Here, an approach is illustrated that links atomistic scale models for the solute-interface interaction with phase field modelling to describe the formation of microstructures with complex morphologies. The overall status of the multi-scale modelling approach will be analyzed for intercritical annealing of dual-phase steels and the rapid heat treatment cycles in the heat affected zone of line pipe steels. An outlook will be provided for developing a phase field based through process model from casting to annealing.

675 Quench sensitivity of Al-Mg-Si and Al-Zn-Mg-Cu alloys. Part 1 experiments

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Quenching can be considered the most critical step during the strengthening heat treatment process age hardening of Aluminium alloys. To obtain optimal technological results, parts should be quenched as fast as needed to achieve maximal strength after ageing. Besides quenching should be done as slow as possible to minimise distortion and residual stresses. These two contrary requirements are tune if cooling is done with the upper critical cooling rate. This defines as the slowest cooling at which any precipitation is suppressed completely and a fully supersaturated solid solution is retained after quenching. The affinity to “lose” solute atoms to coarse quench-induced precipitates is called quench sensitivity. Those precipitates do not contribute to strengthening and reduce the amount of atoms available to form strengthening precipitates during ageing. The quench sensitivity, i. e. the precipitation behaviour during cooling from solution annealing can be investigated by in-situ measurements with differential scanning calorimetry (DSC). As precipitation is driven by diffusion, a very broad cooling rate range is required to gain full understanding of the relevant processes DSC nowadays covers the whole cooling rate range relevant – from slow cooling close to equilibrium transformation up to overcritical fast quenching. Combining DSC measurements with hardness testing and microstructural investigation in the length scales between some 100 μm down to some few nm nowadays allows to understand the mainly driving factors of the quench sensitivity of Al alloys, resulting in complete continuous cooling precipitation (CCP) diagrams - which moreover allows to set-up predictive physically based models.

676 Effects of a? intercritical and? single-phase annealing on texture evolution in cold-rolled dual-phase steel sheets

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Dual-Phase (DP) steel sheets composed of both soft ferritic and hard martensitic phases have been typical advanced high strength steel sheets applicable to a variety of automobile parts. The crystallite texture of the steel sheet is one of the important factors that influence the press formability. The texture of martensite itself in DP steels, however, has not been discussed since the texture was mostly measured by X-ray diffraction method, which did not distinguish the texture of martensite from that of ferrite. The objective of this study is to investigate the effects of the intercritical and gamma single-phase annealing on the texture evolution in DP steels by newly developed analyzing technique using Electron Back-Scatter Diffraction (EBSD) to obtain the textures of each phase separately. The chemical composition of the steel used was 0.1%C-1.2%Si-2.3%Mn-0.1%Ti (mass%). The pre-annealing was carried out at 948K below A_{c1} temperature to finish the recrystallization after hot and cold rolling in order to focus on transformation texture evolution itself. The steels were finally annealed at 1123K in the intercritical region and 1223K in the gamma single-phase region to obtain DP microstructures with approximately 40% volume fraction of martensite. The overall texture including

martensite in case of the intercritical annealing was similar to the initial texture before the annealing, while the texture in case of the gamma single-phase annealing was randomized. Moreover, our unique EBSD analysis clearly showed that the textures of martensite themselves were close to those of ferrite in the two annealing conditions.

677 Characterisation of hydrogen embrittlement of metastable austenitic stainless steel using micro-tensile testing

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With increasing requirements to reduce carbon dioxide emissions, hydrogen has been increasingly used as an energy carrier. Stable austenitic stainless steels such as a type 316L have been used in the hydrogen-relating industry because of their low susceptibility to hydrogen embrittlement (HE). While reducing the content of alloying elements in these steels is important from an economic viewpoint, the reduction of the nickel content particularly decreases the stability of austenitic phase below room temperature. Metastable austenitic steels with low stabilities of the austenitic phase such as a type 304 suffer from a severe HE, which is characterised by quasi-cleavage and flat-faceted features. The formation of quasi-cleavages may be related to deformation-induced martensitic transformations. However, the role of martensite in the mechanism of quasi-cleavages in metastable austenitic steels is still unclear. Therefore, the objective of this study is to elucidate the formation of quasi-cleavages in a type 304 metastable austenitic stainless steel from a crystallographic perspective. We employed micro-tensile testing that enables us to analyse the deformation behaviour of small single crystals with defined crystallographic orientations. Electron backscatter diffraction analyses of the deformation microstructures revealed that a hydrogen-induced quasi-cleavage occurred along the martensite/austenite habit plane, suggesting that excess hydrogen generated by the martensitic transformation plays a crucial role for the HE. Also, we will discuss the effect of grain refinement on the HE in an ultrafine-grained regime.

678 Tailored properties for metallic implant materials

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Ti and Ti alloys: Pure titanium (CP-Ti) is a commonly used implant material especially in dentistry. In that specific biological environment the high strength alloy Ti6Al4V-ELI is rarely used due to the possibility of interactions between the problematic elements aluminum and vanadium with the human body. Nevertheless, for many dental applications a higher strength than that of CP-Ti is requested. With the use of the further developed method of Equal Channel Angular Pressing (ECAP) and tailored optional post-processing we achieved in CP-Ti ultimate tensile strengths > 900 MPa, which is comparable to the Ti6Al4V alloy, as well as a desirable very high ductility of > 20%. Moreover, preliminary tests with osteoblast cells on the ECAP surface show promising results. Mg alloys: Low-alloyed Mg is the most promising metallic material for biodegradable implants. Recently we have succeeded in developing Mg alloys containing only very small amounts (< 1wt%) of the dietary elements Ca and Zn, which show

slow degradation. Using ECAP especially the mechanical properties could be improved towards strength values that are comparable to those of permanent implant materials like CP-Ti. We show latest results of these alloys regarding their mechanical and corrosive properties in dependence of their processing route. The right choice of alloy and processing parameters allows tailoring the mechanical and degradation properties in a wide range. In conclusion, ECAP-processed biomaterials are very promising candidates for applications in highly loaded implants, for longer durability in the biological environment and for the miniaturization of implants while maintaining strength.

679 Technical challenges in narrow-gap root pass welding during tandem and hybrid laser-arc welding of a thick martensitic stainless steel

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As part of a collaborative program to develop advanced manufacturing processes for next-generation hydraulic turbines, this study investigated the technological challenges for joining 25-mm thick martensitic stainless steel (MSS) plates using tandem and hybrid laser-arc welding. Although candidate materials for the intended application typically include wrought AISI 415 and cast CA6NM, a martensitic 410 stainless steel (SS) was especially selected in this study due to its greater crack sensitivity. A narrow-gap groove was designed to minimize the amount of 410NiMo filler metal required to fill the groove using a multi-pass single-sided welding technique. All the welding trials were performed using a 5.2 kW fiber laser. The root-pass quality was characterized in terms of weld bead geometry, defects and microstructure. The main technical challenges observed for the root pass were lack of penetration, lack of fusion and cracking, as detailed in this work.

680 Irradiation swelling simulation of welded joints of 18Cr10NiTi austenitic stainless steel

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Pressure vessel internals (PVI) of nuclear reactor WWER-1000 are made of 18Cr10NiTi austenitic stainless steel. Pores formation and growth take place under conditions of intensive irradiation. In addition, segregations of nickel are formed at the grain boundaries and on pores free surfaces, resulting in exhaust of nickel in austenite matrix. Gamma to alpha transformation occurs leading to further increase in metal volume when the content of nickel in the matrix is less than 5%. These processes are known as irradiation swelling and result in material embrittlement. There are several mathematical models describing the increase in material volume during irradiation swelling. The simplest of them are temperature-dose dependencies of swelling. Novel models account for the effects of stressed state.

Irradiation accelerates thermal creep and irradiation creep strain rate depends on the irradiation swelling. PVI of WWER-1000 are welded structures and tensile residual welding stresses provide for increase of swelling, so we may expect higher values of swelling in PVI welds. Ductile to brittle transition takes place in the material when values of swelling exceed 7%.

Sharp decrease in the tensile strength of the material occur if values of swelling exceed 15%. The report presents the main results of the simulation of swelling and stress-strain state of WWER-1000 core baffle in long-term operation. Problems of estimation of irradiation swelling and deformation of structure are solved taking into account the residual welding stresses.

681 Dependence of frequency and electric conductivity on current distribution in SPS process

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In this study, the FEM analysis of sintering current distribution in the sintering container was carried out to clarify the influence of frequency of current and electric conductivity of materials on the sintering behavior of SPS process. It is considered that the internal current flow through sintering materials and the current distribution in the sintering container strongly influences the sintering behavior on SPP process.

The pulsed current applied to sintering container and materials contains the high frequency components. The current distribution in the sintering container and materials depend on the frequency spectrum of sintering current. Furthermore, frequency spectrum also depend on the type, current pattern and duty ratio of pulsed power supply by fast Fourier transform analysis. And the effect of the frequency will become remarkable in sintering of electrically conductive materials and the ferromagnetic materials according to the skin effect.

From FEM analysis, the current in the container is localized, and becomes difficult to reach the surface of sintering material with the increase in the frequency of sintering current. In addition, the current distribution is also influenced at the conductivity of material and container. The current distribution in the material in which the conductivity changes seems to greatly change with the progress of the sintering. When the electrically conductive material was assumed, the current exists locally on the surface of material with the increase in the conductivity. The density and total amount of current also changes with the increase in the frequency and conductivity of materials.

682 Microstructure evolution during LCF of a 10% Cr steel at room temperature

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The influence of cyclic loads on microstructure and hardness of a 10%Cr steel with 3%Co and 0.008%B was examined at room temperature and total strain amplitude of $\pm 0.25\%$ and $\pm 0.6\%$. LCF curves exhibit a stress peak after a few cycles. Then stress tends to decrease monotonically with number of cycles that is indicative for material softening. Structural characterization was performed in initial state, after hardening, in half-life and failure conditions. Hardening is attributed to an increase in dislocation density; no changes in lath/subgrain size were observed. Further increase in the number of cycles leads to decreasing dislocation density, lath coarsening and subgrain growth. At half-life, +30% and +16% increase in lath thickness takes place, under

cyclic test with $\pm 0.25\%$ and $\pm 0.6\%$, respectively, that comprise 60% of the value of lath coarsening up to failure in both cases. Gliding lattice dislocations are trapped by lath boundaries. However, no knitting reaction between dislocations comprising lath boundaries and trapped lattice dislocation occurs and the width of these boundaries increases. At total strain amplitude of $\pm 0.25\%$, further cyclic loads produce insignificant changes in microstructure, while at total strain amplitude of $\pm 0.6\%$, the knitting reaction leads to the transformation of lath boundaries to subboundaries followed by subgrain coarsening. No effect of cyclic load on a dispersion of secondary phase particles was found. Relation between microstructural evolution under LCF and hardness is considered. The study was financially supported by the Russian Science Foundation, Project no. 14-29-00173.

683 The influence of the deformed texture components on Cube-oriented grains formation during recrystallization of AA1050 aluminium alloy

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The microstructure and texture evolutions during early stages of recrystallization of a commercial AA1050 alloy have been characterized to elucidate the mechanism of cube-oriented grains formation and the resulting texture evolution. Samples were plane strain compressed (PSC) and equal channel angular extruded (ECAE) and then shortly annealed. Particular attention was paid to the description of the misorientations across the recrystallization front and on possible dislocation mechanisms during the primary recrystallization. The textures were measured with the use of scanning electron microscopy equipped with an electron backscattered diffraction facility and X-ray diffraction. In the case of PSC samples, the strong cube texture after recrystallization results from both the increased density of the particular $\langle 111 \rangle$ poles of the four variants of the S orientation and the $\sim 40^\circ$ ($\sim \langle 111 \rangle$)-type rotation. The first mechanism transforms the S^{def} -oriented areas into S^{rex} ones, whereas the second the near S-oriented, as-deformed areas into near cube-oriented grains. After recrystallization of the ECAE deformed samples, the texture of the recrystallized grains was dominated by two twin related $\{110\}\langle 001 \rangle$ and $\{221\}\langle 114 \rangle$ groups of orientations. The cube grains were only sporadically detected by the SEM/EBSD system. Finally the mechanism of crystal lattice re-orientation during recrystallization based on the thermally activated dislocation movement was proposed.

684 Mechanisms of microstructure evolution during hot deformation of a 20%Cr ferritic stainless steel

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Hot rolling of ferritic stainless steels consists of a succession of deformation and inter-pass steps involving continuous cooling. As no phase transformation occurs in these steels, the refinement and homogenization of the coarse as-cast structure is expected to proceed by

(continuous) dynamic and post-dynamic recrystallization. Final properties are thus strongly dependent on the microstructure evolution during hot rolling. A detailed understanding of recrystallization mechanisms is therefore necessary in order to optimize process conditions. The hot deformation behavior of a 20%Cr ferritic stainless steel was studied thanks to high temperature plane strain compression tests. Detailed EBSD investigations were performed to characterize microstructures after deformation and during isothermal holding for various straining conditions. The post-deformed states are well described by the concept of continuous dynamic recrystallisation. Post-dynamic recrystallization kinetics were measured and mechanisms of new grain nucleation and growth were characterized. Several striking results of these experiments will be discussed, including the non-monotonic influence of strain and temperature on recrystallisation kinetics, and the significant grain growth before recrystallization completion. The texture evolution is also described as a function of deformation conditions and compared with crystal plasticity simulations, highlighting the interaction between microstructure and texture evolution.

685 In situ phase investigations of X20Cr13 high Cr steel

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Understanding the microstructure of materials during heat treatment is necessary to optimize process parameters for the improvement of mechanical and chemical properties. In situ observation methods are suitable for a time resolved monitoring and analysis of e.g. phase transitions and the growth of precipitates or phases during the heat treatment. Comparing volume sensitive dilatometer- with relatively surface sensitive high-temperature X-ray diffraction (HT-XRD) experiments leads to different results and thus different interpretations. Comparative investigations were carried out on a ferritic/martensitic high Cr steel (X20Cr13). By applying the same heat treatment cycle, results from the dilatometer show that a martensitic transformation occurred at about 573K (300°C) in the dilatometer, whereas HT-XRD showed the reconstructive ferritic transformation at around 1018K (745°C). Results from the EBSD investigation showed the formation of ferrite grains at the surface during the heat treatment whereas the bulk revealed a martensitic structure. Due to the fact that XRD is relatively surface sensitive (in this case the signal was gained at a depth of less than 4 µm) the HT-XRD showed surface effects and therefore the ferritic transformation. These investigations clearly demonstrated that surface effects must be considered when interpreting results from in-situ experiments.

686 Anisotropy in microstructure and mechanical properties of superalloys (Inconel718) by selective laser forming (SLF)

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Nickel-based Superalloy (IN718C) powders were formed by selective laser forming (SLF). Normally, large columnar dendrites appear epitaxially along the deposition direction in SLF. To evaluate the anisotropic mechanical behavior of IN738LC alloys due to layer-wise build up,

specimens were built with their cylinder axis (loading direction) oriented either parallel to the building direction, or perpendicular to the build direction. Specimens were investigated either under the as-built or as-heat-treated, and compared to the wrought material. The microstructural anisotropy in the specimens was analyzed by EBSD and EDX texture analysis methods, and also checked with anisotropic material behavior by tensile test at room temperature. The Young's modulus and tensile properties (strength and elongation) determined were significantly lower parallel to the building direction than perpendicular to the building direction. These anisotropy was seemed to be not due to the measured crystallographic preferred orientations, but to the some defects such as pores.

687 Heterogeneous nano-structures in austenitic and duplex stainless steels developed by heavy cold rolling and the specific mechanical properties

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SUS316LN, SUS310S austenitic and duplex stainless steels were cold rolled up to 92%. Ultrafine grained (UFGed) heterogeneous structures, a mixture of lamellar, micro-bands and mechanical twins, were homogeneously developed. The average lamellar spacing was less than 50 nm irrespective of the alloys. The as-rolled samples showed quite high ultimate tensile strength (UTS) over 1.5 GPa with ductility around 10%. These mechanical properties were comparable with those obtained by multi-directional forging [1]. Therefore, UFGed structure and superior mechanical properties could be achieved by simple cold rolling, i.e., without severe plastic deformation. When the samples were aged at 773 K, strengths were further raised. While the tensile property showed anisotropy, UTS over 2.6 GPa with ductility of about 5% was constantly attained. Such superior balance of mechanical properties should be attributed to the UFGed heterogeneous structure. That is, dense distribution of twins and micro-bands contributed to suppression of sharp texture evolution and early rupture to induce superior balance of UTS and ductility. Acknowledgments: This research was supported by Japan Science and Technology Agency (JST) under Industry-Academia Collaborative R&D Program "Heterogeneous Structure Control: Towards Innovative Development of Metallic Structural Materials". Reference: [1] Y. Nakao, H. Miura, Mater. Sci. Eng. A 528, (2011) 1310.

688 The effect of Si and Mn to phase decomposition of Cu-Zn alloys during annealing process

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beta-prime phase in Cu- 47.7wt.%Zn alloy decomposes to alpha phase during annealing processing and it increases hardness. Decomposition sequence, from beta-prime single phase to alpha phase has been reported as: beta -prime → alpha_{9R} → alpha_{fcc} reported that Cu-Zn alloy can be strengthened by decomposition controlling by addition of Si and Mn. However, the effect of alloy elements, Si and Mn is not clearly revealed yet. In this study, to understand the effect of alloying elements of Si and Mn, the fixed Zn concentration in Cu-41.7wt.%Zn- (Si, Mn) alloys were prepared and investigated. Alloys were prepared using casting method and they

were subjected homogenization treatment at 873K for 36ks. Solution treatment were conducted at the temperature of 1103K for 0.6ks then quenched using chilled water. Alloys were subjected to annealing treatment at the temperatures of 423K, 473K, 523K and 573K. Vickers microhardness measurement was conducted to understand mechanical property. Microstructure observation was carried out using SEM and TEM.

689 Titanium oxide coating on Ti-based alloys for dental application

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Rutile-type TiO_2 is common white oxide and widely used for pigment, sunscreen, makeup and so forth. We have been studied that high temperature oxide formation on specific Ti-based alloys such like Ti-Nb-Ta-Zr in terms of color tone and exfoliation resistance, which can form white robust oxide layer by heat treatment. Color tone investigation by spectrophotometric colorimetry showed that brightness of the oxide was as almost same as that of opaque resin of a resin facing dental crown. The oxide consisted of Rutile TiO_2 and TiNb_2O_7 . The maximum exfoliation stress was almost as same as an epoxy adhesive (~ 70 MPa), the value of which depended on layer thickness and surface roughness. From the results of fracture surface observation, interfacial fracture between oxide layer and metallic substrate occurred, thus oxide layer is strong against crack initiation. TEM observation at the interface between metallic substrate and oxide indicated that continuous grain growth and dense oxide formation contributed to its excellent exfoliation resistance.

690 Hydroxyapatite formation on Type 316L stainless steel and zirconium by cathodic polarization with pulse current

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Cathodic polarization with pulse current was performed to facilitate the hydroxyapatite (HAp) formation on Type 316L stainless steel and zirconium. The cathodic current was modulated as square wave; the lower, I_{on} : -100 mA/cm^2 and the higher, I_{off} : 0 mA/cm^2 , with t_{on} : 20 ms (pulse on time) and t_{off} : 40 ms (pulse off time) in the solution containing $1.67 \text{ mM K}_2\text{HPO}_4 + 2.5 \text{ mM CaCl}_2 \cdot 2\text{H}_2\text{O} + 138 \text{ mM NaCl}$ for 1 h. Cathodic polarization with constant current (-100 mA/cm^2) was performed in the same solution for comparison. The obtained surface layer was characterized by XRD, SEM and FT-IR. Needle-like depositions were observed on Type 316L stainless steel and Zr that were polarized by cathodic current with/without modulation and were identified as HAp containing carbonate. The deposition formed by constant current was more aggregated than that formed by pulse current. The polarized samples and as-polished ones were immersed in simulated body fluid (SBF) to examine the HAp formation. Ball-like particles of newly HAp formed and totally covered the surfaces of the polarized Zr after 3 days immersion in SBF, whereas no particle was observed on as-polished Zr after immersion in SBF for 7 days. On the other hand, the amount of newly formed HAp on the polarized Type 316L stainless steel

was less than that on the cathodic polarized Zr.

691 Dislocation density of ultrafine grained Cu fabricated by severe plastic deformation

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Ultrafine-grained (UGF) metals having the grain size less than one micrometer fabricated by severe plastic deformation (SPD) processes have several times higher strength compared with the coarse grained metals having the grain size more than several tens micrometers. It had been qualitatively said that UFG have high dislocation density in addition to the high density of grain boundary since the SPD processes are the plastic deformation process. Although there are reports about dislocation density evaluated by different techniques, the report about the comparison of techniques which can provide dislocation density using a unique UFG sample is rare. In the present study, UFG Cu was fabricated by accumulative roll bonding process which is one of the SPD processes. The microstructure characterization, especially the evaluation of dislocation density, was performed using scanning / transmission electron microscopy (S/TEM), X-ray diffraction (XRD), differential scanning calorimetry (DSC) and electrical resistivity. As a result, the dislocation density of UFG-Cu increases after first ARB cycle, and then, the values stay almost constant with increasing ARB cycle number. Although we use the different techniques, the evaluated values of dislocation density are within an order of magnitude, which is between the middle of 10^{14} m^{-2} and the beginning of 10^{15} m^{-2} .

692 Hydrogen production via thermochemical and electrochemical hybrid process by sodium alloy

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Hydrogen production is an important technology to establish a sustainable energy system based on hydrogen as an energy carrier. We propose a hydrogen production technique by thermochemical and electrochemical hybrid process using redox reactions of sodium alloys. This process is composed of three kinds of reactions, which are thermochemical decomposition of sodium alloys (Na-M), electricity generation by the electrochemical process using sodium (Na) and metal (M) electrode materials, and water electrolysis process. In the case of sodium-tin (Na-Sn) alloy, the thermochemical decomposition completely proceeded below 500 °C within 1 h. In addition, corrosion of the Na-Sn alloy was drastically weaker than those of Na oxides and peroxide. The electrochemical process between Na and Sn has been studied as a Na ion battery using liquid electrolyte so far, suggesting that basic chemical reactions are well understood. Thus, it is expected that the control of electrochemical process below 500 °C is not difficult even though the solid electrolyte such as alumina is used. The water electrolysis is well established technique, and hydrogen can be produced with high efficiency. Therefore, this hybrid process is recognized as a potential hydrogen production technique operated below 500 °C, which can be obtained from solar heat and/or unused heat.

693 Characterization of light emission from Si quantum dots with Ge core

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Currently, light emission from Si-based nanostructures including Si quantum dots (QDs) has gained considerable interest because of its potential advantage in combining both photonics and electronics on a single chip [1,2]. The enhancement of the light emission has been conducted intensively with deliberate approaches which include not only the confinement of injected carriers but also the use of strained structures and impurity doping. So far, We have reported the formation of Si-QDs with Ge core on ultrathin SiO₂ by controlling the thermal decomposition of pure SiH₄ and 5% GeH₄ diluted with He alternately, and demonstrated unique charged storage characteristics of an individual Si-QD with a Ge core, and confirmed type II energy band alignment between Si-clad and Ge core, that is, electrons are stored in a Si clad and holes in a Ge core, from the surface potential measurements before and after electron injection and emission by means of AFM/Kelvin probe force microscopy [3]. In this presentation, our recent achievements on high-density formation and characterization of Si-QDs with Ge-QDs on SiO₂ have been reviewed. And we focus on how photoluminescence (PL) properties from Si-quantum dots are changed with embedding of Ge core and P-doping to Ge core. Acknowledgements. This work was supported in part by Grant-in Aid for Scientific Research (S) No. 15H05762 of MEXT, Japan. References [1] R.J. Walters et al., Nature Mat. 4 (2005)143. [2] K. Makiyara et al., Jpn. J. Appl. Phys. 51 (2012) 04DG08. [3] Y. Darma et al., Nanotech. 14 (2003) 413.

694 Thermal conductivity of cubic boron nitride particle dispersed Al matrix composites fabricated by SPS

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Cubic boron nitride (cBN)-particle-dispersed-aluminum (Al) matrix composites were fabricated in solid-liquid co-existent state by Spark Plasma Sintering (SPS) process from the mixture of cBN powders, Al powders and Al-5 mass%Si powders. The microstructures and thermal conductivities of the composites fabricated were examined. These composites were all well consolidated by heating at a temperature range between 798 K and 876 K for 1.56 ks during SPS process. No reaction at the interface between the cBN particle and the Al matrix was observed by scanning electron microscopy for the composites fabricated under the sintering conditions employed in the present study. The relative packing density of the Al/cBN composite fabricated at a pressure of 300 MPa was higher than 99% in a volume fraction range of cBN between 35 % and 50 %. Thermal conductivity of the Al/cBN composite increased with increasing the cBN content in the composite in a volume fraction range between 35 and 45 vol%. The highest thermal conductivity was obtained for Al-45 vol%cBN composite and reached 305 W/mK. The coefficients of thermal expansion of the composites were a little higher than the theoretical values estimated by the upper line of Kerner's model, indicating the bonding between the cBN particle and the Al matrix in the composite is weak a little.

695 Influence of severe plastic straining on mechanical properties of an AA5024

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Effect of severe plastic straining (SPS) on mechanical properties of an Al-4.57Mg-0.35Mn-0.2Sc-0.09Zr (in wt%) alloy subjected to equal channel angular pressing (ECAP) at a temperature of 300°C to a strain of $\epsilon \sim 12$ followed by cold rolling (CR) was studied. ECAP provides increase in the yield stress (YS) from 260 to 315 MPa, due to extensive grain refinement, mainly. Ductility of 23 pct. remains unchanged with increasing number of passes. The cold rolling with total reduction 90 pct. leads to the growth in the yield stress (YS) and ultimate tensile strength (UTS) values 470 and 580 MPa, respectively, while ductility decreasing to 7 pct. This increment is attributed to an increase in density of lattice dislocations and the formation of numerous low-angle boundaries owing to extensive deformation banding within interiors of initial fine grains. The relationship between mechanical properties and microstructure, and mechanisms of strengthening are discussed in some detail.

696 Otoacoustic emissions as a promising diagnostic tool for the early detection of mild hearing impairment. Technical advances in acquisition, analysis and modeling

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Otoacoustic emissions are a by-product of the active amplification mechanism located in the cochlear outer hair cells, which provides high sensitivity and frequency resolution to human hearing. Being intrinsically sensitive to hearing loss at a cochlear level, they represent a promising non-invasive, fast, and objective diagnostic tool. On the other hand, the complexity of their linear and nonlinear generation mechanisms and other confounding physical phenomena (e.g., interference between different otoacoustic components, acoustical resonances in the ear canal, transmission of the middle ear) introduce a large inter-subject variability in their measured levels, which makes it difficult using them as a direct measure of the hearing threshold using commercially available devices. Nonlinear cochlear modeling has been successfully used to understand the complexity of the otoacoustic generation mechanisms, and to design new acquisition and analysis techniques that help disentangling the different components of the otoacoustic response, therefore improving the correlation between measured otoacoustic levels and audiometric thresholds. In particular, nonlinear cochlear modeling was able to effectively describe the complex (amplitude and phase) response of the basilar membrane, and the generation of otoacoustic emissions by two mechanisms, nonlinear distortion and linear reflection by cochlear roughness. Different phase-frequency relations are predicted for the otoacoustic components generated by the two mechanisms, so they can be effectively separated according to their different phase-gradient delay, using an innovative time-frequency domain filtering technique based on the wavelet transform. A brief introduction to these topics and some new theoretical and experimental results are presented and discussed in this study.

697 Grain boundary dynamics and grain rotation in aluminum bicrystals

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The dynamic properties of grain boundaries and their anisotropy can be essential for microstructure development during grain growth in polycrystals. The experiments and molecular statics simulations demonstrated that the inclinational anisotropy of grain boundary energy is not restricted to the “special” low Sigma CSL boundaries only, but also applies to low-angle tilt boundaries with low index rotation axis: boundaries with misorientations lower 15° did not attain a curved shape (remained straight or formed facets) at any temperature and, thus, did not move under a capillary driving force. An impact of the energy anisotropy on the evolution of the boundary shape and boundary kinetics is confirmed by molecular dynamics simulations. Further simulations showed that the shrinkage of grains with pure tilt boundaries was accompanied by grain rotation. In contrast, grains with the tilt-twist boundaries did not rotate during their shrinkage. An analysis revealed that the boundary structure is crucial for the observed rotational behavior.

698 Thermomechanical bonding between metallic glasses and various die materials

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Thermoplastic forming is a promising method of fabricating metallic glasses (MGs) products with complex shapes, avoiding the difficulties encountered in other manufacturing processes, such as casting that requires high cooling rate and machining that causes catastrophic cracking. However, bonding between dies and MGs during thermoplastic forming restricts its production. This paper studied the bonding between MGs (La-based and Zr-based) and various widely-used dies (electroless Ni-P, Al_2O_3 , Si and Si_3N_4) in the thermoplastic forming process. The bonding behaviour for each case was investigated using optical microscope, scanning electron microscope (SEM), as well as electron dispersive spectroscopy (EDS). Two parameters were introduced that correlate adequately with the bonding results. It was found that bonding inevitably occurred between MGs and dies, but the extent of bonding varied significantly with die materials due to the pivotal effect of wetting, chemical reaction and diffusion. Among the die materials used in the present study, Al_2O_3 had the best performance, attributed to the higher bonding dissociation energy of its constituent bonds, along with its comparatively low surface energy. The interface of the Si die and the La-based MG formed a distinct nanometric layer, indicating the chemical bonding between the die and La-based MG. Moreover, the diffusion between the La-based MG and Si die atoms was confirmed by using EDS mapping analysis. The present study concludes that the surface energy and bonding dissociation energy of the constituent bonds can be used as proper indicators for selecting die materials in the thermoplastic forming of MGs.

699 Synthesis and corrosion effect on A₂Ni₇ intermetallics used as electrode material for Ni-MH batteries (A= La, Gd, Y, Sm or Mg)

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To improve the properties of Nickel-Metal Hydride batteries, new promising A₂Ni₇-type alloys are developed to replace the commercial ANi₅ ones used as negative electrode material (A=mix of rare earths). These A₂Ni₇ alloys can reach 400 mAh.g⁻¹ by partially substituting the rare earth with Mg¹. Beside their high capacity, corrosion resistance of these alloys in KOH concentrated electrolyte is of major importance for the battery life duration. The corrosion kinetic depends on temperature, electrolyte concentration (calendar corrosion) and cycling conditions. It is thus essential to understand the corrosion processes as a function of the alloy composition of the negative electrode, in order to optimize both capacity and life duration. In this study, model binary A₂Ni₇ alloys were synthesized using induction melting and powder metallurgy. The calendar corrosion of such alloys in alkaline medium (KOH 8.7M) was investigated for soaking time ranging from 6 hours to 16 weeks. The corrosion processes were also electrochemically investigated from a few to several tens of cycles. Structural and elemental characterization techniques (X-ray diffraction, Raman micro-spectroscopy, scanning and analytical transmission electron microscopies) have been used to identify the corrosion products that developed at the surface as a function of time. The influence of the rare earth or magnesium composition on the corrosion product as well as the corrosion rate are discussed²⁻³ and compared to previous works on LaNi₅-based materials⁴.

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700 Hemp Nanofibrils Reinforced Polycaprolactone Composites

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Hemp fibers were pre-treated with alkaline and acid solutions to eliminate the matrix made up principally of lignin, hemicellulose and pectin embedding the cellulosic fibers. Then a cryo-crushing with liquid nitrogen (to further reduce fibers length and size) was performed. Finally, a 30 minutes ultrasonic treatment, (power 750 W, frequency 20 KHz, time 30 minutes), was carried out on a water suspension of cellulosic material to obtain micro and nanofibrils. To avoid the issue of the aggregation of the fibrils after drying sol-gel treatments were studied to decrease the formation of intramolecular hydrogen bondings. Two different sols were prepared by: a)acidic hydrolysis of titanium (IV) isopropoxide as a precursor stirred with 40 ml of

isopropyl alcohol and 5 ml HCl 0.01M for 24 h at room temperature, adding to the mixture hemp fibrils in a ratio 10:1 and 2:1 compared to titanium (IV) isopropoxide; b) acidic hydrolysis of dimethyldiethoxysilane as a precursor stirred with 40 ml of isopropyl alcohol and 15 ml HCl 0.01M for 24 h at room temperature, adding to the mixture hemp fibrils in a ratio 10:1 and 2:1 compared to the organosilicon compound. The solutions were then centrifugated at 3500 rpm for 30 minutes, then rinsed and dried in an oven at 105 °C for 4 hours. Polycaprolactone composites containing 5% of dried fibers were compounded using a Mini extruder. Extruder's conditions were 80 °C and 60 rpm for 10 min. After drying at 60 °C until constant weight, the composites were press-molded in a hydraulic press to obtain films for dynamic mechanical analysis. Thermal and morphological characterization of the materials produced were carried out.

701 Dynamic recrystallization mechanism of coarse grained oxide dispersion strengthened ferritic steel

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The ferritic oxide dispersion strengthened alloys are manufactured using the mechanical alloying process. The development of a coarse grained microstructure during the recrystallization of ferritic ODS steels have been noted and discussed by a number of authors but, the mechanism of grain control remains uncertain. Recent work has emphasized the large influence of non-uniformities on the development of the recrystallized microstructure. This has the effect of greatly reducing the recrystallization temperatures and of enhancing the nucleation of recrystallization thereby giving a fine-grained microstructure. The heterogeneity can be introduced by having a non-uniform starting microstructure or by introducing a non-uniform plastic strain in the sample. In this study, an alternative approach to generate fine grain microstructures is explored. The mechanism of dynamic recrystallization as a function of temperature and strain rate is investigated. The effect of strain, strain rate and temperature on a FeCrAl ODS ferritic steel were investigated. Hot compression tests were performed in the temperature range of 900 -1100 °C and strain rate range of 0.1 – 10 s⁻¹. At high temperature and high strain rate, this alloy undergoes discontinuous dynamic recrystallization, whereas at lower temperature and strain rate, continuous dynamic recrystallization occurs.

702 The influence of filler metal composition on microstructural evolution and isothermal solidification during transient liquid phase bonding of nickel

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The present work involved a thorough microstructural, chemical and calorimetric analysis on braze joints formed between a pure nickel substrate and two commercial Ni brazing foils in-situ via differential scanning calorimetry (DSC). Transient liquid phase bonding (TLPB) results as a consequence of boron and silicon additions in the brazing foils which act as melting point

suppressants, thus enabling the formation of a transient liquid phase at temperature below the melting point of the Ni substrate. The study involved interrupted tests as a means of preserving the microstructure formed after being heated to various temperatures for a specified duration. Cyclic tests were also performed which enabled the in-situ monitoring of liquid content with solidification time. Characterization included; optical microscopy, scanning electron microscopy and x-ray diffraction. The results indicated that the liquid present in both systems was a consequence of a ternary eutectic phase (Ni-Ni₃Si-Ni₃B), which was progressively depleted with the simultaneous coarsening and evolution of the epitaxial Ni borides and silicon rich gamma nickel. This work greatly supported the development of a fundamental understanding regarding TLPB in super alloys and will support further research into the more practical, and more complex commercial systems such as Inconel 625 and 718, where it is expected to provide insight into issues related to detrimental intermetallic formation and improving the overall TLPB process. [oral presentation]

703 The effect of post processing heat treatments on the microstructure of the nickel-based superalloy CM247LC following selective laser melting

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Selective laser melting has the potential for producing components with complex geometries that cannot be fabricated using conventional processing routes. However, the severe thermal cycles experienced in the material generate microstructures that do not provide optimal properties and carefully designed post deposition treatments are generally required. This is particularly true to high performance structural components in gas turbines that are made from nickel-based superalloys. In this work, the microstructure of the nickel-based superalloy, CM247LC, has been studied in the as deposited condition and following a series of heat treatments in the vicinity of the gamma prime solvus. Depending upon the heat treatment conditions, recovery and recrystallization of the as deposited microstructure are observed. The effects of these processes on the texture, elastic anisotropy and distribution of secondary phases are discussed.

704 Evaluation of solidification crack susceptibility in laser beam welds of reduced activation ferritic/martensitic steel F82H

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Nuclear fusion is expected as a next-generation electric power source. The reduced activation ferritic/martensitic steel F82H is developed by the Japan Atomic Energy Agency and supposed to be a structural material of the blanket module, which is the device set on the inner wall of nuclear fusion reactors to guard the wall from neutron damage, to convert the neutron energy

into electricity and to produce tritium. However, weldability of the steel using laser beam welding (LBW) has not been examined adequately. A problem of particular concern for the LBW is solidification cracking. Especially, in this study, center-line cracking, which is one of typical solidification cracking in LBW, has been investigated. To evaluate the crack susceptibility and critical strain, the side-bead test was adopted as a testing method. The cracks propagated at the center of weld beads in test coupons due to in-plane deformation and stopped by increasing of rigidity at the deformed side in the coupons. As compared with the experimental results and the calculation by a thermo-elastic-plastic analysis, cracks were found to be stopped at a certain value of curvature measured at the crack tip. Therefore, due to evaluate the critical curvature value, in the case of LBW for the steel, it was assumed that the critical strain to prevent the solidification cracking at center of bead can be determined.

705 Evolution of deformation microstructures in cold-rolled ferritic steel

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Deformation microstructures developed in cold-rolled ultra low carbon (ULC) steel as well as those in low carbon (LC) steel have been investigated by using TEM and SEM-EBSD techniques. Particular attention has been paid to the effect of solute carbon on the development of those microstructures. Dislocation structures characteristic to the preferred orientations such as gamma-fiber (ND//<111>) and alfa-fiber (RD//<011>) have been revealed by the same area observation employing both TEM and SEM-EBSD techniques.

TEM images of dislocation cell boundaries observed in ULC are relatively clearer than those in LC structures. Images of dislocation line segments were separately distinguished in cell structures in ULC, while in LC they were indistinguishable because of high density of dislocations. This indicates that dislocation density increases with increasing the amount of solute carbon, which was confirmed also by XRD measurement. In grains of ND//<111>, fine microbands and/or shear bands (SBs) were developed while in RD//<011> grains such remarkable inhomogeneous microstructures were not observed, which suggests that work-hardening in ND//<111> grains is more prominent than that in the other preferred orientations. In {111}<211> grains of LC steel, the same kinds of SBs as observed in Fe-Si steels were formed as the most characteristic microstructure, where elongated fine-grained structures with the orientation scattering of 35° between the {111}<211> and {110}<001> Goss orientation were found.

706 Mechanical properties of duplex stainless steel with martensitic phase and austenitic phase

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Mechanical properties of duplex stainless steels with martensitic phase and austenitic phase were investigated. Nominal chemical compositions of the stainless steel are 13%Cr - 7%Ni - 3%Si - Low C - Low N. Duplex structure was obtained by quench and reversion heat treatment at 873-913K (between austenitic phase transformation start and finish temperature). Martensitic

phase was obtained by quenching, then 50% volume fraction of austenitic phase was transformed from martensitic phase by continues reversion heat treatment at 908K, 7.2ks. The austenitic phase was stabilized by diffusion of Ni element from martensitic phase during reversion heat treatment. The effect of grain size of austenitic phase for mechanical properties was investigated. Grain size was controlled by cold rolling reduction rate before reversion heat treatment. 0.2% proof stress of duplex stainless steel with 1micrometer austenitic grain size was 980MPa, tensile strength was 1010MPa, and total elongation was 22.5%. On the other hand, 0.2% proof stress of duplex stainless steel with 10micrometer austenitic grain size was 765MPa, tensile strength was 980MPa, and total elongation was 21.9%. Thus, duplex stainless steel with martensitic phase and fine grain austenitic phase shows high-strength and good-ductility.

707 Crystallographic investigation of the initial solidification grain structure in Al-Si alloy

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As-solidified structure of an ingot is composed of the chill, columnar and equiaxed zones. The whole solidified structure is strongly affected by the chill crystals. Some initial solidification grains have been observed on the ingot surface and thought to be traces of the nucleation point. The aim of this study is, therefore, to develop the experiment technique to make one 'grain' and to crystallographically investigate the initial solidification grain using EBSD analysis.

In order to start solidification at a very specified position, a small metallic protrusion was installed on an insulating plate. Al-6 wt%Si alloy was melted at 800 °C and was poured on the metallic protrusion. In this study, the amount of protrusion was varied to investigate the growth mechanism of the initial solidification grain. The longitudinal cross section of the specimen was observed by an optical microscope, a scanning electron microscope. The starting position of solidification was the area that was on the metallic protrusion. In this initial solidification grain, it was difficult to observe the dendritic structure. The shape of this grain was about hemispherical. The grain area seemed to increase with increasing the amount of protrusion. The results of EBSD analysis showed that almost all initial solidification grains were composed by several crystals. The reason of this is that the nucleation frequency may increase with the amount of protrusion. The dendrite grew radially from the initial solidification grain continuously. The crystallographic structure was also continuous on the boundary of the initial solidification grain.

708 Effect of deformation structure on strength of a low-alloyed Cu-Cr-Zr alloy

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The microstructure evolution and strength properties of a Cu-0.096%Cr - 0.057%Zr alloy subjected to equal channel angular pressing (ECAP) at a temperature of 673 K via route B_C to total strains from 1 to 12 were examined. After first ECAP passes the formation of planar low-angle boundaries with moderate misorientations occurs within initial grains. Upon further processing the misorientations of these boundaries progressively increase and the formation of

new ultrafine grains occurs. Continuous dynamic recrystallization (cDRX) takes place. Partially recrystallized ultrafine structure evolves after strain above 4. After strains of 12 the grain/subgrain size attains 0.29 microns. Large plastic straining provides significant strengthening: the ultimate tensile strength increases from 190 MPa in the initial state to 465 MPa after 12 ECAP passes. A modified Hall-Petch analysis is applied to investigate the contribution of grain refinement and dislocation density to the overall strengthening.

709 Transparent fluoride ceramics for laser applications

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Since the 1995s, transparent ceramics have demonstrated their potentiality to replace single crystals as amplifying media for light (laser). First significant results were brought within oxide compounds when fluoride materials were attracting lower attention due to inherent complexity of fabrication and weakest performances. Simultaneously, a fluoride single crystal, Yb:CaF₂, has reached a leading place in the 2000s by holding many laser records: short pulse duration, high energy delivery, wide wavelength tunability... Calcium fluoride also gathers interesting physical properties (high thermal conductivity and laser damage threshold) and attractive optical properties (broad absorption and emission bands). Finally, it has demonstrated a huge potential as a near-infrared high average power amplifier allowing efficient laser-diode pumping around 980 nm. However, these interesting results were obtained with Yb:CaF₂ single crystals when the challenge would lie in the transposition of these results to a ceramic form which could provide technical advantages: easier, cheaper and quicker production, fewer shape restrictions, no macroscopic segregation of the dopant ions, and an enhanced fracture resistance compared to the single crystal form. We will describe the preparation process of Yb:CaF₂ ceramics synthesized from raw nanopowders obtained by soft chemistry route. The green body is prepared through a pressureless method and its densification includes a single step sintering requiring moderated temperature. The process is energy extensive. Finally, some laser properties of the ceramics will be presented after a precise characterization of the ceramics (grain structure, grain boundaries, segregation and defects).

710 Life+12 ENV/IT000439 Greenwoolf: Green Hydrolysis conversion of wool wastes into organic nitrogen fertilisers

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EU-27 has the second world sheep population, numbered about 100 million heads in December 2011, the majority of which were based in the UK (25%), Spain (20%), Romania (10%), Greece (10%), Italy (9%), France (9 %) and Ireland (4%) (Source EU-Eurostat 2012). The EU flock is made of crossbred sheep not graded for fine wool production; the primary role is meat, whilst the milk market is relatively small, being confined to Mediterranean regions. The annual wool clip amounts to more than 200 000 t (18-20 are produced in Italy) and its

management is a problem for the EU livestock sector. Indeed, wool from sheep farming and butchery industry is very coarse making it practically unserviceable for the textile industry. Unserviceable wool is mostly disposed in landfills or illegally thrown over, with serious ambient threats since it can affect the pastures and bring illness, where it does not readily degrade. Thus, shearing, storage, transportation and disposal of waste wool in accordance with current EU Regulation, heavily weigh on the profit of sheep farming.

The Life+ 12 ENV/IT000439 GreenWoolF, aims at demonstrating that waste wools can be recycled into organic nitrogen fertiliser for grassland management and other cultivation. Controlled hydrolysis with direct steam converts wool keratin (the wool protein) into simpler compounds, tailoring the release speed of nutrients to plants. Wool contains elements such as carbon, nitrogen and other nutrients, which play an essential role in plant nutrition. Wool, when added to the soil, increases the yield of grass grown, absorbs and retains moisture very effectively, reduces run off of contaminants such as pesticides, and can aid in water conservation.

711 Additive manufacturing of energetic materials: Enabling a new design parameter for controlled performance

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Additive Manufacturing (AM) provides control over micron-to-mm internal structures of components at a level impossible to achieve through traditional machining techniques, opening new avenues of control over material characteristics and performance. This has implications in the strength and flexural properties of AM generated metal and plastic parts, enabling a higher strength to weight ratio, tunable rigidity and vibrational transmission properties by design. This modification of internal structure gives rise to unprecedented possibilities in the control by design of the safety and performance of energetic materials. Application of AM to reactive materials allows, for the first time, exquisite control of microstructure and properties so that the interplay between structure and reactive chemistry may be elucidated and exploited.

Experimental investigations on the effect of this structural control on the ignition and burn characteristics of energetics have been performed in close coordination with theoretical modeling of their structural properties and performance. Using Direct Ink Write (DIW), Fused Deposition Modeling (FDM), and variants of these techniques, we are conducting research to develop a theoretical understanding necessary to design and fabricate structural features into energetic hierarchies that have simply not been previously possible. We will present the results of these experiments on the possibilities of using AM production of energetics to manipulate, by design, the ignition and burn performance and the propagation of shock fronts within the part. LA-UR-15-29230

712 Rapid solidification effects in powder metallurgy

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Over 90% of all manufactured goods contain some metallic parts. For the vast majority of these, solidification is the primary processing step, with the microstructure and length scales established during solidification often surviving subsequent processing operations to influence the properties of the manufactured article. Over recent years the manipulation of solidification microstructures via rapid solidification processing has become important; particularly via powder metal techniques such as gas atomization in which cooling rates $> 100,000$ K/s can be achieved. In this presentation, I will look at some of the properties enhancements that can be achieved in powder metals compared to the equivalent bulk solidified material. Specific examples will include cast iron powders in which even relatively modest cooling rates of 200 K/s can lead to the elimination of flake graphite with a resulting ferrite matrix twice as hard as the equivalent bulk material. With further increases in cooling rate (decrease in particle size) the proportion of retained austenite increases with a Martensitic transformation occurring for cooling rates $> 20,000$ K/s, wherein a further factor of two increase in hardness is observed. Other examples will include Ni-based intermetallics where high cooling rates may be utilised to improve room temperature ductility and Raney type sponge-Ni catalysts in which rapid solidification can increase the catalytic activity six-fold. We will also look at the homogeneity of gas atomized powders showing, somewhat surprisingly, that there can be large ($> 10\%$) variations in phase compositions both between particles from the same powder batch and within individual particles.

713 Microstructure and oxidation resistance of bond coats on Ni-based single crystal superalloys

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In order to enhance the durability and total life of jet engines, further development of thermal barrier coatings (TBCs) on turbine blades is required. TBCs consist of two layers, a ceramic top coat for thermal insulation and a metallic bond coat for protect Ni-based superalloy substrates against accelerated oxidation, by forming a protective Al_2O_3 layer on its surface. In addition, bond coats literally “bond” the metallic layer and the ceramic layer by the presence of Al_2O_3 thus the lifetime of turbine blades is largely affected by the bondcoat.

When the coated blades are exposed to high temperature, microstructural changes in the coated layer and the substrate are inevitable due to the interdiffusion of metallic alloying elements, one of the examples is the formation of a secondary reaction zone (SRZ), which are often observed for aluminized coatings. The SRZ destroys the gamma and gamma' coherent two-phase microstructure, it accordingly leads to a deterioration of mechanical properties of the substrate. On the other hand, in the case of gamma + gamma' two phase coatings, oxidation resistance and microstructural changes are affected by alloying elements in coatings and substrates and Kirkendall-type bond coats are sometimes observed. This presentation reviews the microstructural changes of bond coats, focusing on aluminized coatings and Pt-based diffusion coatings. The effect of pre-surface treatment, coating parameters, alloying elements in the coatings and substrates, and thermal exposure history will be discussed.

714 Friction and wear properties of AlB₁₂- and SiB₆-based ceramics

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Lubrication with water has been receiving attention in industries related to water pumps, food machinery, medical devices because water is an ecological material. Currently, Si₃N₄-, Al₂O₃-, SiC-based ceramics are generally used as the sliding materials in such water-lubricated systems. However, these ceramics exhibit friction coefficients higher than 0.5 just after sliding was started. It was found in our previous study that AlB₁₂ and SiB₆ ceramics exhibited friction coefficients as low as 0.1 and low specific wear rates when they were slid against Si₃N₄ in water. However, the friction and wear properties of the AlB₁₂ and SiB₆ ceramics have not been investigated at room temperature and high temperatures in air. Moreover, the AlB₁₂ and SiB₆ ceramics exhibited low fracture toughness. It is thought that adding a small amount of metallic binder phase to the boride specimens may increase their fracture toughness. In this study, the friction and wear properties of the AlB₁₂ and SiB₆ ceramics were investigated at room temperature and high temperatures in air. Also, the AlB₁₂-NiAl type cermet specimens were prepared by spark plasma sintering. It was found that the friction coefficients of the AlB₁₂ ceramic specimens at room temperature in air were as low as those at room temperature in water. It was also found that AlB₁₂-20vol% NiAl cermet specimens could be obtained by spark plasma sintering. We will explain the rest of the results in this presentation.

715 Development of dislocation densities under uniaxial loading in Ni 201 and SS 316

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Strain-hardening of a polycrystalline aggregate is governed by the ability of the material to store dislocations that obstruct the motion of other moving dislocations. Plastic deformation is thus accompanied by the storage of ever-increasing amounts of dislocations when strain-hardening is present: (i) statistically-stored dislocations (SSDs) - are stored by trapping one another in a random way, while (ii) geometrically-necessary dislocations (GNDs) - are stored by the microstructure to maintain continuum deformation. Recent developments of the Electron Back-Scatter Diffraction (EBSD) and high-resolution diffraction line profile analysis (LPA) techniques provide an opportunity for studying the way in which a material stores different types of dislocations. In general, the diffraction LPA techniques are sensitive to the strain field caused by the presence of all types of dislocations, while the EBSD technique is sensitive to a variation in the lattice curvature, which is attributed to the presence of GNDs. The rest of the dislocations have no geometrical consequence at the length scale studied, and are thus undetected by the EBSD lattice curvature analysis, and can be considered as SSDs. In the present study the high-resolution synchrotron diffraction and EBSD techniques have been used to study the development of the total (SSD + GND) dislocation density and GND density during the uniaxial loading of Ni201 and austenitic 316 steel. It is shown that the strain-hardening of both materials is governed by the collaborative effect of both SSDs and GNDs.

716 Phase stability and mechanical properties of Ti-Cr-Sn-Zr alloys containing a large amount of Zr

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Meta-stable beta Ti alloys with low Young's modulus are attractive for biomedical application, because large difference of the Young's modulus between the human bone and the metallic implant materials may cause the damage on human bone due to stress shielding. The Young's modulus of Ti-Cr-Sn-Zr alloy varies with the composition of Cr, Sn and Zr, which elements act as beta stabilizer. Some Ti-Cr-Sn-Zr alloy shows very low Young's modulus under 50GPa. The amount of Zr in the alloys with very low Young's modulus increases with the decrease of Cr. We investigated the Young's modulus and deformation behavior of Ti-xCr-Sn-Zr ($x=0\sim 2\text{mass\%}$) alloys containing large amount of Zr.

The quenched microstructure of Ti-Cr-Sn-Zr alloys changes from martensitic structure to beta single-phase structure if the beta stabilized elements increase. The Young's modulus decreases with the increase of Zr if the amount of Cr and Sn are fixed. And then through minimum, the Young's modulus increases with the increase of Zr. The Ti-Cr-Sn-Zr alloys with composition close to the transitional composition of microstructure show minimum Young's modulus. The clear microstructural transition disappears if the amount of Cr becomes too small, and then the minimum Young's modulus increases. We indicate the effect of the varying alloy composition on the microstructure, the Young's modulus, the deformation mechanism and the deformation behavior in the Ti-Cr-Sn-Zr alloys containing large amount of Zr.

717 Harnessing the multifunctionality in nature: A bioactive agent release system with self-antimicrobial and immunomodulatory properties

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All implantable biomedical systems face several risks once in contact with the host tissue: i) excessive immune response to the implant; ii) development of bacterial biofilms and iii) yeast and fungi infections. A multifunctional surface coating which can address all these issues concomitantly would significantly improve clinical outcomes. We develop here for the first time a coating that address these three issues simultaneously. We hypothesized that polyarginine (PAR), a synthetic highly cationic polypeptide, can act on macrophages to control innate immune response because arginine is an important component of macrophage metabolism. Moreover, PAR is susceptible to act as an antimicrobial agent due to its positive charges. We developed a new polyelectrolyte multilayer films based on PAR and hyaluronic acid (HA). The PAR/HA films have a strong inhibitory effect on the production of inflammatory cytokines released by human primary macrophages subpopulations. This could reduce potential chronic inflammatory reaction following implantation. Next, we show that PAR/HA films were very effective against *S. aureus* for 24h. The PAR/HA films can be easily further functionalized by embedding antimicrobial peptides, like catestatin (CAT), a natural host defense peptide. This PAR/HA+CAT film proved to be effective as an antimicrobial coating against yeast and fungi. The cytocompatibility of the PAR/HA films was assessed with

human umbilical vein endothelial cells (HUVECs). This all-in-one system that limit strong inflammation and prevent pathogen's infections constitutes an original strategy to coat implants in an active way. Adv Healthc Mater. 2015, 4, 2026 J. Leuk. Biol. 2015, 1b.5VMR0415-166R.

718 Microstructure study of nickel-based superalloys after deep cold rolling

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Mechanical surface treatments are performed on aerospace components in order to improve their fatigue life through inducing compressive residual stresses, cold work and smoother finish. The microstructure of the component surface and subsurface after treatment influences the crack nucleation and crack propagation significantly. This paper studies the effect of Deep Cold Rolling (DCR), a subsurface process which uses hydrostatically controlled balls, on the resulting microstructure of IN100, a nickel-based superalloy used in high temperature aerospace applications. In this study, DCR of IN100 is performed by varying two significant process parameters, ball size and pressure. Residual stress profiles of the rolled samples are measured using X-Ray Diffraction (XRD). Vickers hardness is measured to characterize the work hardening of the samples. The microstructure of IN100 subsurface before and after DCR in both the rolling and transverse directions is analyzed further. The results show that apart from inducing deep compressive stresses, DCR causes a significant variation in the microstructure at the surface and the subsurface.

719 An amorphous phase formation at palladium / silicon oxide (Pd/SiO_x) interface by electronic excitation

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A Pd-Si amorphous phase was formed at a palladium/silicon oxide (Pd/SiO_x) interface at room temperature by electron irradiation at acceleration voltages ranging between 25 kV and 200 kV. The formation of a Pd-Si amorphous phase occurred under electron irradiation with an acceleration voltage under the threshold acceleration voltage of the electron knock-on effect of Si, O and Pd, indicating that the electron knock-on effect is not necessary for the formation of a Pd-Si amorphous phase. The total dose required for a Pd-Si amorphous phase formation decreased with decreasing acceleration voltage. Electron irradiation induced metallic amorphous formation caused by the electronic excitation at metal/silicon oxide interface was found at Pd/SiO_x interface.

720 High fatigue strength of Ti-12Cr rod as spinal fixation devices

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High fatigue strength is required for spinal fixation devices. However, this property was difficult to be obtained when beta-type titanium alloys with low Young's modulus was employed previously. Therefore, cavitation peening was applied to improve fatigue strength of spinal implant rods made of beta-type titanium alloy with changeable Young's modulus, Ti-12Cr. In this alloy, Young's modulus increases only at deformed part because deformation-induced omega phase transformation occurs, while it remains low at non-deformed part. Deformation-induced omega phase is formed near the surface of Ti-12Cr rod by cavitation peening. Such the deformation-induced omega phase in addition to work hardening and residual compressive stress introduced by cavitation peening enhances the fatigue strength of Ti-12Cr rod as the spinal fixation devices.

721 Misorientation measurement of individual grains in fatigue of polycrystalline alloys by diffraction contrast tomography using ultrabright synchrotron radiation

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A three dimensional grain mapping technique for polycrystalline materials, called X-ray diffraction contrast tomography (DCT), was developed at SPring-8, which is the brightest synchrotron radiation facility in Japan. The developed technique was applied to a commercially pure iron and austenitic stainless steel. The shape and location of grains could be determined by DCT using the apparatus in a bending beam line of SPring-8. To evaluate the dislocation structure in fatigue, the total misorientation of individual grains was measured by DCT. The average value of the total misorientation over one sample was increased with the number of cycles. In a grain, the change of the total misorientation was largest for primary slip plane. For austenitic stainless steel (fcc), the change of the total misorientation in fatigue was larger for planes with larger Schmid factor, while it was not depended on the Schmid factor for commercially pure iron (bcc). This different behavior must come from planer slip in fcc structure and wavy slip in bcc structure.

722 Unusual wetting on surface fine crevice structure by laser irradiation

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Prof. T. Tanaka found "unusual wetting", in which a liquid metal spreads by capillary action on a porous metal surface layer formed under atmospheric oxidation-reduction and proposed a

metal-metal joining using “unusual wetting”. However, it is difficult to subject only a specific area to the atmospheric treatment, and a region-selective “unusual wetting” is not performed. As a new challenge of “unusual wetting”, we pay attention to a laser treatment to create an adequate structure for “unusual wetting” on metal surface. A laser enables us to provide a region-selective treatment that cannot be achieved by the atmospheric treatment. On Cu surface, a laser irradiation forms the structure that looks like splashing water the outermost surface and the crevices at intervals of several tens of microns below the outermost surface. We termed the surface structure formed by laser treatment a “surface fine crevice structure” based on its characteristic morphology. The wetting experiments of laser-treated Cu plate by liquid metals were performed to confirm region-selective “unusual wetting” on the “surface fine crevice structure”. We also challenged to join Cu parts with “surface fine crevice structure” by “unusual wetting”.

723 Electric conductivity along lattice defects in lithium niobate

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Lattice defects in ferroelectric crystals have received attention in recent years because they often exhibit distinct electrical conduction although ferroelectrics are insulating. It is suggested that structural charge at lattice defects attracts carriers with the opposite charge such as electrons or holes and accordingly electrical conduction will be brought about by the carriers. However, the number of experimental researches on the electrical conduction have been limited due to the difficulty of systematic experiments. Here we report electric conductivity at boundary dislocations and charged domain-walls in ferroelectric crystals fabricated using the bicrystal technique and discuss about how the conductivity occurs.

724 Picosecond time-resolved X-ray diffraction studies on phase-transition dynamics under non-equilibrium high pressures

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We have studied the phase-transition dynamics of bismuth poly-crystals under non-equilibrium high pressures induced by laser shock compression using picosecond time-resolved X-ray diffraction. The experimental system was set at the NW14A beamline of the Photon Factory Advanced Ring. Picosecond time-resolved X-ray diffraction was achieved using laser pump and X-ray probe technique with 100-ps X ray pulses. By using a plasma confined target (25-micron plastic layer, 3-micron Al ablator layer, and 20-micron sample) and 10-ns laser pulse (wavelength of 1064 nm) at around 1 J/pulse, we generated approximately 11 GPa. The results show that a sudden transition from Bi-I to Bi-V phase occurs within approximately 5 ns, and sequential V–III–II–I phase transitions occur within 30 ns during the pressure release process. We have also examined the thin (5 micron) samples, and observed the almost all volume of the sample transforms to the Bi-V phase within 5 ns. We also present the structural

deformation of aluminum, which is used as the ablator layer. In addition to the X-ray diffraction results, we also present our new development of the system with high-power glass laser (10 ns and 16 J/pulse), which enable to study the structural dynamics of materials at much high-pressures up to 30 GPa.

725 Fabrication and anisotropic properties of oriented $\text{Li}_{1+x-y}\text{Nb}_{1-x-3y}\text{Ti}_{x+4y}\text{O}_3$ solid solutions by slip casting in a high magnetic field

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$\text{Li}_{1+x-y}\text{Nb}_{1-x-3y}\text{Ti}_{x+4y}\text{O}_3$ ($0.06 < x < 0.33$, $0 < y < 0.09$)(LNT) forms with a superstructure, and which is the so-called M-phase. The M-phase field does not present itself as a solid solution but rather a homologous series of commensurate intergrowth structures with LiNbO_3 -type slabs separated by single $[\text{Ti}_2\text{O}_3]^{2+}$ layers. The period created by the insertion of an intergrowth layer becomes shorter with increasing Ti content. As an application of the unique qualities of an electro-ceramic with an anisotropy structure, we prepared an oriented LNT ceramic by slip casting in a high magnetic field (12T). The oriented bulk ceramics were synthesized with various Ti content. The magnetic direction was parallel to the slip cast direction. The compact was densified by cold isostatic pressing at 392 MPa and then sintered at 1373 K for 15 h. Characterizations of the oriented specimens were performed using XRD, TEM and SEM. Anisotropic electrical properties were observed in the oriented LNT specimens. The electric resistivity on the parallel to the c-axis is higher than that on the perpendicular direction. The values were decreased with increasing Ti content in the LNT specimens. As a result, the intergrowth layers acted as an electric conduction path. The relative permittivity was decreased but the Q value increased with increasing Ti content. The anisotropic structure had a great influence on the Q value. Acknowledgement: This work (H. N.) was partially supported by a Grant-in-Aid for Scientific Research (c) No. 25420709 by the Japan Society for the Promotion of Science.

726 Development of a new powder/solid composite for bio-mimic anisotropic implant materials by electron-beam additive manufacturing

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Electron beam melting (EBM) is suitable for fabricating hierarchically designed products at various scale levels. Such materials should possess function to suppress stress shielding in order to avoid the bone absorption and degradation of anisotropic bone quality which is closely related to mechanical integrity of bone. In this study, new biomaterials composed of powder and solid parts for exhibiting bone-mimic anisotropic mechanical properties are proposed. The raw material used was gas-atomized Ti-6Al-4V ELI powder comprising spherical particles with a diameter of approximately 80 μm . Cube-shaped products composed of 27 ($3 \times 3 \times 3$) unit cubic compartments that were occupied by solid or powder part were designed using 3D-CAD.

The products were fabricated by EBM (Arcam AB, Sweden) according to the specifications shown in the CAD drawing. New powder/solid composite products were fabricated by the EBM method. The residual un-melted powder in the products does not need to be removed to make the products more mechanically integrated. Moreover, the layout of the powder and solid parts in the products were arranged to achieve bone-mimic mechanical anisotropy. The products demonstrate isotropic or anisotropic Young's modulus, yield stress, and toughness, all of which are changeable by conditions of EBM and following heat treatments for developing necks among un-melted powder particles. In conclusion, novel powder/solid products comprising solid cubic parts and functionalized powder particles between them were successfully developed, which might be useful in biomedical and industrial applications.

727 High temperature mechanical properties of harmonic structure designed SUS304L austenitic stainless steel

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Through many years, conventional material developments have emphasized on microstructural refinement and homogeneity. However, "Nano- and Homogeneous" microstructures do not, usually, satisfy the need to be both strong and ductile, due to the plastic instability in the early stage of the deformation. As opposed to such a "nano- and homo-" microstructure design, we have proposed "Harmonic Structure" design. The harmonic structure has a heterogeneous microstructure consisting of bimodal grain size together with a controlled and specific topological distribution of fine and coarse grains. In other words, the harmonic structure is heterogeneous on micro- but homogeneous on macro-scales. In the present work, the harmonic structure design has been applied to an SUS304L austenitic stainless steel via a ball milling process and a large size (50 mm in diameter) SPS sintering process. At a macro-scale, the harmonic structure SUS304L compacts exhibited significantly better combination of strength and ductility, under quasi-static tensile loadings, as compared to their homogeneous microstructure counterparts. High temperature tensile tests revealed that they also indicated high strength at elevated temperatures.

728 Spectroscopic studies on graphite and graphene under high pressure

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Graphite is the most prevalent carbon allotropes. It consists of Bernal stacking of graphene sheets. When we compress graphite, an interlayer distance is easy to be shortened but a honeycomb lattice made of strong covalent bond is hard to shrink. The magnitude of linear modulus along the a-axis is 40 times as large as that along the c-axis. However, the lattice transformation is not simple when graphite is pressurized with hydrogen. The honeycomb lattice isotropically lengthens while the interlayer distance is shortened under pressure lower than 1 GPa. It is supposed that hydrogen-graphite intercalation-compound (GIC) is generated by pressurization. In this case hydrogen enclosed in the interlayer space may bring a direct application of stress to graphene, resulting in the stretching of honeycomb lattice. On the other

hands the static structure obtained using a density-functional calculation showed the formation of C-H bond under high pressure. This suggests that a phonon system is made in a two-dimensional nano-space under high pressure. In this talk we present recent results of Raman study on hydrogen-GIC under high pressure and discuss an effect of hydrogen phonon to graphite. We also introduce our recent study on few layer graphene under high pressure.

729 Crystallographic features of the approximant H phase in the Mn-Si-V alloy system

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The dodecagonal quasicrystal is one of two-dimensional quasicrystals with the translational symmetry along one crystallographic direction. Its atomic arrangement is suggested to involve dodecagonal atomic columns, which are characterized by an array of coordination polyhedra with coordination number 14 along the crystallographic direction. Because it is hard to obtain stable dodecagonal quasicrystal, however, both the presence of dodecagonal column and the detailed features of its arrangement have not been understood sufficiently. To clarify these points, in this study, we focused on the hexagonal H phase as an approximant of the dodecagonal quasicrystal in Mn-Si-V alloy system, and have investigated its crystallographic features, mainly by transmission electron microscopy.

The experimentally-obtained data showed that two kinds of areas are present in prepared samples with compositions of about 20.%Si-10at.%V. Bright-field images of one area gave rise to uniform contrast, while contrasts due to structural defects were often observed in the other. An array of bright dots was also detected in their high resolution electron micrographs with the electron beam incidence parallel to the c axis. Because each dot corresponds to a dodecagonal column, as a result, uniform-contrast areas had the H structure, which is characterized by a periodic array of dodecagonal units consisting of 19 dodecagonal columns. On the other hand, both an array of penetrated dodecagonal units and a random array of dodecagonal columns were found to be involved in defected areas. Based on these, a dodecagonal atomic column can be identified as a structural unit of the H structure.

730 Magneto-impedance effect in soft-magnetic metallic glass nanowire and microwire

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We report the magneto-impedance measurements in soft-magnetic $\text{Co}_{36}\text{Fe}_{36}\text{B}_{19}\text{Si}_5\text{Nb}_4$ metallic glass nano/micro wires produced by gas atomization. The wire impedance varies with external magnetic fields and the impedance peak position depends on the frequency, indicating ferromagnetic resonance. The postulation of anisotropy directions in ferromagnetic resonance allows to the determination of magnetic anisotropies in the wire. By decreasing the wire diameter, the reduction of magnetic anisotropy is observed.

731 Effect of shearing distance on transfer characteristic of Al thin plate formed by compression shearing method at room temperature

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Although processing methods for milli and nano-sized parts have improved, few methods are available for micro-sized parts. For example, while the discharge machining processing method can be used to produce micro-sized parts, the process is complicated to apply to mass production and the creation of complex shapes is difficult. Consequently, as a new processing method for micro-sized parts, attention is being paid to the transcription of mold surface shapes using the compression shearing method at room temperature (COSME-RT). In this study, we formed aluminum (Al) thin plates, applied Vickers mark indentations to mold surfaces, and then transferred those marks using COSME-RT. Additionally, the Al plates formed by different shearing distance were tested to determine the effects of shearing distance on the transcriptional characteristics. The results show that COSME-RT can be used to transfer micro-shapes to Al thin plates by changing the shearing distance.

732 Microstructure formation of high pressure torsion processed (alpha gamma) two phase stainless steel

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(alpha + gamma) two phase stainless steel (Fe-21%Cr-5%Ni-1.5%Mo) powder was processed by high pressure torsion (HPT) and consolidated at room temperature. The received powder had fully alpha single phase due to the rapid cooling by gas atomizing process. Specimens after HPT process were heat treated at 1173K-3.6ks. X-Ray Diffraction (XRD) analysis revealed that decomposition of alpha phase to gamma phase took place during the heat treatment. Detailed microstructure observation showed that an equiaxed (alpha + gamma) micro-duplex structure was developed and its average grain size was approximately 3.2 micro meters. The same heat treatment given to the material without HPT resulted in a coarse two phase microstructure, therefore, it is considered that an ultra fine grained microstructure was caused by increasing of nucleation sites for gamma phase due to severe plastic deformation (SPD) of HPT process. Electron backscatter diffraction patterns (EBSD) analysis indicated that alpha phase has a {110}/<001> strong texture, that is, the alpha phase seems to have single orientated coarse grain structure. The gamma precipitates indicated a {111}/<001> strong texture, and the crystallographic orientation relationship of Kurdjumov-Sachs was observed. Noteworthy is that only a single variant was observed. In other words, there exists a severe variant selection rule for choosing a single variant from 24 variants. Such a variant selection rule was probably attributed to the limitation of nucleation sites for gamma precipitates in alpha phase.

733 Laser processing of biomedical materials

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Laser-based processing of biomedical materials has undergone significant development in recent years. The ability to modify a variety of materials with short production times and at small length scales provides laser technologies with unique capabilities for fabrication of medical devices. In many laser-based processes, microscale and sub-microscale structuring of materials is governed by computer-generated models. In this review, use of various laser-based processes for fabrication of microstructured and nanostructured medical devices is considered. Several techniques are discussed, including laser machining, matrix-assisted pulsed-laser evaporation direct write, microstereolithography, two photon polymerization, and pulsed laser deposition. Use of in vitro and in vivo biological techniques for assessing laser-modified materials will be considered. Laser-based processes may be used for processing a wide variety of advanced medical devices, including patient-specific prostheses, biosensors, drug delivery devices, and tissue engineering scaffolds.

734 Influence of nano reinforcement volumepercentage on fabrication of surface nanocomposite by FSP

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This work deals with the effect of volume percentage of nano reinforcement to fabricate nano surface composite by Friction Stir Processing (FSP) and also studied the role of tool rotational speed and traverse speed to get the defect free condition to fabricate successful surface composite. The material flow pattern, dispersion of the reinforcement particles in the stir zone was examined. From the phase/volume fraction analysis, it was observed that the nano Al_2O_3 particles were well dispersed in the stir zone. The results indicate that the better microstructural, mechanical properties were obtained at 1150rpm /15mm/min condition. A significant improvement in microhardness was exhibited by surface nano composite as compared to the as - received aluminum.

735 Precipitates and mechanical properties of metallic biomaterials

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Metallic biomaterials such as NiTi and Co-Cr alloys have been used for self-expandable and balloon-expandable stents because of their excellent strength, corrosion resistance and biocompatibility. In stent applications, the size and phase of the precipitates, especially non-metallic inclusions, of the alloys need to be controlled because stents have a fine mesh structure. Non-metallic inclusions deteriorate the mechanical properties of the fine-structured stents. In this study, the precipitation behavior of the NiTi and Co-Cr alloys was clarified, and the change

in mechanical properties with precipitation was investigated. The precipitates in the NiTi (ASTM F2063) and Co-20Cr-15W-10Ni (mass%, ASTM F90) alloys were separated from the metallic matrix using electrolytic extraction for XRD analysis. In NiTi alloys, $Ti_4Ni_2O_x$ oxide and $Ti(C,N,O)_x$ carbide were detected as precipitates, depending on the oxygen and carbon content. It was suggested that the presence of $Ti_4Ni_2O_x$ oxide lowered the fatigue strength of the NiTi alloy wires. In the Co-20Cr-15W-10Ni alloy, eta-phase ($M_6X-M_{12}X$ type) and $M_{23}X_6$ type (M: metallic elements, X: C and N) precipitates were formed during heat treatment at temperatures ranging from 973 to 1473 K. The ductility of the alloy was found to deteriorate owing to the presence of eta-phase precipitates.

736 Characterization of product phases formed from austenite during isothermal treatments around the M_s temperature in a low-C high-Si steel

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Isothermal treatments around the martensite start temperature (M_s) are of crucial importance for the design of Advanced Multiphase High Strength Steels. These steels are mainly formed by a matrix of bainite and martensite, which allow these steels to have a very good balance between tensile strength and ductility. Previous investigations have shown that bainitic ferrite can form from austenite in isothermal treatments below M_s , where its formation kinetics is accelerated by the presence of the athermal martensite. However, the distinction of the nature of the product phases by microscopy is difficult due to similarities in morphology between bainitic ferrite and tempered martensite. The aim of this study is to identify morphological differences between tempered martensite and bainitic ferrite. For this purpose, dilatometry experiments were performed at temperatures above and below the M_s in a low-carbon high-silicon steel, where tempered martensite and bainitic ferrite were obtained as product phases. A combination of characterization techniques, such as SEM and EBSD, was used to identify structural and morphological differences between both product phases. The transformation characteristics as observed in dilatometry were included in the analysis.

737 On the role of alloying elements in gamma/gamma prime cobalt-base superalloys

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Since the discovery of a ternary $Co_3(Al,W)$ phase in the Co-Al-W system in 2006, precipitation strengthened Co-base superalloys increasingly win worldwide scientific interest. They possess a very similar microstructure compared to Ni-base superalloys consisting of a high volume fraction of cuboidal intermetallic gamma prime precipitates, which are coherently embedded within a gamma fcc solid solution. Despite these similarities, there are also many differences. In this study several quaternary and quinary systems have been examined by a wide variety of methods including scanning electron and atom probe microscopy, neutron diffraction, differential scanning calorimetry, thermodynamic calculations and creep tests. The aim is to

study the influence of alloying elements on various properties of Co-Co₃(Al,W)-based superalloys such as phase transformation temperatures and stability, gamma/gamma prime partitioning behavior and lattice misfit, and mechanical properties. Special emphasis is placed on the role of the base element in superalloys. To systematically address the change of alloy properties, when the base element changes from Co to Ni, two sets of model superalloys Ni-XCo-9Al-8W-(8Cr) were designed, where Ni was successively substituted by Co. Atom probe microscopy investigations, for example, revealed that Al is strongly enriched in gamma prime in the Ni-rich alloys and is distributed almost equally between gamma and gamma prime in the pure Co-base, while W is enriched in gamma for the Ni-base alloys and in gamma prime for the Co-base alloys. This different partitioning behavior affects various properties and its implications are presented and discussed.

738 Further development of a predictive tool for managing distortion in electron beam additive manufacturing

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Residual stress and shape distortion are inherent features of the wire fed Electron Beam Additive Manufacturing (EBAM) due to the high thermal gradients associated with the high deposition rates. Uncontrolled distortion can cause deviation from tolerances for dimensions of built parts and lead to high rejection rates. As a result, parts may require stress relief heat treatments during deposition which can add to costs. Accurate predictive models of residual stress and distortion are of high interest as crucial tools in the development of active methods for distortion control and management. Through collaboration, CSIRO and Boeing have developed a tool named C-THRU that has been successfully applied to predict residual stress and distortion and to manage post-built distortion for built parts having T-shape and complex geometry. The modelling results provide an understanding of the evolution of temperature, deformation and stress during and after completion of the build. Some challenges faced by machine operators during building parts having large and complex geometry in this process are also discussed.

739 Influence of flash treatment on pseudoelastic behaviour of biomedical Ti-25Nb-3Zr-3Mo-2Sn alloy

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The effect of heat treatment on tensile properties, especially pseudoelastic behaviour of cold-rolled Ti-25Nb-3Zr-3Mo-2Sn alloy has been studied. The as-rolled alloy shows high strength due to the fine grains and the presence of nanoscale w and a phases, but it suffers from limited ductility. The conventional solution plus ageing treatment improves the ductility, but it has an adverse effect on the pseudoelastic properties. By applying a flash treatment (500 °C for 120 s), the alloy is found to have not only balanced strength and ductility, but also improved

psudoelastic properties, such as higher recovery strains and lower elastic modulus. The microstructural changes associated with the flash treatment are revealed.

740 In-situ investigation of the kinetics of reverse austenite formation in supermartensitic stainless steel

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The kinetics of austenitisation of supermartensitic stainless steel X4CrNiMo16-5-1 (1.4418) was investigated in-situ by synchrotron X-ray diffraction, dilatometry and calorimetry. Austenitisation occurred in two stages: depending on the heating rate, the austenitisation paused at 55 to 75 vol.% of austenite and first continued after heating for at least 100 °C before the remaining martensite was transformed. For a heating rate of 1 Kmin⁻¹ the A_{c1} temperature was 550 °C; for heating rates from 5 to 50 Kmin⁻¹ the A_{c1} temperature approached a constant value, just above the Curie-temperature. Analysis of a series of samples subjected to tempering between 600 and 700 °C showed an increase of the phase fraction of reversed austenite with tempering temperature. Energy dispersive X-ray spectroscopy on the lamellar two-phase microstructure of thin foils revealed that the growth of austenite lead to nickel partitioning. For increasing tempering temperature the Ni-enrichment of austenite decreased and the M_s-temperature of reversed austenite approached the general M_s-temperature of the alloy, i.e. 120 °C. For high Ni-partitioning the M_s-temperature of reversed austenite was moved to the sub-zero Celsius region.

741 Influence of chemical composition on precipitation behaviors in high-Cr ferritic steels

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Today, structural materials in power plant have to stand higher temperature and steam pressure in order to achieve higher efficiency. Crofer[®] 22 H, a new grade of Laves phase strengthened ferritic steel, which shows excellent steam oxidation and long-term creep resistance has been developed during the last decade, and might become a promising alternative to traditionally used ferritic-martensitic steels. The present study focused on three modifications based on Crofer[®] 22 H differing in W- and Cr-content. The precipitation behaviors in the three steels were investigated by means of thermodynamic equilibrium calculations and qualitative/quantitative image analysis of high resolution SEM micrographs in parallel. In the experimental part, solution treatment (1200°C×20min) followed by precipitation annealing (600 and 650°C×10-1000h) was carried out for all the specimens. The calculation results suggested that there were two types of Laves phases present in this type of alloys, which had different chemical compositions, appeared in different temperature ranges but shared the same crystal structure; and the existence and amounts of the two types of Laves phases were influenced by the W- and Cr-content of the alloy. The experimental results, mainly concerning the evolution of precipitations up to 1000 hours at 600 and 650°C turned out to be quite complex, but became understandable in regards of possible existence of two types of Laves

phases.

742 Nanoscale transformation toughening of the hardest oxide: Nanocrystalline bulk SiO₂ stishovite

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Silicon dioxide (silica) is the most abundant oxide component on the Earth's surface and has been widely used in industry. Stishovite is a high-pressure polymorph of silica stable above 9 GPa. This material has been known as the hardest oxide at ambient conditions ($H_V = 30$ GPa). A previous study reported synthesis of nanocrystalline bulk stishovite from a bulk silica glass rod. Fracture toughness of this material was reported to be about $10 \text{ MPa m}^{1/2}$, whereas single crystal stishovite is known as a very brittle material ($K_{Ic} = 1.6 \text{ MPa m}^{1/2}$). Nanocrystalline bulk stishovite is a very hard and toughed material. In order to understand an active toughening mechanism in this material, we performed Si-K XANES measurements and TEM observations for fracture surfaces of nanocrystalline bulk stishovite. Our experimental results show that amorphous silica exists on the outermost fracture surfaces. These results indicate that huge tensile stress at the crack tip induces solid-state amorphization from stishovite to amorphous silica. This transformation accompanies a huge volume expansion of 95%. This volume expansion causes transformation toughening. In addition, this volume expansion is much larger than that of tetragonal to monoclinic transition in zirconia (4%), resulting in a thinner transformed region whose thickness is several tens of nanometers in nanocrystalline bulk stishovite materials. Thus, this toughening mechanism can be called as "nanoscale transformation toughening".

743 Effect of the severe plastic deformation on magnetic properties in superconductors

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The bulk nanostructured metals prepared by severe plastic deformation (SPD) provide a unique system to study superconducting materials, because superconducting properties can be controlled by ultrafine-grained microstructures and introduced disorders. Recent studies on bulk nanostructured Nb prepared by high-pressure torsion (HPT) have shown that superconducting parameters, such as critical temperature T_c , upper critical field H_{c2} , and critical current density J_c , increase with rotation numbers N of HPT [1]. In this study, a powder mixture of Nb - 47 wt.%Ti was processed by HPT ($N = 1, 2, 5, 10, 20, 50, 100$) [2] and magnetization measurements were performed in order to study the evolution of superconductivity during NbTi alloying process. The T_c value of NbTi is lower than that of pure Nb after HPT and decreases with increasing N . In the magnetic field, two kinds of superconducting transitions, which correspond to upper critical fields of Nb and NbTi, are observed in NbTi ($N < 5$). With increasing N , the transition becomes single phase transition of NbTi and H_{c2} increases. The results indicate that the homogeneous superconductivity appears in NbTi ($N > 20$). The vortex phase diagram and vortex pinning properties are also discussed in the present talk.

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744 High pressure synthesis of new transition metal nitrides with using laser-heated diamond anvil cell

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Nitrides have attracted much attention in the field of fundamental solid state chemistry and the functional material such as BN and GaN. The high pressure and high temperature technique offers some advantages to synthesize better crystalline nitrides, because the high pressure condition is closed reaction system. Furthermore, fluid molecular nitrogen under high pressure and temperature shows high chemical reactivity with transition metal. This potentially leads to a discovery of novel nitrides, although the high pressure synthesized sample is tiny. In this presentation, we will present the recent results of high pressure synthesis experiments on the transition metal nitrides by using laser-heated diamond anvil cell apparatus up to the pressure of 70 GPa. Thin foil of late transition metal (3d, 4d and 5d) was located at the center of sample chamber and then it was filled with nitrogen cryogenically. The infrared laser was irradiated through the diamond after compression to the desired pressure. As a result of sample characterization by X-ray diffraction, Raman spectroscopy, SEM-EDS, TEM-EELS and XPS, new nitrogen-rich transition metal nitrides were successfully synthesized. The dedicated characterization offers important information with respect to the systematicity in the crystal structures and electronic properties of new transition metal nitrides. The details of the synthesized products will be given in the presentation.

745 Metal matrix composites as environment-friendly protective coatings

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Protective coatings able to provide improved tribological behavior and corrosion resistance are demanded in many industrial applications. Although hard chrome is a very effective solution and is widely used, environmental issues related to hexavalent chromium push to develop alternative treatments. Within the framework of the HardAlt European Project, electrodeposited composite coatings have been developed and main results of this investigation are reported in this work. Boron carbide micro- and nano-particles were dispersed in a Ni-P matrix under different operating conditions, in order to produce coatings with different composition and particle content. Structural and morphological characterization was performed by X-ray diffraction (XRD) and scanning electron microscopy (SEM). Mechanical properties were evaluated by micro-indentation and the tribological performance was assessed by block-on-ring tests. Electrochemical experiments were carried out to examine the corrosion behavior of the composite coatings. Heat treatments were also performed to exploit the hardening effect of the Ni-P matrix. Interesting results were obtained for composite coatings as regards hardness values, tribological behavior and corrosion resistance, even though some improvements can be

realized. However, the results presented in this work show that composite coatings may represent a promising alternative to hard chrome for selected applications and also suggestions for further investigation are gained.

746 Characteristic features of the modulated structure appearing in the multiferroic material $\text{Bi}_{1-x}\text{Sm}_x\text{FeO}_3$ around $x=0.15$

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The simple-perovskite oxide BiFeO_3 is one of multiferroic materials exhibiting ferroelectric and antiferromagnetic properties. According to the previous studies on $\text{Bi}_{1-x}\text{Sm}_x\text{FeO}_3$ (BSFO) having BiFeO_3 as an end material, an increase in the Sm content leads to the state change from the ferroelectric R3c state to the paraelectric Pnma state around $x=0.14$. The notable feature of BSFO is that, in addition to the R3c and Pnma states, the state accompanying a modulated structure appears near the state boundary, where a remarkable piezoelectric response was obtained experimentally. However, the detailed features of the modulated structure are still open questions. We have thus examined the crystallographic features of prepared samples with $0 \leq x \leq 0.20$, mainly by transmission electron microscopy.

The present experimental data revealed that the state near the state boundary consisted of an assembly of two kinds of regions with an average size of about 300 nm. From an analysis of electron diffraction patterns taken from these two regions, the modulated structure present in one region was found to be characterized by the appearance of two modulated waves, while the other region had the R3c symmetry. As for the detailed features of the modulated structure, its normal state could be identified as the Pnma structure and wave vectors of two modulated waves were specified by $q = [1/2 \ 0 \ 0]_o$ and $q = [0 \ 1/2 \ 0]_o$ in terms of the orthorhombic notation. It was also understood that these two waves had transverse-wave characters with eigenvectors along the same $[001]_o$ direction.

747 Microstructure and mechanical properties of selective laser melted metals for biomedical applications

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Selective laser melting (SLM) process has received much attention because this process enables us to fabricate complicated shape without mold or conventional cutting tools. We are focusing on cobalt-chromium-molybdenum (Co-Cr-Mo) alloys which have been widely used in orthopedics and dentistry. The Co-Cr-Mo alloys show excellent strength and wear resistance but limited ductility owing to the formation of martensite (hcp) and the precipitation of carbides or sigma phase. It was reported that elongation of Co-29Cr-6Mo alloy could be improved due to the suppression of martensite when the selective laser melting (SLM) process was applied for the fabrication process. On the other hand, the addition of nitrogen to Co-Cr-Mo alloys improves elongation and strength with increasing Cr content in the as-cast alloys. In this study, high-nitrogen contained Co-Cr-Mo alloys builds were fabricated by SLM process to enhance

the mechanical properties. SEM observation revealed that fine cellular dendrites (c.a. 3 μm) was formed in the SLMed Co-33Cr-5Mo-0.3N alloys. This cellular dendrites was aligned parallel to the building direction, which was similar to the SLMed Co-29Cr-6Mo alloy. It should be noted that yield stress (743 MPa) and elongation (22.4%) of the SLMed Co-33Cr-5Mo-0.3N was higher than those (503 MPa and 16.4 %) of the SLMed Co-29Cr-6Mo alloy. Therefore, the application of SLM process is promising for the enhancement of the mechanical properties of Co-Cr-Mo alloys.

748 Iron-water interface under electrochemical condition

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The atomic scale understanding of metal/aqueous interface is very important in the field of electrochemistry, corrosion, and environment science. In particular, from the point of view of practicality, not only experimental but also numerous theoretical studies of iron and water interface have been carried out. Although the structure of water layers, especially the stratification of water molecules near electrode surfaces has been studied vigorously such as by experiments using X-ray and scanning tunneling microscopy (STM), still the structure of water at metal surfaces, in particular at charged surfaces remains unclear. In the present study, we have been addressed structural and electronic properties of water molecules on charged Fe(100) and Fe(110) surface by density functional theory (DFT) calculations. In addition, ab initio molecular dynamics calculations have been performed at room temperature to evaluate the thermal behavior of water molecules. Our calculations show that the charging of the surface leads to characteristic change in water molecules orientation distribution.

749 Implanted MgO is osteoinductive through the formation of a bone-inducing matrix

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The induction of bone formation by metallic magnesium was reported early in the last century [1]. The exploitation, in orthopedic surgery, was hampered by the release of hydrogen gas during the corrosion of Mg to MgO. This can be avoided by implanting MgO, transforming to MgOH and MgCO₃ in the tissue, and still inducing bone formation. Methods: MgO was implanted into rat tibia as described [2]. Samples were taken after 1 week, 2 weeks and 3 weeks of healing. The bones were analysed as by histology and EDX [2]. Results: The results show higher levels of Mg in the bone marrow of MgO-treated animals (0.2-0.3%) than in controls (0.1%) after 1 week of healing. Callus bone was seen in controls and MgO-treated bone, indicating normal bone healing in the MgO-treated tibia [3]. After 2 weeks of healing, EDX analysis showed normal levels of Mg for bone, together with C, P, and O levels compatible with hydroxyapatite, and the cortical bone did not differ from that of controls. After 3 weeks of healing the bone marrow space was filled with bone marrow, with spherical bodies, showing levels of Mg, Ca, O and P compatible with that of normal bone. Light microscopy showed amorphous bodies covered with normal bone. The results indicate that MgO implanted in bone marrow transforms to a compound matrix that induces bone formation after 3 weeks.

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750 Design of bragg-edge spectrometer at steady-state neutron source

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Neutron diffraction is a powerful tool for structural analysis and then utilized for various materials. Most of conventional neutron diffractometers are classified into two types: angle dispersive instruments used at steady-state neutron sources and time-of-flight (TOF) instruments used at pulsed neutron sources. In addition, a spectroscopic measurement method of diffraction, called “Bragg-edge transmission analysis”, is rapidly evolving with the recent progress of the pulsed neutron source. Using the Bragg-edge method, each Bragg peak of the crystal structures of samples is observed as an edge on a neutron transmission spectrum. This new method opened up a new field such as neutron diffraction imaging. The Bragg-edge transmission analysis is also valuable at the steady-state neutron source. The wavelength dependence of neutron transmission can be measured using a position sensitive detector (PSD) and an analyzer such as a prism or a bent crystal without angle scanning or TOF analysis. This concept enables effective use of a wide wavelength band of continuous neutron beam and realizes a higher efficiency diffraction measurement method than the conventional ones at the steady-state neutron sources. In this presentation, a practical design for the Bragg-edge-type diffraction instrument at the steady-state neutron sources is provided. The beam port CN-2 at Kyoto University Research Reactor (KUR) was chosen as the model of the neutron source. The result of ray tracing is shown. Possible designs applicable to materials science are discussed.

751 Compressive residual stresses and associated surface modifications induced in Ti6Al4V by laser shock processing

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Laser Shock Processing (LSP) is a materials processing technique increasingly applied as an effective procedure for the improvement of metallic materials mechanical properties in different types of components. As reported in previous contributions by the authors, a main effect resulting from the application of the LSP technique consists on the generation of relatively deep compression residual stresses fields allowing an improved mechanical behaviour, explicitly the life improvement of the treated specimens against wear, crack growth and stress corrosion cracking. In this paper, additional work by the authors in the line of practical development of the LSP technique at an experimental level (aiming its integral assessment from an interrelated theoretical and experimental point of view) is presented. Concretely, experimental results on the residual stress profiles and associated mechanical properties modification successfully reached in typical materials under different LSP irradiation conditions are presented. In this case, the specific behavior of a widely used material in high reliability components (especially in aeronautical, energy and biomedical applications) as Ti6Al4V alloy is analyzed. The

synchrotron radiation determined intensities of compressive residual stress fields achieved in the material and their mechanical protective character are discussed along with relevant associate surface modification effects (i.e. roughness, microstructure and electrochemical behaviour) for different representative cases.

752 Deformation microstructures and mechanical properties of an austenitic stainless steel subjected to warm rolling

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The deformation microstructures and mechanical properties of an austenitic stainless steel subjected to warm plate rolling were studied. The warm rolling was carried out at 300°C to different total true strains of 0.5, 1, 2 or 3. The structural changes during warm rolling were characterized by the elongation of original grains towards the rolling direction and the development of spatial network of strain-induced high-angle boundaries leading to the evolution of ultrafine-grained microstructure at sufficiently large strains. The grain refinement was assisted by the development of deformation twinning. After straining to 3, the transverse grain size decreased down to 220 nm in the warm rolled samples. The warm plate rolling resulted in significant strengthening. The microhardness increased from 2910 MPa to 4192 MPa with increase in the total strain from 0.5 to 3. Correspondingly, the yield strength approached 1005 MPa after warm rolling to a total strain of 3. The relationship between the deformation microstructures and the tensile behavior is discussed in terms of structural and substructural strengthening. The financial support received from the Ministry of Education and Science, Russia, under Grant No. 14.575.21.0092 (ID number RFMEFI57514X0092) is gratefully acknowledged.

753 Grain coarsening in niobium containing steels studied in-situ by 3DXRD microscopy

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Coarsening of austenite grains in two carbon-manganese steels alloyed with niobium (Nb) is investigated in-situ by using three-dimensional x-ray diffraction (3DXRD) microscopy at the European synchrotron radiation facility. The alloys (with Nb:C atomic ratios of 1.3 and 11.5) were annealed isothermally at three temperatures each: 1000, 1050 and 1100°C for the low Nb alloy and 960, 1000 and 1100°C for the high Nb alloy. The results of isothermal annealing reveal a very interesting behaviour: no significant change in the average grain size takes place at temperatures up to 1100°C for durations greater than 2 h. However, the material did show significant changes in grain volume at the level of individual grains. Only few grains in the specimens disappeared during the isothermal annealing. Remarkably, grains smaller in radius than about 40 nm in the low Nb alloy and 15 nm in the high Nb alloy show a net increase in grain volume, whereas larger grains show a net decrease in grain volume. These results are in contrast with 'normal' grain coarsening behaviour of a Fe-2 wt% Mn alloy (which has been

studied with the same technique) and with the traditional models and theories of grain coarsening.

754 First-principles study of energetics of deformation twinning in pure Mg

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The energetics of deformation twinning has not fully clarified from first-principles, especially for the twinning systems which require not only affine shear strain but also non-affine atomic shuffling motion, like for the $\{10\text{-}12\} \langle 10\text{-}11 \rangle$ twinning system of Mg. We here computed the free energy surface in a space spanned by two variables: strain which describes shape change of a periodic supercell, and shuffling which describes non-affine displacements of the internal degrees of freedom. The minimum energy path of the deformation twinning involves juxtaposition of both. From the shape of the free energy surface, we found the $\{10\text{-}12\} \langle 10\text{-}11 \rangle$ twinning of Mg is shuffling-controlled, that is, the reaction coordinate is dominated by non-affine displacement instead of strain. Shuffling-controlled deformation twinning is expected to have different temperature and strain-rate sensitivities from strain-controlled deformation twinning due to reduced importance of long-range elastic interactions.

755 Crystal plasticity finite element analysis of micro-tensile behaviour of dual-phase steel subjected to pre-straining

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Micro-tensile testing combined with crystal plasticity finite element method (CPFEM) was employed to clarify the deformation behaviour of the inhomogeneous microstructure developed through pre-straining in a ferrite-martensite dual-phase (DP) steel. A DP microstructure with a martensite fraction of 29% was obtained through a thermo-mechanical treatment using an Fe–0.14% C–1.00% Mn steel. Pre-strains were introduced by cold rolling at thickness reductions of 60% and 88%, CR60 and CR88, respectively. The crystallographic orientations of the pre-strained microstructures were determined using electron backscatter diffraction (EBSD) analysis. The deformed microstructures of the CR60 and CR88 samples were characterised by plastic flow and ultrafine grains in the ferrite region, respectively. Micro-tensile specimens with gauge section dimensions of $20 \times 20 \times 50 \mu\text{m}^3$, including a ferrite/martensite interface, were fabricated using a focused ion beam. Tensile tests were performed at room temperature in air and at a displacement rate of $0.1 \mu\text{m/s}$. CPFEM analysis models of the tensile specimens were uniformly divided into 5600 elements, and Euler angles obtained from EBSD analysis were allocated to each element. Considering the strong dependence of plasticity on the habit plane orientation of the lath martensite, different critical resolved shear stress values were given for in-habit-plane and out-of-habit-plane slip systems in the martensite crystals. CPFEM simulations were in good agreement with the experimental results. The CR88 specimens exhibit significantly low ductility when compared to the CR60 specimens. CPFEM simulations revealed that the strain localisation in the ultrafine-grained ferrite with a strong texture leads to

serious ductility loss in the severely-deformed DP steel.

756 Hot compaction of mechanically alloyed high nitrogen stainless steel powders by plasma sintering

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Nitrogen alloying is an effective way to improve mechanical and chemical properties of stainless steels, especially austenitic stainless ones. In addition, nitrogen is an effective element for austenite forming, nitrogen use in these alloys solves the problem of cost saving of expensive alloying elements such as nickel. High nitrogen steels are commonly produced using nitrogen pressure processing such as high pressure melting solidification and high pressure solid state diffusion at high temperature. On the other hand, it has been reported that high nitrogen stainless steel powders with nanostructures can effectively be produced by mechanical alloying (MA) of blended elemental metallic powders either in a nitrogen gas atmosphere or with metal nitrides such as iron nitride. However, since MA products are in the form of powders, much attention has been focused on hot compaction of such MA powder products. Spark plasma sintering (SPS) is a process capable of sintering at lower temperature in shorter holding time compared with a conventional hot processing process, there is possibility that microstructure produced during MA processing is retained after SPS compaction. In this study, high nitrogen nanocrystalline austenitic stainless steel powders were prepared by MA of elemental powders with iron nitride powders in an AR atmosphere by use of planetary ball mill. A microstructure of MA processed powder developed during SPS process was investigated.

757 Practical use of computer model STAN 2000 for improvement and creation of regimes of steels hot rolling on mill 2000 of SEVERSTAL

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An integral computer model STAN 2000 designed for off-line simulation and control of hot rolling on mill 2000 of SEVERSTAL was developed. The capabilities of the model include the following features: calculations of power parameters for all stands of mill 2000 for a given rolling schedule; strip temperature calculations depending on selected rolling and accelerated cooling regimes; follow-up of steel microstructure evolution at all stages of strip production and prediction of ultimate mechanical properties (yield stress, ultimate tensile stress, relative elongation and toughness); optimization of rolling regimes for existing steel grades and developing them for a new one. The STAN 2000 program is written in C++ programming language and can work on all modern Microsoft Windows family operating systems. The program has a well-designed and user-friendly interface facilitating its practical use.

The integral model was calibrated using an extensive data base on rolling regimes and forces, measured temperatures and final mechanical properties for a number of steel grades rolled on mill 2000 of SEVERSTAL with chemical compositions covering the following range of alloying elements content (mass.%): C(≤ 0.65); Mn(≤ 2.0); Si(≤ 1.0); Cr(≤ 0.9); Ni(≤ 0.6); Cu(≤ 0.5); Mo(≤ 0.4); Nb(≤ 0.05); V(≤ 0.065); Ti(≤ 0.06); B(≤ 0.003). The calculation results for

temperatures, rolling forces and mechanical properties are presented and compared with experimental data. The examples of STAN 2000 program practical implementation in hot strip production on mill 2000 of SEVERSTAL are presented and discussed.

758 Dissimilar metal joining of A5052 aluminum alloy and AZ31 magnesium alloy using laser brazing

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This investigation into the microstructure and mechanical properties of a joint produced by laser brazing between A5052 and AZ31 with AZ125 filler wire reveals that an increase in laser power from 480 to 620 W reduces the weld toe angle and increases the bead width, resulting in an enhanced wettability. Any further increase in power, however, results in a rapid increase in the thickness of the intermetallic compound (IMC) reaction layer. The IMC layer consists of $Mg_{17}Al_{12}$ on the AZ125 side and Mg_2Al_3 on the A5052 side. The tensile shear strength is increased by using a laser power of 480–590 W owing to the increased wettability, but is reduced when using a laser power of 590–620 W owing to the thickness of the reaction layer and the presence of voids, which cause brittle fracture through the brazed filler metal. At an optimal power of 590 W, a maximum tensile shear strength of 120 N/mm was obtained, which is about 55% that of the base metal.

759 Effect of misch metal addition on thermal conductivity and mechanical properties of Mg-4Zn-0.5Ca alloys

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Recently, with increasing the usages of LED lightening and smart phone with high performance and large LCD window, the researches on materials for the heat sink have been in progress. One of the important properties of materials for the heat sink is thermal conductivity, because high thermal conductivity assures a uniform temperature distribution which reduces thermally-induced stresses thereby prolongs product life. Although Mg alloys have attracted attention due to low density and castability to make thin parts, the thermal conductivities of cast magnesium alloys should be improved to use as heat sink materials. Also, increased applications of Mg alloys for heat sink industry require the development of low-cost alloys with good thermal conductivity and sufficiently high strength. According to our preliminary test, Mg-4Zn-0.5Ca-1.0La alloy has exhibited the relatively high thermal conductivity and mechanical properties in Mg-Zn-Ca-La alloy system, however the cost of this alloy was higher than that of conventional AZ91 alloys. Therefore, in this study, to reduce the cost of Mg-4Zn-0.5Ca alloy without the decrease in thermal conductivity and mechanical properties, misch metal was chosen as a low-cost element and the effect of misch metal addition on thermal and mechanical properties was investigated. The thermal conductivity of these alloys had been determined by tests for thermal properties, such as specific heat and diffusivity, from room temperature to 200 degree Celsius.

760 A study on the microstructure and the tensile fracture behavior of infiltrated TiC-steel composite

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Metal matrix composites have drawn attention owing to its low density and excellent high-temperature strength. Especially, ceramic reinforced metal matrix composites integrate respective superb properties of the metallic matrix and ceramic reinforcements. TiC is well-known for reinforcement in steel matrix because of low density, high elastic modulus and excellent wettability. In general, TiC-Fe composites have been produced by powder metallurgy but there are problems like high cost and limited scale-up of components. The present work was initiated to study the feasibility of producing of TiC-SKD11 composite by infiltration process which has cost-effectiveness and accommodates enlargement. The microstructure and the fracture behavior of this infiltrated composite have been investigated. The composite having TiC particles as much as approximately 60 vol.% was successfully manufactured by the infiltration. The complete infiltration of the molten steel was confirmed from the microstructure showing that the penetration of the liquid metal reached geometrically complex regions without any interfacial flaws. The results about atomic arrays gotten from HRTEM show that TiC and steel atoms are connected through semi-coherent nature. After the tensile tests at room temperature and 700°C, the composites were fractured prior to a detectable yielding. The TiC was fractured preferentially along the {100} plane regardless of manufacturing methods, heat-treatment and tensile test temperature. This implies that the higher tensile strength of the composite might be attributed to the load transfer strengthening from the steel matrix to the TiC particles. The tensile fracture mechanism was discussed in terms of TiC-steel matrix interface and dislocation.

761 Structural study of the electrolyte material Li₂S-P₂S₅ glasses at SPring-8

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The atomic and electronic structures of the electrolyte material Li₂S-P₂S₅ glasses used as solid electrolytes are modeled by a combination of density functional theory (DFT) and reverse Monte Carlo (RMC) simulation using X-ray diffraction, neutron diffraction, and Raman spectroscopy data. The ratio of PS_x anions based on the Raman spectroscopic results is reflected in the glassy structures of the 67Li₂S-33P₂S₅, 70Li₂S-30P₂S₅, and 75Li₂S-25P₂S₅ glasses, and the plausible structures represent the lithium ion distributions around them. It is suggested that the edge sharing between PS_x and LiS_y polyhedral increases at a high Li₂S content, and the P₂S₇ anion is more favorable for the Li⁺ ions than the PS₄ anion. The electron structure of the DFT/RMC models suggests that the electron transfer between the P ion and the bridging sulfur ion weakens the positive charge of the P ion in the P₂S₇ anions. The P₂S₇ anions of the weak electrostatic repulsion would cause it to be more strongly attracted to Li⁺ ions than the other PS_x polyhedral anions, and suppress the lithium ionic conduction. Thus, it has been demonstrated that the observation of the local structure is important for understanding the origin of high lithium ionic conduction. We suggest that the high ionic conduction in solid electrolytes can be

controlled by the polarization of PS_x polyhedral anions. This finding is a crucial key concept for designing new solid electrolytes.

762 Effects of alloy composition on phase transition temperatures of CoMnSi compounds

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CoMnSi compound shows the martensitic phase transformation from Ni_2In structure to TiNiSi structure. Furthermore, the CoMnSi compound with TiNiSi structure shows the first order metamagnetic phase transition from ferromagnetic to antiferromagnetic phases. Thus, this compound is expected as magneto refrigerant materials and energy harvesting materials, because this compound exhibits the large magnetization gap and also large magnetocaloric effects at the metamagnetic transition temperature. Control of magnetic transition temperatures is important knowledge to practical applications. In the present study, the effect of substitution of Co and Si for Mn on the phase transition temperature of CoMnSi alloys has been investigated. $Co_{(1+x)}Mn_{(1-x-y)}Si_{(1+y)}$ compounds are prepared by arc-furnace under Ar gas atmosphere. Small piece of sample cut from the button ingots. The samples are annealed at 1223 K for 2 days and quenched into ice water. The martensitic transition temperature was measured by DSC and the magnetic transition temperatures were measured by VSM. The martensitic transition temperature decreases with increasing Co, while the Si substitution for Mn is not effect on the martensitic transition temperature. The Curie temperature increases with increasing Co and Si. The metamagnetic transition temperature decreases with increasing the Si content, while that decrease with increasing Co content. Thus, the Si substitution for Mn increases the difference between the Curie temperature and metamagnetic transition temperature. These results suggest that the Si substitution more effective to control the magneto-caloric effects.

763 Soft matters containing self-propelled nanometer and micrometer-scale particles spontaneously generate large-scale mechanical network

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Cytoskeletal architectures contribute to diverse cellular activities including mitosis, cellular morphogenesis, and migration. Among these various structures, protein motors and protein filaments such as microtubules or actin filaments play important roles in the formation. However, the formation mechanism and physical properties of protein filament-motor network remain elusive. To elucidate the spatiotemporal dynamics of protein filament organization as a kind of soft matter, we established a model microtubule-motor system to characterize the various dynamics that can emerge including static networks, active networks characterized by self-rupture, and aggregated structure. The modification of motor properties can also result in a population of asters. To determine how the system properties define spatiotemporal dynamics, we constructed a coarse-grained model where a biofilamentous network is stretched and ruptured based on motor concentration. Our model reproduced all of the experimentally observed patterns and predicts a characteristic of the cytoskeletal network where elastic energy

can be stored and abruptly released as mechanical work. Together with this finding, our model system brings new insights into cytoskeletal mechanics and the development of self-regulating active materials.

764 Diffusion brazing of single crystal aerospace superalloys using composite powder as interlayer material

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The progress that has occurred with aerospace single crystal (SC) nickel-base superalloys in the past two decades has not been matched by adequate understanding of appropriate techniques for joining and repairing components made from these extremely difficult-to-weld materials. Diffusion brazing, with the use of interlayer material comprising of composite powder, has evolved as an attractive alternate method for joining difficult-to-weld structural materials. Unfortunately, commercial exploitation of this technique for joining SC nickel-base superalloys had been widely considered unfeasible due to the formation of stray-grains within the brazement, which compromises high temperature properties of the bonded materials. In this research, numerical simulation coupled with careful experimental analysis was used to develop an effective approach for utilizing composite powder as interlayer material for the diffusion brazing of SC nickel-base superalloys without the detrimental formation of misoriented stray-grains in the joint. The numerical and experimental results of the research will be presented and discussed.

765 Solid solution strengthening and atomic displacements in equiatomic high-entropy alloys with the FCC structure

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High-entropy alloys (HEAs), consisting of five or more elements, may exist as a stable single-phase solid-solution crystallizing in simple crystal structures such as the BCC and FCC structures. They are known to exhibit excellent oxidation- and wear-resistance, high hardness, excellent malleability, high-temperature strength and so on. Although the solid-solution strengthening (SSS) will be one of the most important mechanisms that dominate the mechanical strength of HEAs, it is impossible to define “solvent” or “solute” in HEAs where there are multiple principal elements. Therefore, the classical theories for SSS, which were established mostly for dilute solid solutions, cannot be applied to HEAs. In dilute solid-solutions, the solvent atoms around the solute atoms are shifted from the ideal lattice points due to the atomic size difference between solute and solvent so as to generate a spherically symmetric strain field, which acts as obstacles for moving dislocations. On the other hand, it is not so simple to estimate the magnitude of local lattice strain in HEAs because multiple principal elements with different sizes interact with each other so that all atoms are shifted from

the ideal lattice points. In the present study, therefore, we have experimentally measured the magnitude of atomic displacements averaged over the constituent elements in the quinary equiatomic CrMnFeCoNi HEA with the FCC structure by single-crystal synchrotron X-ray diffraction as well as deduced the magnitude of atomic displacements for each of the constituent elements by first-principles calculations in order to understand the SSS of the HEA.

766 Effect of focusing condition on molten area in micro-welding of glass by picosecond pulsed laser

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An ultra-short pulse laser can make it possible to absorb laser energy at the focusing point inside a glass by non-linear absorption, and micro-welding of glass can be expected without an intermediate layer and adhesive. However, molten area is greatly affected by focusing condition, because one part of this process is related to non-linear phenomena, which can be generated at high-energy intensity condition. Therefore, in this study, effect of focusing condition on micro-welding of borosilicate glass (D263) were discussed using picosecond pulsed laser. Molten area was observed by a high-speed video camera, and breaking stress of the weld glasses was evaluated. Molten area was created by the periodic movement of absorption point up and down in coaxial direction of laser beam. The usage of optical system with the spherical aberration correction made it possible to create a large volume weld joint, because absorption rate of laser energy increased. Absorption rate of laser energy was strongly influenced by pulse repetition rate, and short time period between laser pulses was effective to obtain a large molten area. An optical system with the spherical aberration correction led to stabilizing the shape of molten area, which resulted in the reliable and strong joint.

767 Thermo-mechanical and low cycle fatigue failure behavior relevant to temperature regime in a TBCed superalloy specimen

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In order to get basic knowledge on reliability of Ni-base superalloy components with thermal barrier coatings (TBCs), low-cycle fatigue (LCF) and thermo-mechanical fatigue (TMF) tests were carried out at wide temperature regimes ranged between 400-900°C. Here the TBC specimens consisted of three layers: polycrystalline superalloy substrate, CoNiCrAlY alloy bond coat and YSZ ceramic top coat. The fatigue lives of the TBCs were compared with those of the substrate alloy and the specimen only with bond coat. The experimental work clearly demonstrated that while the failure mechanisms were complicated, they were significantly changed strongly depending on the temperature regime during TMF and LCF. These characteristics are discussed based on the measurements of mechanical properties of bond coat alloy, ceramic top coat and thickness of thermally grown oxides.

768 Osteoconductivity of superhydrophilic Ti- and Zr-alloys for biomedical application

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Surface hydrophilicity is considered to have a strong influence on the biological reactions of bone-substituting materials. However, the influence of a hydrophilic or hydrophobic surface on the osteoconductivity is not completely clear. In this study, we produced super-hydrophilic and hydrophobic surfaces on Ti- and Zr- alloys using a hydrothermal treatment at 180 °C for 180 min. in the distilled water. Hydrothermal treated samples were stored in x5 PBS(-). This maintained less than 10 (deg.) in an apparent water contact angle of them. The osteoconductivity of the surface treated samples with several water contact angle was evaluated by in vivo testing. The surface properties, especially water contact angle, strongly affected the osteoconductivity and not the surface substance.

769 Early stage of phase transformation in MgYZn ternary alloys from rapidly quenched ribbons

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Phase transformation kinetics from rapidly solidified ribbons into LPSO structures in Mg-Y-Zn ternary alloys have been examined by in-situ and simultaneous small-angle and wide angle scattering/diffraction measurements utilizing synchrotron radiation. First step of transformation is crystallization of amorphous ribbons, characterized by sharp peak in differential scanning calorimetry where rapid and very strong SAXS signal corresponding growth of crystallites was observed. Very small cluster was observed even in this early stage, but there was no indication suggesting periodical segregation in c axis in this stage. After the completion of crystallization, gradual developments of cluster microstructure eventually lead to anisotropic arrangements of cluster, suggesting formation of LPSO. The relationship between SAXS and WAXD are discussed during the process. Present work has been supported by Grant-in-Aid for scientific research by MEXT, and the in-situ SWAXS measurements were performed at Photon Factory, KEK and SPring8.

770 Luminescent property and crystal structures of green-emitting phosphors Ba-Al-O:Eu²⁺

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“Barium hexaaluminate” was first described to be BaAl₁₂O₁₉ with the magnetoplumbite structure by Toropov and similar hexagonal aluminates have been investigated since it was the so-called barium hexaaluminate. Iyi has reported that there are two phases in which Ba_{0.79}Al_{10.9}O_{17.14} is phase I and Ba_{2.34}Al_{21.0}O_{33.84} is phase II. Their structures are controlled by Ba ions within Ba-O layers and the superstructure was formed by the ordering of excess Ba

ions in the later compound. In this work, we attempted to use the $\text{Ba}_{0.79}\text{Al}_{10.9}\text{O}_{17.14}$ as a host material for the phosphor. The starting powders were mixed by ball milling and pressed into pellets, heated at 1037 K for 5 h, and finally at 1823 K for 4 h. Doping of Eu^{2+} was achieved by annealing the Eu_2O_3 -containing materials at 1473 K for 3 h under 97% N_2 –3% H_2 reductive atmosphere. Excitation and emission spectra were obtained using a spectrometer (F-7000, Hitachi). In order to clarify the effect of Eu^{2+} ion doping on the photoluminescence properties, we have prepared the $(\text{Ba}_{1-x}\text{Eu}_x)_{0.79}\text{Al}_{10.9}\text{O}_{17.14}$ phosphors with the x-values ranging from 0 to 0.16. The phosphors emitted the bluish green-color, in which the broad emission spectrum was observed in the wave range of 450–550 nm when excited by ultraviolet light at around 305 nm. One of the phosphors with $x = 0.12$ showed the highest PL intensity.

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771 Interaction between recrystallization and austenite formation in cold-rolled dual-phase steels during non-isothermal inter-critical treatments

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Dual-phase steels are obtained by an intercritical treatment during which recrystallization and phase transformation may occur simultaneously. This study is focused on the interaction between recrystallization phenomena and austenite formation in a cold-rolled advanced high strength steel (0.17C-1.76Mn-0.42Cr-0.34Si). Interrupted isothermal heat treatments below A_{c1} have been performed in order to assess the kinetics of recrystallization via hardness measurements. These data were used to calibrate a Johnson-Mehl-Avrami-Kolmogorov law leading to a unique set of parameters in a wide range of temperature and time. This approach has been extended to non-isothermal treatments (i.e. combination of heating and holding). It has been used to investigate the recrystallization and phase transformation overlapping occurring during isothermal inter-critical treatments. It suggests that (i) recrystallization always starts before the massive transformation during heating and (ii) the degree of recrystallization at the austenite start temperature is greatly influenced by the heating rate and plays a major role on the austenite formation kinetics. Results of this paper highlight large temperature-time domains where recrystallization and austenite formation advance concomitantly. This approach provides a powerful tool for optimizing the heat treatments depending on heating rate and holding time and temperature.

772 Fabrication of nano/micro structure of III-V semiconductors by anodic etching and their application

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Although conventional lithography techniques have been widely used to produce various ordered microstructures, it is still necessary to develop novel micromachining processes that

can control the pattern precisely through a relatively easy process at a low cost. We have reported the fabrication of ordered silicon microstructures such as silicon convex arrays and silicon nanopores patterns with regular periodicity of the order of micrometers by combining colloidal crystal templating and site-selective chemical etching using patterned noble-metal thin films as catalysts [1]. In addition, sub-100 nm silicon nanohole arrays were fabricated by resist-free nonlithographic method, that is, a combination process involving site-selective electroless deposition of noble metals through anodic porous alumina and subsequent metal-assisted chemical etching. Under optimum conditions, the formation of deep straight holes with an ordered periodicity (e.g., 100 nm interval, 40 nm diameter, 2 μm depth, and high-aspect-ratio of 50) was successfully accomplished [2]. Micro patterning of GaAs and InP in the same manner was also performed. In the present study, we fabricated ordered microstructures of GaAs, with high-aspect-ratio of 350, such as triangular pillar arrays and hole arrays, as well as other semiconductors by combining anisotropic chemical etching through a honeycomb photoresist mask prepared by sphere photolithography and isotropic anodic etching [3]. Application of these microstructures will be discussed.

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773 Structure and ionic conductivity of Na-P-S superionic conductors studied by neutron and X-ray scattering

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All-solid-state sodium batteries with excellent safety have attracted much attention as one of potentially candidates of post-lithium ion batteries because of great abundance and low cost of sodium. Recently, it has been reported that Na_3PS_4 glass-ceramic synthesized by annealing of $(\text{Na}_2\text{S})_{75}(\text{P}_2\text{S}_5)_{25}$ glass has a conductivity in the order of 10^{-4} Scm^{-1} at room temperature (RT). Furthermore, all-solid-state sodium rechargeable cells with Na_3PS_4 glass-ceramic as a solid electrolyte were confirmed to work successfully at RT ^[1].

In this study, we performed a structural analysis based on neutron and X-ray scattering data for $(\text{Na}_2\text{S})_x(\text{P}_2\text{S}_5)_{100-x}$ glasses ($x = 50, 60, 67, 75$) and Na_3PS_4 glass-ceramic. Neutron and synchrotron X-ray total scattering experiments were carried out with the high intensity total diffractometer (NOVA) ^[2] at the BL21 beam line of MLF in J-PARC and with a horizontal two-axis diffractometer at the BL04B2 beam line ^[3] in SPring-8, respectively. Moreover, reverse Monte Carlo (RMC) modeling ^[4] based on neutron and X-ray scattering data was performed to derive three-dimensional atomic structures of $(\text{Na}_2\text{S})_x(\text{P}_2\text{S}_5)_{100-x}$ glasses and Na_3PS_4 glass-ceramic. In our presentation, we will report a relationship between structures and ionic conductivities in Na-P-S superionic conductors.

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774 Rapid measurement of texture of metals by time-of-flight neutron diffraction at iMATERIA and its applications

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Crystallographic texture is one of the most important aspects of microstructure. Neutron diffraction can detect the crystal orientations not only from grains on a surface but also from inside of the bulk material. Therefore, the texture measured by neutron diffraction relates to the properties of the overall sample. We have developed the rapid texture measurement scheme by using the multi-detector Time-Of-Flight neutron diffractometer, iMATERIA, with the high power neutron source at J-PARC, Japan. By applying Rietveld Texture Analysis (RITA) method, it became possible to measure an ODF (Orientation Distribution Function) of metallic materials with enough angular resolution within several minutes. This enabled in situ texture measurement with a fine time resolution during plastic deformation and heat treatment.

775 Revealing the mechanism of magnesium oxidation with a synchrotron light

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Nowadays, everyone agrees that magnesium(Mg) is a material extremely attractive for various applications in light-weight mobility and bio-medicine. At the same time, it is well recognized within magnesium community as well as among other experts working in adjoining areas that a major improvement in the control of Mg (bio-)corrosion is absolutely necessary to make this material widely adopted. A significant obstacle on the way of Mg (bio-)corrosion control is insufficient understanding of basic mechanisms involved in the oxidation of magnesium surface. Until recently, experimental techniques available to scientists and engineers in this area only allowed to prepare surfaces of basic materials, and then to examine the products of oxidation. However, in order to achieve a significant advancement in this field, deeper, lower-scale understanding is necessary.

Herewith we present a novel approach allowing comprehensive investigation of atomic-level mechanisms of oxidation initiation and propagation in magnesium and Mg alloys. Namely, X-Ray photoelectron spectroscopy(XPS) using synchrotron light at Elettra was used as a surface sensitive direct method to determine the valence of Mg and O and valence band states at the early stage of oxide formation. In particular, very beginning and kinetics of oxidation on three principal crystallographic planes of pure Mg, (0001), (10-10) and (11-20), were investigated using this technique. In addition to clarifying the mechanisms of oxygen adsorption on free magnesium surface followed by chemical oxide formation, this investigation also revealed significant differences in both initiation and growth of MgO film on crystallographic orientation of Mg.

776 In situ tests of the steam generator

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The demonstration tubes made of new developed martensite steel ZL3 were tested in two power plants of Kievenergo (Ukraine) within the project “Z-ultra” of EC 7th framework programme. Two types of steam superheaters were chosen in which four demonstration tubes have been inserted. The aim was to select by calculations the most loaded sites of superheaters which were recognized and qualified as the most preferable places for new tubes insertion.

Two tubes (#1 and #2) $\varnothing 42 \times 6 \times 735$ mm and two tubes (#3 and #4) $\varnothing 42 \times 7 \times 735$ mm have been produced, non-destructive tested and welded into corresponding superheaters. Actual temperature data were recorded on line from thermocouples situated at top of tubes connected to tubes #2 and #4. Then tubes #2 and #4 were cut out after about 3850 hours of service, but tubes #1 and #3 were left for further service during about one year more.

Main results of the creep experiments on steel ZL3 were used for life assessment of mentioned tubes. The Larsson-Miller parameter was calculated using the creep data in order to plot the von Mises equivalent stress versus this parameter and then to extrapolate the curve to the actual equivalent stress on the external surface of each tube. With respect to the temperature of external surface of tubes this kind of extrapolation gives the values for the Larsson-Miller parameter and from the definition of the LM parameter the lifetime of the tubes.

777 A new method to study the thermodynamics of homogeneous nucleation of bcc phase from fcc in pure iron by molecular dynamics

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A new method is developed in the present paper to study the thermodynamics of the homogeneous nucleation of bcc phase in pure fcc iron at 200 K by Molecular Dynamics simulations using an embedded-atom method (EAM) potential. In this method, the potential energy of a selected group of atoms is recorded as a function of time. Initially these atoms all have the FCC-structure, but a BCC-nucleus is formed within the selected group of atoms and time frame. The plot of the energy change of the nucleus versus the nucleus size allows the comparison of the simulation results with the classical nucleation theory (CNT). From that comparison, the critical nucleus size (r^*) and the interface energy (γ) were computed and found to coincide well with experimental values. The energy barrier of the homogeneous nucleation of bcc phase is related to the driving force for the fcc-to-bcc transformation at 200 K for the applied EAM potential.

778 Application of thermo-calc TCFE7 for high-alloyed mottled cast iron

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The thermodynamic modeling of alloy systems consisting of stable and metastable phases e.g. high-alloyed mottled cast iron can be problematic. Thermodynamic databases are well-developed for low, medium and high alloyed steels (e.g. HSS) but the application of those databases is not yet very common for high-alloyed (mottled) cast irons.

This paper shows the application of the Thermo-Calc TCFE7 database to predict the microstructural phases of selected mottled cast irons. The examined materials contain different amounts of strong carbide forming elements such as niobium, vanadium, molybdenum and chromium. The microstructure consists of different carbide types (MC , M_2C/M_6C , M_3C , $M_7C_3/M_{23}C_6$), existing next to a considerable amount of graphite precipitations, embedded in a martensitic matrix. The Thermo-Calc software together with the TCFE7 database are used to calculate so-called property diagrams which show the amounts of the formed phases in relation to the temperature. Additionally Scheil-Gulliver calculations are performed to simulate the effects of microsegregation during solidification. The results from the thermodynamic calculations are compared with measurements on cast samples in the as-cast and heat treated condition. Those measurements include quantitative light-optical analysis, SEM with SE/BSE detector, EDX measurements for the distribution of the alloying elements as well as XRD (X-ray diffraction) measurements. The investigations show the possibilities which are offered by thermodynamic calculations for high-alloyed mottled cast iron as well as the limitations and the compromises which have to be taken into account when calculating stable and metastable phases existing next to each other.

779 Dissolution and precipitation behaviour in steels microalloyed with niobium during thermomechanical processing

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The thermomechanical processing of high strength low alloy (HSLA) steels during low-temperature roughing, followed by rapid reheating to higher temperatures was investigated to better understand the Nb dissolution kinetics in austenite, and the subsequent precipitation behaviour during the final finishing passes. For comparative purposes, two experimental 0.06 wt% C steels were studied, one containing 0.03 wt% Nb (Nb steel), and the second containing both 0.03 wt% Nb and 0.02 wt% Ti (Nb-Ti steel). Processing of these steels consisted of a simulated roughing schedule, with the final roughing pass taking place at 850°C. The strain-induced precipitation intensity in the steels subsequently quenched were characterised using transmission electron microscopy. Following this, the steels were rapidly reheated at a rate of 10°C/sec to a temperature of 1200°C, held at temperature for various times, and water quenched to room temperature so that both the precipitate dissolution kinetics, together with the austenite grain coarsening kinetics could be established.

780 Deformation behavior of inhomogeneous layered microstructure

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High temperature deformation of material having inhomogeneous layered microstructure has shown remarkable improvement in ductility in comparison to that shown by material having constituent microstructures. It is clear that materials with this type of microstructure are not following “rule of mixture” type of behaviour. Concomitant microstructural evolution and high strain rate sensitivity throughout the deformation is the reason behind high uniform superplastic elongation. Multipass friction stir processing (FSP) technique can be used to get inhomogeneous layered microstructure in any material. Different proportions of fine and coarse grain microstructure can be generated using FSP. In the present work, material with inhomogeneous layered microstructure generated using FSP was subjected to deformation at temperature of 500 °C at strain rate in the range of 5×10^{-4} to $1 \times 10^{-2} \text{ s}^{-1}$. It was observed that the inhomogeneous layered microstructure with more than 50% fine grain microstructure is required to get higher elongation to failure.

781 The role of preliminary heat treatment in the formation of ultrafine-grained structure in the implementation of the combined process "rolling

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In recent years was developed a number of new methods of processing of metals by pressure aimed at obtaining metal with sub-ultrafine-grained structure, the basic principle of which is the realization in the process of deformation of simple shear scheme. One of those ways is pressing blanks in matrices of different designs, in particular in equal-channel step die. However, this method has a significant disadvantage - it can't be used for deforming of lengthy billets. Another disadvantage of this method of deformation is that it does not provide the continuity of the pressing process.

For removing these shortcomings at the Department "Metal Forming" of Karaganda state industrial University was developed a combined process "Rolling-pressing" using equal-channel step matrix with calibrated and smooth rolls.

This work investigates the impact of the proposed combined process "rolling-pressing", and also preliminary and final heat treatment during the implementation of this combined process on the evolution of microstructure and mechanical properties of copper alloy. Obtained in the course of these studies, the results indicate that the proposed technology can be recommended for implementation in production to produce blanks of ferrous and non-ferrous metals and alloys with ultrafine-grained structure and high level of mechanical properties.

782 Fundamental aspects of rolled zn alloy sheet formability: Structure-property and failure mode relationships

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Sheet metal formability is an important quality parameter of the Zn alloy rolled sheet for the manufacturing of specially formed parts in construction industry, such as gutters, tubes and roofing assemblies and accessories using harsh stressing conditions under high strain rate forming techniques at low or even subzero temperature environmental conditions. Bending stress orientation is also an important process parameter, since texture sensitivity of Zn alloys strip affects ductility capacity in direction transverse to the original rolling direction. In the present work, critical testing methods are employed in order to assess the formability of a Zn-Ti-Cu alloy, evaluating, therefore, the anisotropic properties of the produced sheet. The determination of plastic strain ratios and the induced combined mathematical expressions, utilizing bi-axial strain measurements for the various test directions (0, 45 and 90 degrees towards the RD), together with the performance of cupping tests are compiled, aiming to rank and interpret the bending and sheet metal roll-forming capability. Moreover, the microstructural characterization is realized to address the influence of grain and phase structure on the sheet metal formability and identify potential optimization routes. Fracture analysis approach elucidated the micro-mechanisms prevailed in damage evolution and accumulation during monotonic loading, signifying the importance of microstructure development during thermomechanical process history.

783 Nanostructure and thermal stability of the oxide dispersion strengthened ferritic steel PM2000 after dynamic plastic deformation

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Oxide dispersion strengthened (ODS) steels have extraordinary properties in terms of strength, creep resistance, and irradiation tolerance, which are supposed to originate from the well-dispersed oxide nanoparticles. The oxide nanoparticles are commonly thought to be both stable and strong, to maintain their properties at high temperatures. In this study, an ODS ferritic steel PM2000 containing yttrium-aluminium oxide nanoparticles as dispersoids was compressed by dynamical plastic deformation (DPD) at high strain rates and ambient temperature. In samples deformed to an equivalent strain of 2.1, nanoscale lamellar structure with lamellar boundaries almost perpendicular to the compression axis and an interlamellar spacing of about 70 nm was obtained. The nanoscale lamellae exhibit a strong $\langle 100 \rangle + \langle 111 \rangle$ duplex fibre texture. Different boundary spacings and different stored energy densities for regions belonging to either of the two fibre texture components result in a quite heterogeneous deformation microstructure. Upon subsequent annealing at 715 °C, preferential recovery and formation of recrystallization nuclei is found in the $\langle 111 \rangle$ -oriented lamellae, which had the higher stored energy density in the as-deformed condition. In the course of recrystallization, the initial duplex fibre texture is replaced

by a strong <111> fibre recrystallization texture. The evolution of the oxide nanoparticles with increasing plastic strain was characterized in detail by electron microscopy and oxide nanoparticles with diameters less than 20 nm were found to deform plastically.>

784 Innovative thin films by DC reactive pulsed co-sputtering

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In the present study, biphased thin films coatings of silicon dioxide and titanium dioxide have been investigated. The purpose is to combine the optical properties of transparency and good mechanical properties of a material like vitreous silica with the photocatalytic properties of titanium dioxide. We explore the opportunity to employ an innovative co-sputtering process working in DC reactive pulsed sputtering. DC reactive pulsed sputtering is, a recent technology in the field of thin films. This technology has appeared in several huge coating companies and perspectives are really promising. A pulse of very high amplitude is applied to the cathodes. The purpose is to improve significantly the ionization of sputtered species material. The optical, mechanical properties and microstructures of the “pulsed” coatings are estimated and compared with those of coating presenting the same composition but elaborated using conventional magnetron sputtering processes.

Combined techniques allowed us to understand the mechanisms of formation of the composite thin films and the evolution of their properties. XRD and absorption spectroscopy enable to analyze the nature of the material. Mechanical properties (indentation, scratch tests) permit to reflect the properties of thin films while methylene blue tests are achieved to demonstrate photocatalytic effects of the titanium dioxide thin films. Our results bringing new insights on the DC reactive pulsed sputtering process that could lead to significant advances on surface treatment and coating of industrial glass.

785 Experimental investigation of influence of high strength fiber reinforcement on concrete

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In this work, the mechanical properties of polypropylene fiber reinforced micro concrete and the effect of super plasticizer is experimentally investigated. Cement castings of dimensions 150mmx150mmx150mm cast in accordance with IS:516-1959. The concrete is mixed and compacted for homogeneity and allowed to cure at 27°c and 90% humidity. The specimen cured for 7 days and 28 days were tested for compressive strength using compressive testing machine. Also the XRD and SEM studies are conducted for the tested samples.

Keywords: Compressive test, IS, polypropylene fiber reinforced micro concrete, XRD, SEM.

786 Development of novel Mo-Ni-Si-B metallic glass with high sustainability

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Superior properties of materials in harsh environments are required in modern industries, and metallic glasses (MGs) with excellent properties have been the subject of active research for applications in extreme conditions. However, the drawback in low thermal stability of the MGs has limited their applications as structural materials at high temperature. While refractory metal-based MGs have been expected to have high thermal stability and desirable mechanical properties, refractory metal-based alloys do not easily form fully amorphous state due to their high T_m . Here, we report a novel Mo-Ni-Si-B MG fabricated by melt-spinning process, with high T_x of over 1100 K, extremely high HV of ~27.5 GPa and relatively low E of ~364.3 GPa. The dense cluster-packing model suggests that the addition of B up to 10 at.% can occupy vacant cluster-interstices of (Mo,Ni)-Si cluster arrays, which results in a more efficiently dense-packed cluster structure, and systematically increases the GFA in Mo-Ni-Si-B alloys. In particular, the H/E and $H^2/(2E)$ ratios of Mo-Ni-Si-B MG, which reflect wear resistance and resilience, exhibit the highest values among various hard ceramic materials as well as MG-forming alloys developed up to now. These advantages of Mo-Ni-Si-B MG can be used more widely to form a MG coating layer on various substrates as a promising high temperature wear-resistant coating material. Furthermore, the same idea might be used to form a MG-nitride nanocomposite coating layer by reactive deposition in N_2 ambient, with highly lubricative property and high wear-resistance, especially at high temperature.

787 Microstructural factors affecting deformation and fracture behaviors of advanced austenitic steels during creep

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The demand for higher efficiency and reduced CO_2 emissions in energy-conversion systems, draws attention to the use of higher steam temperature and pressures. In order to establish the technical feasibility of the new power concept, development of appropriate materials and their properties should be crucial. Therefore, Ni-based superalloys which are typically strengthened by γ' intermetallic are considered. However, they have problem on the production costs. For this reason, the present works aim to study on the feasibility of microstructure design to improve creep rupture life and to increase oxidation resistance of austenitic stainless steels, especially Super 304H and AFA(Alumina-Forming Austenitic) stainless steels. Conventional Super 304H (Fe-18Cr-9Ni-3Cu-0.8Mn-0.5Nb) has 0.8% wt Mn that is known for bad effect of oxidation property. Thus, this study reduced Mn 0.8 to 0.5. And, AFA steel (Fe-20Ni-14Cr-0.9Nb-2.5Al) was recently developed by ORNL to increase oxidation property. This alloy has single γ phase and capable of alumina scale formation. This Al_2O_3 is more stable than Cr_2O_3 . After creep test, it was observed from both steels that internal cracks initiated from grain boundaries. Therefore, optimal microstructures were suggested, and the practical methods have been successfully

implemented to obtain suggested microstructures. The modified heat treatment has been developed to produce the serrated grain boundaries as for the one way to strengthening grain boundaries at high temperature. Furthermore, 5% pre-strain was taken to induce intragranular precipitate. The microstructural factors were discussed in terms of their contribution to better creep resistance.

788 Dynamic tensile extrusion behavior of metals (Cu, IF-Steel, and Ta)

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Dynamic tensile extrusion (DTE) tests with the strain rate order of $\sim 10^5 \text{ s}^{-1}$ were conducted on OFHC Cu, interstitial free (IF) steel, and Ta. DTE tests were carried out by launching the sphere samples to the conical extrusion die at a speed of 450~550 m/sec in a vacuumed gas gun system. Ta was fragmented into 6~7 pieces and showed a DTE elongation of ~900% (DTE elongation: sum of the axial elongation of each fragment). OFHC Cu was fragmented into 4 pieces with a DTE elongation of ~500%. IF steel exhibited the smallest DTE elongation of ~450%. In order to examine the DTE behavior of these metals under very high strain rates, a numerical analysis was undertaken by using a commercial finite element code (LS-DYNA 2D axis-symmetric model) with the Johnson - Cook model. The results of numerical analysis reasonably agreed with experimental in terms of the number and shape of fragments and DTE elongation. In addition, microstructural evolution of these metals during DTE deformation was examined by electro back scattered diffraction technique.

789 Effect of Aluminum content on texture formation behaviors in magnesium alloy

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Magnesium alloy was an attractive material due to its specific strength and lightweight alloy. However, magnesium and its alloy have low room temperature formability, due to its hexagonal close packed crystal structure. In order to improve the formability, many studies on texture control have been extensively investigated, whereas Studies on the effect of the solute element in the formation of the texture is insufficient. In the previous studies on texture formation behavior in Al-Mg alloy during high temperature compression, the specific texture developed with an increasing of solute concentration. In this study, the texture formation's behavior in reverse composition as Mg-Al based alloys (AZ31, AZ61, AZ91) were examined under various deformation conditions. AZ system magnesium alloys with different Al solute concentration were machined out and rolled with a rolling reduction of 30% at 673K. Uniaxial compression test specimens were annealed at 723K for 1h. Uniaxial compression tests were carried out in various deformation conditions. After deformation, Texture measurements and crystal orientation distribution were conducted by X-ray diffraction methods on mid-plane section of the specimens. In this study, (0001) orientation fiber texture was formed and dynamic recrystallization was occurred regardless of the deformation conditions. Dynamic recrystallization and formation of basal texture occur continuously during the deformation. This

means that the basal texture is stable during the deformation. Also The main component and its sharpness of texture vary depending on the Al concentration and deformation condition. AZ91 with highest aluminum concentration show that the (0001) orientation remarkably develops.

790 Effects of processing parameters on microstructure for semisolid forging of A356 alloy

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The effects of processing parameter of semisolid forging on microstructures of A356 alloy were investigated. The evolution of microstructures was affected by the solid fraction, the forging pressure, and the elemental addition of Sr. Electromagnetic stirring was used in forming the semisolid slurry followed by forging. Two specimens from different positions, which one corresponds to the location where forging pressure is applied directly, and the other where the slurry was squeezed and extruded indirectly, was tested. The microstructural characteristics, such as the morphology of the primary Al particles and the eutectic Si, were observed and compared. As the solid fraction increased, the morphology of the primary Al particles was changed from a rosette-like to globular. As the forging pressure increased, the grain size of the eutectic Si became finer. An increase in the forging pressure improved the interfacial heat transfer coefficient between the semisolid slurry and the mold wall, resulting in an increase of the cooling rate for growth of eutectic Si. Sr was added to modify morphology of the eutectic Si to fine and functioned as nucleation site.

791 Cracking in Hot-Dip Zn-Al-Mg alloy coatings on a steel sheet

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The present study was aimed at investigating a basic cause of cracking in hot-dip Zn-Al-Mg alloy coatings on an extra deep drawing quality (EDDQ) steel sheet. The electron back scattering diffraction (EBSD) technique was employed to examine the crystallographic planes of the cracks generated before and after bending deformation of the coated steel sheets. It was clarified that the occurrence of cracking in the Zn-Al-Mg alloy coatings absolutely depends on the orientation of the primary Zn and eutectic (Zn) phases. Finally, we proposed a cracking model based on the combined effect of the anisotropy of the Young's modulus in the phases constituting the coating and tensile stresses induced in the coatings due to the difference of the coefficients of thermal expansion between the substrate and coating.

792 Wool keratin fibrils sponges for bone tissue engineering

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Among the natural polymers proposed for biomedical applications, seeking for biocompatibility, biodegradability and, enhanced cellular adhesion, keratin-based materials have emerged as promisingly candidates. Keratin, found in hair, wool, horn, hooves and nails, shows the highest amount of the amino acid cystine in comparison with other proteins. Wool shows a complex histological structure: the external envelop (cuticle) is made of overlapping cuticle cells arranged like roof tiles which wrap the cortex and the cell membrane complex. The cortex is the bulk made of elongated spindle shaped "cortical cells" (fibrils), while the cell membrane complex, sticks the cortical and cuticle cells together.

Keratin fibrils sponges have been produced by disruption of the histological structure of the wool fibres through mild alkali treatment, followed by ultrasonication, casting and salt-leaching. The sponges showed highly interconnected porosity (93 %) and a microscopic structure which mimicks the Extracellular Bone Matrix (ECM). The alkali treatment converts intermolecular disulphide cystine bonds into shorter monosulphide lanthionine bonds resulting in an improvement of the thermal and water stability. The sponges show a volume swelling in water up to 38 %, however, sponges were stable in water without structural changes and showed excellent resilience to repeated compression stresses. The sponges showed cell adhesion and proliferation for the SAOS-2 cell line, according to in vitro biocompatibility-cell viability assays, due to the excellent biocompatibility of wool keratin and the unique structure of the cortical cells network, with controlled-size macroporosity for cell guesting, in addition to structural interconnected micro-porosity suitable for nutrient feeding.

793 Modelling and simulation of Q&P steels

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Two important objectives of the automotive industry are the decrease of the body-in-white weight and the improvement of the passenger safety. High strength steels (HSS) are widely used to achieve these objectives. Quenching and partitioning (Q&P) has recently been proposed to achieve high strength martensitic steel grades, which contain a considerable amount of retained austenite. Due to their microstructure these new steel grades combine a high tensile strength with good elongation values, as long as cementite precipitation is avoided.

A model describing the involved phase transformations is presented. Special focus is put on the cementite precipitation and how it is influenced by Silicon and Aluminium additions. Exemplarily results of these simulations are compared with microstructures obtained in laboratory scale tests.

794 Recrystallization twinning during primary recrystallization in stable single crystals of fcc metals

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The early stages of recrystallization have been characterized in stable single crystals of fcc metals of different stacking fault energy to rigorously quantify the orientation relations between nuclei and simple deformation structures as well as the intensity of recrystallization twinning. Goss{110}<001> and brass{110}<112> oriented samples were deformed in a channel-die to develop a homogeneous structure composed of two sets of symmetrical microbands and then lightly annealed. SEM and TEM orientation mappings demonstrate a strong relation between as-deformed orientations and the limited number of recrystallized grain orientations. The misorientation angles across the recrystallization front are mostly grouped in the ranges of 25-55° around axes located near, but not at, the normals of all four {111} planes. The quantity of recrystallization twins strongly depends on the analysed surface; the intensity is the highest in areas near-the free surface and significantly decreases as the sample centre approaches. The most frequent situation occurs when the twinning plane normal is situated near the rotation axis, i.e. near of all the four {111} planes. Another case of twinning occurs if the normal to the twinning plane is situated well away from the rotation axis, i.e. the twinning in the grain occurs on a plane other than that of the <111> pole lying near the rotation axis. This usually leads to different misorientations of both recrystallized areas with respect to the deformed state.

795 New materials for all-solid-state thin film Li and Li-ion batteries

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The explosive growth of portable electronics, generally battery-powered, has triggered the race for the development of high-performance microprocessors, Systems-on-a-Chip (SoCs) or DRAM, using low power consumption integrated circuits. As a consequence, the energy supply of such optimized components can be operated today also by miniaturized power sources such as microbatteries (1 to 500 µAh). All-solid-state microbatteries have the advantage to be manufactured by vacuum deposition processes, widely used in the microelectronics industry. They are built up through the deposition by PVD techniques of current collectors, the positive electrode, the electrolyte and the negative electrode on a rigid or flexible substrate. Actually, about ten layers are stacked to form a complex system with a total thickness of about 10 µm. The choice of the active materials (electrodes, electrolyte) appears quite different from the one for conventional Li-ion batteries due to the specific applications of these micro-power sources and their manufacturing process. Therefore, our aim is to tailor thin film properties by tuning the sputtering parameters in order to improve the performance of each active constituent of the cell as well as the overall behavior of the microbattery [1-5]. [1] V. P. Phan, B. Pecquenard, F. Le Cras, Adv. Funct. Mater., 22 (2012) 2580-2584 [2] M. Ulldemolins, F. Le Cras, B. Pecquenard, Electrochem. Comm., 27 (2013) 22-25 [3] B. Pecquenard, F. Le Cras et al., ACS Appl. Mater. Interfaces, 6 (2014) 3413-3420 [4] B. Pecquenard, F. Le Cras et al., Electrochem. Comm., 51 (2015) 81-84 [5] F. Le Cras, B. Pecquenard et al., Adv. Ener. Mater., 2015, DOI: 10.1002/aenm.201501061

796 Design of hybrid and composite tool steels by mechanical milling and spark plasma sintering

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Power metallurgy is a well consolidated route to produce tool steels. Hot Isostatic Pressing followed by extrusion/forging and final heat treatment lead to a finer microstructure and improved properties than similar wrought grades obtained from ingots.

The microstructure can be further refined by mechanical milling. The repeated impacts between powder particles and the balls/vial cause the their fragmentation and induce a high defects density. The crystallite size is reduced down to nanometric scale and considerably strain hardening is observed. During sintering recrystallization is generally easier than in unmilled powders, so that the use of sintering techniques like Spark Plasma Sintering, which permits to achieve near full dense materials at lower temperature and for shorter time than HIP, is fundamental to preserve the benefits induced by mechanical milling. Present results highlight a final grain size close to 1µm for AISI H13 hot work tool steel and even finer for AISI M3:2 high speed steel. The process described above has been extended to the production of a hybrid tool steel obtained by mechanical milling and spark plasma co-sintering blends of these two grades. The results highlight the possibility to modulate the properties (hardness, fracture toughness) of the new steel according to the relative amount of the two base powders.

The same powder metallurgical route has been used for the production of particle reinforced tool steel. A tool steel powder has been mechanically alloyed with TiC, TiN and TiB₂ particles showing that a remarkable increase in wear resistance can be obtained.

797 Crystal growth under steady shear-flow field on molecular dynamic simulation

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Mechanism of solidification is of great interest both to experimentalist and theorist, as it determines the mechanical and thermophysical properties of formed crystalline structures, as well as connects with the problem of glass-forming ability. In realistic situation, solidification is always accompanied by some flow field, either in a natural way (e.g. gravity) or exerted way (e.g. electromagnetic field). Although effect of this flow field has been widely investigated in experiment and numerical simulation, a first principle, i.e. atomic level understanding, is still lacking. We conduct molecular dynamic simulation for the composition Ni₅₀Al₅₀, with the crystal-liquid interface along <001> direction. Steady shear flow field is introduced by momentum reversing method. Advantage of this method has: (1) crystalline structure keeps static in the flow field; (2) pressure on the direction perpendicular to the interface is controlled. Results show that a linear flow velocity profile is established ahead of the interface, during the process of interface heading or backing. In supercooled liquids, strong flow field can switch the growth of crystal to melting, acting as temperature increasing. However, we find a non-monotonic behavior for the growth velocity in small flow field region. This indicates weak flow field can facilitate atomic attachment on interface, then increase growth velocity, which coincides with experimental observation.

798 A ceria-dispersed nickel aluminide coating with the increased resistance to high temperature oxidation

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Diffusion aluminide coatings are widely used on superalloys in gas turbines, specially the hot section components to protect their surface from oxidation, because they can thermally grow a protective scale of alumina. In a practical high temperature atmosphere, the aluminide coating is degraded, because of aluminum consumption not only by oxidation but also by the interdiffusion between the aluminide and the underlying metallic substrate. Recently, a CeO₂-modified ultrafine-grained (UFG) aluminide coating has been developed through a low-temperature aluminizing on an electrodeposited nanocrystalline Ni with dispersion of CeO₂ nanoparticles. Compared to conventional coarse-grained (CG) and UFG aluminide coatings, the novel CeO₂-dispersed UFG counterpart exhibits increased resistance, not only against high temperature oxidation through promoting the formation of an alumina scale with enhanced adhesion to the coating substrate, but also against an unacceptable degradation of the aluminide by interdiffusion through self-forming a CeO₂ diffusion barrier between the aluminide coating and metal substrate. These results and responsible mechanisms of CeO₂ on aluminide oxidation and interdiffusion will be presented in three parts of the presentation: i) effect of CeO₂ on thermal growth of the alumina scale, ii) effect of CeO₂ on the adhesion of the alumina scale, and iii) self-formation of the CeO₂ diffusion barrier.

799 Microstructure and mechanical properties of welded joint of TMCP890 steel and the effect of postweld heat treatment

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TMCP890 is a HSLA steel produced by thermo-mechanical controlled process, which has yield strength of 890 MPa and good toughness. After welding the heat affected zone toughness is higher than that of parent metal. 250°C postweld heat treatment doesn't affect the toughness of welded joint. But after 480°C postweld heat treatment the toughness of weld metal and heat affected zone is reduced remarkably. After 600°C postweld heat treatment the toughness of weld metal and heat affected zone is damaged seriously. Optical microscope, SEM, TEM and EBSD are used to analyze the microstructure of experimental samples. It is found that filmy residual austenite and low quantity small sized M-A constitutes contribute to the high toughness of HAZ in a great deal, besides the reason of small effective grain size. The adverse effect of high temperature postweld heat treatment on toughness is because of the precipitation of large sized cementite particles.

800 Modelling the static recrystallization kinetics of microalloyed twip steels with different alloying contents

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In recent years, high-Mn austenitic steels have attracted increasing interest due to their excellent strength-ductility combination and energy absorption capacity. Although many works have analyzed their mechanical behavior, in order to introduce TWIP steels with superior mechanical properties the parameters of thermomechanical processing should also be optimized. For instance, austenite grain size evolution and the extent of strain accumulation during hot rolling can only be controlled if the static recrystallization kinetics are known. However, at the moment, experimental data and models describing the recrystallization behavior of high-Mn steels are scarce, and they do not consider the effect of the different C and Mn contents present in these steels. In this work a quantitative model for the determination of the static recrystallization kinetics, valid for a wide range of high-Mn steel compositions, is presented. In order to do so, softening data determined for steels with different Mn (20 to 30%), Al (0 to 1.5%) and C (0.2 to 1%) levels at different deformation conditions (temperature, strain and strain-rate) were considered. Static recrystallization kinetics of the high-Mn steels follow Avrami's law, with n Avrami exponents which are temperature dependent and lower than those determined for low C steels. Modifying the base composition from 20%Mn-1.5%Al to 30%Mn or microalloying addition in solid solution does not affect the softening kinetics of these steels. However, a dependence of the (time for 50% recrystallized fraction) on the carbon content was observed and it was incorporated into an equation for the calculation of this parameter.

801 Influence of initial heat treatment on microhardness evolution in an Al-Mg-Sc alloy processed by high-pressure torsion

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An Al-3% Mg-0.2% Sc alloy was subjected to annealing or solution treatment and further processed by HPT at room temperature. Microhardness measurements were taken along the middle-sections of the discs and they demonstrated that a very substantial hardening is achieved during HPT processing regardless of the initial heat treatment. Hardness values of ~200 Hv were detected in the edge of the samples although the microhardness distribution remained inhomogeneous along the diameters of the discs after 20 turns. In addition, the microhardness of the solution treated Al-Mg-Sc samples continued to increase with equivalent strain even after 30 turns of HPT whereas the hardness at the edges of the annealed discs saturated after 20 turns. These differences in hardness evolution are attributed to the higher Mg content in solid solution for the solution treated samples and its influence in delaying the recovery rate of this aluminium alloy.

802 Laboratory simulations of strip casting for production of dual-phase and transformation-induced plasticity steels

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Production of steels using strip casting technology shortens the processing route and saves resources. However to-date, strip casting is utilised only for production of single phase steels. Using a Gleeble thermo-mechanical simulator, conventional dual-phase (DP) and transformation-induced plasticity (TRIP) steels were processed according to the strip casting route. The microstructures were characterised using optical, scanning and transmission electron microscopy, as well as electron back-scattering diffraction. Mechanical properties were determined using tensile testing machine.

The results have shown that while varying the processing parameters the required volume fractions of phases could be obtained, overall the microstructures remain coarse compared to those of the DP and TRIP steels produced using conventional thermo-mechanical processing routes. This is a result of very coarse prior austenite grains, which are typical for cast strip. With increase of the volume fraction of polygonal ferrite to ~0.75, the microstructure of DP steel also becomes more homogeneous. As a result, the obtained mechanical properties are comparable with those of hot and cold rolled steels only for the steels with high volume fraction of ferrite when the deformation was carried out at high temperature of 1050°C.

One of the main limitations in controlling the microstructure of steels during strip casting is the availability of only one rolling stand with the maximum reduction up to 0.4 strain. Thus, only the effect of deformation temperature could be considered for microstructure-properties improvements. The outcomes of such investigation for DP steel will be presented and discussed.

803 Magneto-structural stability of magnetic shape memory alloys quenched from high-temperature

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Magnetic Shape-memory alloys (MSMA) are a special kind of smart materials because of their unusual properties, such as the shape-memory effect, the possibility of magnetic field induced martensitic transformation or the high variant's mobility that can be used in a large variety of applications like energy harvesting, magnetic refrigeration, magnetoresistive devices, mechanical actuators or high damping devices. All this applications are based on the magnetic and structural properties of the austenite and martensite phases related through a martensitic transformation (MT). The knowledge of the phase's properties and those of the phase transformation itself is essential to outline its potential future applications.

Thermal treatments strongly modify the properties of the alloys and usually samples are quenched from high temperature to avoid decomposition processes. Nevertheless, this is an unstable thermodynamic state and the microstructure (order, defects, phase changes, etc.) evolves in operating conditions, so the stability of the alloy must be well controlled. In particular, the atomic ordering on magnetic SMA has shown to be really crucial to determine

their properties. This diffusion process is mainly controlled by the vacancy dynamics and the control of the vacancy density and its relation to the atomic diffusion must be well established. Large scale defects like dislocations or grain boundaries must also be analyzed to set the austenite-martensite stability. In Fe-Pd ferromagnetic shape memory alloys a reduction in the dislocation density reduces the irreversible transformation temperature and so, increases the stability range of the alloy.

804 Understanding grain size effects in pure Mg polycrystals

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The aim of this work is to present a well-grounded explanation for the transition between twinning to slip dominated flow that takes place in pure Mg polycrystals with grain size refinement, with decreasing strain rate and with increasing temperatures. This is a very controversial topic, and contradictory evidence regarding transition grain sizes, strain rates and temperatures has been reported in the literature. With that purpose, two pure Mg polycrystals with grain sizes of 19 and 5 microns and very similar textures and grain boundary distributions, were prepared by rolling and annealing. They were then tested in compression along the rolling direction in a wide range of temperatures and strain rates. Our results reveal, first, that twinning is replaced by basal slip, which takes place along well-defined deformation bands. Second, the dominance of basal slip is related to the connectivity of favorably oriented grains, which facilitates slip transfer across grain boundaries. Such connectivity is shown to be related to the fraction of boundaries with misorientation smaller than a specific threshold, which is well characterized as a function of the testing conditions, as well as to their local rearrangement. It will be shown how decreasing grain size and strain rate, and increasing temperature all contribute to enhance the connectivity between grains that are well oriented for basal slip. The wide scatter in the reported Hall-Petch constants for pure Mg and Mg alloys is rationalized on the light of these findings.

805 Behavior of tubes from Zr-based alloys under prolonged neutron irradiation

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Texture and substructure changes in cladding tubes from Zr-based alloys under prolonged neutron irradiation were investigated by use of X-ray methods. For this aim in the "hot" laboratory of NIIAR the automated X-ray diffractometer was set and the technique to handle with irradiated samples was elaborated. Studied tubes had worked in the atomic reactor during 6 years. The samples of a reduced size were cut out from different regions of cladding tubes in their upper, middle and lower parts. It was found, that the resistant radiation activity of these parts, proportional to the measured intensity of X-ray radiation, correlates with integral texture Kearns parameters of tube, so that the texture weakens as the experienced dose of neutron

irradiation grows. Texture changes in tubes have predominantly the deformation nature, conditioned by operation of the heat carrier and radiation growth. Aside from those, the mechanism of texture changes in cladding tubes is connected with vacancy loops, which are formed under neutron irradiation, move in the axial stress field of vertical tube and cause the rotation of beta-Zr crystalline lattice. Since the initial substructure of cladding tubes is very inhomogeneous, its changes under neutron irradiation are also inhomogeneous. According to data of X-ray method of Generalized Pole Figures, the influence of neutron irradiation on the substructure of grains is controlled by their condition: the substructure of relatively perfect grains, corresponding by their orientations to texture maxima, whereas the substructure of relatively distorted grains, corresponding by orientations to texture minima, becomes more perfect.

806 Effect of high and low temperature exposure on the mechanical properties of self-hardening Al-based alloy

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Cost-effective manufacturing method for producing modified self-hardening Al-based alloy is proposed for automotive and aircraft industries. Ti as grain refiner has been added, in form of Al-5Ti-1B master alloy, to the AlZn10Si8Mg basic alloy produced by permanent mould casting technique. Experimental results on their mechanical behaviour obtained at different temperatures demonstrate their applicability for structural components development in the aforementioned area. Additionally, investigations on their morphology and on their corrosion resistance confirm that the proposed alloy reveal good stability for long exposure at high temperatures up to 200°C, as well as at sub-zero-temperatures.

807 TEM analysis of creep deformation micromechanisms in the AD730 Ni-based disk superalloy

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For more than 30 years, several studies have been performed to understand and improve the mechanical properties of Ni-based superalloys designed for turbine blades as well as turbine disk application. These alloys experience severe using condition combining tensile test, creep and fatigue at different temperatures. The development and the optimization of these materials have been possible through multiscale approaches combining experiment and modeling. These researches have allowed a thorough understanding of the relation between the macroscopic behavior and the characteristics of the microstructure. But, in the current competitive aeronautic context, efforts have still to be done to allow the development of new superalloys.

This work is also aimed to focus on the characterization of creep deformation micromechanisms in the new AD730TM Ni-based polycrystalline superalloy. More precisely, the microstructure, which consists in multimodal gamma prime precipitates, has been characterized using conventional TEM observations and TEM spectroscopies. Then, post mortem TEM

observations and in situ TEM straining experiments have been performed to identify the pertinent deformation micromechanisms, which control the macroscopic deformation. The interactions between the dislocations and the gamma prime precipitates have been identified and compared to previous results obtained in similar Ni-Based disk superalloys. Else, the effect of heat treatment on the microstructure and the influence of any evolution of the initial microstructure on the creep behavior have been carefully analyzed.

808 Effect of the secondary phase precipitation on the corrosion resistance of different duplex stainless steels

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Duplex Stainless Steels (DSS) are austeno-ferritic stainless steels, widely employed in the chemical, petrochemical and nuclear industry owing to their favourable combination of strength and corrosion resistance properties. However, DSS are affected by phase instability during thermal cycles which causes significant decreases in steels features. In particular the precipitation of secondary phase at the grain boundary can negatively affect the corrosion resistance of these steels. In detail the phase precipitation is caused by thermal cycles, that can vary for different DSS. The aim of the present work is to study the effects of different heat treatments, and so of different percentages of precipitated secondary phases on the corrosion resistance of various DSS grades (SAF2101, SAF2304, SAF2205 and SAF2507). The secondary phase precipitation was evaluated and quantified with scanning electron microscope (SEM) observation and image analysis. The corrosion resistance was studied with potentiodynamic polarization tests and critical pitting temperature (CPT) tests. Anodic polarization tests were performed in acid or neutral environment. CPT test was performed in accordance with ASTM G-150. A remarkable decrease in the corrosion properties was found for high percentages of secondary phases especially in SAF2304 and SAF2205 DSS. Low percentages of secondary phase cause also a slight decrease in the corrosion resistance.

809 Computer system for comprehensive optimization of material processing technologies

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Fast progress in modelling of metal forming processes encourage researchers to look for better technologies, which can be done through optimization of their design. On the other hand, the application of modern models combined with the iterative optimization techniques is difficult for inexperienced engineers and requires high computing costs. Solutions are even more complex when microstructure evolution models are applied and product properties are included in the objective function. Therefore, searching for the efficient optimization strategy for metal forming processes was the first objective of this paper. Earlier Authors research on optimization of metal forming [1,2] were summarised and synthetized in this paper and the efficient computer system was proposed. The second objective was the presentation of the developed by the Authors ManuOpti – user friendly computer system dedicated to automatic optimization of production cycles. Methods and models used in the system are described and capabilities of the

system are demonstrated. Case studies for a chosen metal processing problems are presented, as well. **KEYWORDS:** metal forming, manufacturing cycles, microstructure evolution, optimization, ManuOpti computer system **REFERENCES** [1] J. Kusiak, D. Szeliga, Ł. Sztangret: Optimization techniques in metal forming, in: Microstructural evolution in metal forming processes: modelling and applications, eds, J. Lin, A. Balint, M. Pietrzyk, Woodhead Publ., 2012, 35-66. [2] Ł. Rauch, M. Skiba, J. Kusiak: Computer system dedicated to optimization of production processes and cycles in metal forming industry, Computer Methods in Materials Science, 14, 2014, 3-12.

810 Modelling of grain-boundary mobility and nucleation rate in Ni–Nb alloys during discontinuous dynamic recrystallization

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During hot working and thermomechanical treatments of superalloys, dynamic recrystallization controls the evolution of microstructures, and therefore the most important material properties for aeronautical applications.

A mesoscale model was developed for discontinuous dynamic recrystallization. Each grain is considered in turn as an inclusion, embedded in a homogeneous equivalent matrix, the properties of which are obtained by averaging over all the grains. The model includes: (i) a grain-boundary migration equation (ii) coupled with a single-internal-variable (dislocation density) constitutive model for strain hardening and dynamic recovery, and (iii) a nucleation equation governing the total number of grains by the nucleation of new grains. All the system variables tend to asymptotic values at large strains, in agreement with the experimentally observed steady-state regime. With some assumptions, both steady-state stresses and grain-sizes are derived in closed forms, allowing immediate identification of the mobility of grain boundaries and the rate of nucleation. An application to Ni–Nb-pure-binary model alloys is presented. More specifically, from experimental steady-state stresses and grain sizes, variations of the grain boundary mobility and the nucleation rate with niobium content are addressed in order to quantify the solute-drag effect of niobium in nickel. An advanced model is presented in a companion paper in the conference by Smagghe et al., in which some topological features are introduced to predict more realistic grain-size distributions allowing coupling with metadynamic models.

811 Trace element-added Al-Mg-Si alloys

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Al-Mg-Si alloys are the most frequently used group of age-hardenable aluminum alloys. The adverse effect of natural aging on the artificial aging, which increases the necessary duration of the heat treatment and reduces the achievable strength of the material, has not been fully resolved since it was discovered in 1939. Moreover, natural aging has negative implications for the success of Al-Mg-Si alloys for automotive applications, because it affects the formability of the material. We discuss the concept to reduce the negative effects of natural aging via the

addition of the trace elements Sn and In. At room temperature, these trace elements trap quenched-in vacancies and natural aging kinetic is retarded, while at higher temperatures these vacancies are released and support diffusion for fast nucleation of the β'' -phase. We evaluate the performance of trace element-added Al-Mg-Si alloys at various natural and artificial aging temperatures. Moreover, the effects of the overall alloy composition and its implications for maximum suppression of natural aging and strengthening potential upon artificial aging are shown. The results are discussed in terms of clustering theories and atomistic diffusion phenomena and can be used to improve the industrial alloy design of Al-Mg-Si alloys.

812 Towards frictionless surface

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Frictionless surface is a dream of generations of mechanical engineers. Self-lubricant solids are promising candidates for such ambitious challenge when compared to traditional oil-based lubricants. Graphite/graphene and transition metal dichalcogenides (TMD) exhibit the lowest friction on a nanoscale; the latter is frictionless even at macroscopic contacts under ultra-high vacuum conditions. In general, TMD-based coatings are known to form tribolayer, which significantly decreases friction and wear. We investigate key properties of such tribolayer and its impact on tribological properties. We have found that tribolayer is well ordered TMD with basal planes parallel to sliding direction; it is formed almost regardless on atmosphere (vacuum, dry/humid air) within wide range of contact conditions (sliding speed, contact pressure). We show some successful microstructural designs of TMD-based self-adaptive coatings doped with metals, carbon or nitrogen.

Thanks to extremely low thickness of such tribolayer, typically few nanometers, atomistic simulations are natural tool to investigate friction at sliding interface. We presents recent advances in atomistic simulations and in situ nanoscale observations of sliding process related to 2D, thin film and bulk TMDs. We briefly review theoretical aspects of TMD sliding at nanoscale and shortcomings of actual nanoscale models, particularly ab initio and molecular dynamics simulations. Then we compare frictional characteristics of 2D TMD layers with those of bulk crystal and relate them to macroscopic. We demonstrate that friction is scalable, i.e. similar values of friction coefficient are obtained by nanoscale (FFM) and macroscale (pin-on-disc) methods provide contact pressure is identical.

813 Austenite formation along dislocations in medium manganese steels

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In a binary martensitic Fe-9wt%Mn alloy with very low carbon content (75 wt ppm) we observe a serrated appearance of the stress-strain curve at 450 °C and discontinuous yielding after tempering at 450°C. These phenomena have so far mainly been observed in the Fe-C system, where they are referred to as dynamic and static strain ageing, respectively. Correlative TEM/atom probe tomography experiments give direct evidence for pronounced equilibrium segregation of Mn (up to a factor of 3) and the absence of C at dislocations. The tubular-like

Mn enriched zone has the chemical composition of austenite when in thermodynamic equilibrium with the present ferrite phase (partitioning). In conjunction with the thermodynamically calculated radius of a subcritical metastable particle of the second phase, we hence suggest that some of the dislocation segments might have undergone segregation-induced phase transformation to austenite.

814 Formation and interaction of point defects in group IVb transition metal carbides and nitrides

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Group IVb carbides and nitrides are technologically important materials due to extraordinary physical properties: extreme hardness, high wear resistance, high melting points. A common feature of group IVb carbides and nitrides is a sizeable off-stoichiometry--they can accommodate up to 50% of vacancies on the non-metal sublattice. On the one hand, such a severe off-stoichiometry strongly affects the properties of these materials. On the other hand, it makes their investigation a complicated task. In this work we investigate point defects in group IVb transition metal carbide and nitrides using density functional calculations. We focus on a description of a complex vacancy behavior of the substoichiometric carbides and nitrides and find a strong tendency toward vacancy clustering in the carbides. Our results demonstrate that a special type of a stable point defect, a metal vacancy “dressed” in a shell of six carbon vacancies can be a dominant type of metal-vacancy-containing defect in the carbon-deficient sub-stoichiometric carbides, whereas the simplest point defects appear to be dominant in the nitrogen-deficient sub-stoichiometric nitrides. We also show that such clusters are strongly bound in carbides and that temperature has a relatively small effect on the overall defect stability of group IVb transition metal carbides and nitrides.

815 Evolution of ni structure under ecap and dcap and further annealing

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The main goal of the present study is to compare the evolution of structure of commercially pure Ni (99.6 %) under equal-channel angular pressing (ECAP) and dynamic channel-angular pressing (DCAP) and to investigate the thermal stability of the structures obtained. The DCAP is similar to ECAP, but instead of press machinery the energy of powder combustion gasses is used ensuring plastic deformation of massive specimens at high speeds. Under the ECAP a heterogeneous microstructure is formed with pronounced deformation bands. After the four passes of deformation by the Bc4 route the widths of the deformation bands is about 1-2 mm, and they contain fine grains the size of 150-200 nm, separated by coarser anisotropic grains. Under the DCAP more uniform structure is formed, than under the ECAP, consisting of slightly curved wide bands with high-angle misorientation, inside of which there are fine equiaxed areas with low-angle misorientations. After the three passes of DCAP the average size of crystallites is about 250 nm. The microhardness increases faster under the DCAP and gets by a factor of

2.5 higher than in the initial state after 3 passes of DCAP or 8 passes of ECAP. The thermal stability of the structures obtained is similar. The microhardness decreases beginning from 300°C annealing, and drops dramatically at 400°C due to recrystallization and grain growth. The work was supported by the State Program “Spin”, the Program of Fundamental Research of UB RAS (project 15-9-2-44) and by RFBR (project 15-03-03103).

816 Tantalum nitride structure selection: A new route to control diamond nucleation and growth?

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Because of their unique mechanical, chemical and electrical properties, transition metal nitride thin films have attracted considerable attention in recent years. In particular, tantalum nitride (TaN) thin films are very promising materials as suitable diffusion barriers for microelectronic applications and diamond deposition due to their high thermal stability, hardness, chemical inertness and conductivity. In the case of diamond deposition, TaN thin films have been used as buffer layers and diffusion barriers against cobalt diffusion during diamond deposition on sintered WC-Co cutting tools. Ideally, they should also play a positive role on carbon diffusion phenomena during the process in order to enhance diamond nucleation and growth.

According to the phase diagram, TaN exhibits two crystallographic structures: hexagonal and face-centered-cubic. An accurate control of the cathodic sputtering conditions allows us to isolate the both phases. Their efficiency as a cobalt diffusion barrier has been proved. However, their influence on diamond coating formation remains unestablished and phenomena occurring during diamond nucleation and growth are not well understood. This is the purpose of this study. We have demonstrated that TaN carburization mechanisms occurred during the CVD diamond deposition process and that they affect diamond nucleation and growth drastically. Those carbon diffusion phenomena and their consequences on diamond coating formation are discussed based on the TaN selected structure. The understanding of the specific responses of each phase during the CVD process opens up new horizons for diamond coating performances.

817 Coupling of computational thermodynamics with kinetic models for predictive simulations of materials properties

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Solid-state phase transformations, including precipitation, govern a variety of materials properties of alloys such as strength and microstructural stability. Modern applications comprise multi-component multi-phase systems, which make systematic process and product optimization by experimental investigation difficult, time-consuming and costly. On the other hand, combining computational equilibrium thermodynamics of complex alloy systems with kinetic models for diffusion mobilities, precipitate growth and coarsening comprises a huge potential for the prediction of temperature- and time-dependent microstructural stability and related mechanical properties. We present successful examples of CALPHAD thermodynamics-based precipitation simulations for three large alloy groups: hardenable Al-

alloy, single-crystal Ni-base superalloy, and austenitic stainless steel. Underlying physical models for special features such as metastable precipitate, energies of diffuse interfaces between coherent precipitates and matrix, evolution of excess vacancies during quenching and continuous aging, and precipitation of incoherent particles at grain boundaries will be discussed.

818 Study of mechanical properties of nanocrystalline Ti-35Nb alloy processed by severe plastic deformation

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Titanium and its alloys have been widely used as biomedical materials to replace dysfunctional hard tissue in human body due to their high strength, light weight, relatively low elastic modulus, and excellent biocompatibility and corrosion resistance. A Ti – 36.5 Nb (wt%) alloy without undesirable elements, aluminum and vanadium like with the standard biomedical alloy Ti6Al4, was used for study. Purpose of this investigation was to examine the mechanical properties of this alloy with potential for biomedical use. The investigated alloy presents a low Young's modulus, an important condition for preventing stress shielding phenomenon, which is one of the main factors that causes implant failure. However, while the Young's modulus is low and optimal for these purposes, the strength should be higher. By processing the material by high pressure torsion (HPT), i.e. applying both hydrostatic pressure and shear deformation, under pressures of 4.0 GPa and 8 GPa until torsional shear strains of about 150, in order to obtain a submicro- or even nanocrystalline structure. The mechanical characterization was carried out by Vickers hardness measurements and nanoindentation. Keywords: Ti - Nb alloy; high pressure torsion; microhardness.

819 Continuous versus conventional heat treatment of hardenable steels

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Short-time continuous annealing is an interesting and economic alternative to conventional heat treatments with comparably long isothermal holding times. Fast heating rates can, for example, easily be achieved by using inductive heating. Due to rapid heating to comparably higher target temperatures, isothermal holding times during e.g. hardening and tempering of steels can be significantly reduced which represents a great cost reduction potential. Furthermore, continuously heat-treated steels are known to exhibit less deviation in mechanical properties and less decarburization. Whereas the microstructural evolution during isothermal hardening and tempering of steels is well known, the differences in homogenisation and phase transformation kinetics during short-time continuous heat treatments still have to be established in order to process parts with similar or even better mechanical properties. The current study will showcase the microstructural evolution during continuous versus conventional hardening and tempering of a 42CrMo4 steel and an HS6-5-2 tool steel. Each step of the heat treatment cycle has been studied comprehensively using state-of-the-art microstructural characterisation techniques such as scanning and transmission electron microscopy, electron back-scatter

diffraction, micro probe, and atom probe tomography. The corresponding microstructure-property relationships of the final parts achieved by both heat treatment approaches will be discussed.

820 Design and characterization of new titanium alloys combining high strength, high strain hardening and improved ductility

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Titanium alloys have been continuously attractive for industrial applications, however, major drawbacks of both low ductility (typically < 20%) and a lack of strain hardening, are still limiting their potential in advanced applications. This work aims at designing Ti-Cr and Ti-Mo based alloys with a new and promising combination of mechanical properties. The mechanical properties display an extremely high ductility (approaching 45% of plastic deformation) accompanied by both a high strength and a very high work hardening rate. Extensive microstructural characterization (TEM, EBSD) consistently revealed a complex sequence of deformation mechanisms consisting in activation of both intense {112}<111> and {332}<113> mechanical twinning accompanied by both ω and α'' phases stress induced precipitation. In this talk, design strategy, occurrence and chronology of deformation mechanisms will be highlighted in order to understand the improvement of work hardening. Future directions towards the development of a new family of metallic materials will be discussed.

821 Photolithography of 3D scaffolds for artificial tissue

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The present study is focussed on the design and fabrication of novel functional 3D-hydrogel scaffolds for regenerative medicine. In order to critically analyse the effect of the microarchitecture of 3D scaffolds for driving the cellular fate and diffusion of progenitor stem cells we have fabricate a number of scaffolds with different geometry, stiffness and composition. The physical characteristics of the scaffold determine indeed, as well the biochemical factors, the fate of the cells. We use an innovative composite material consisting of hydrogel with different molecular weight and with suitable accordion-like honeycomb-structured in order to tailor stiffness and elasticity conferred to the final structure. These novel 3D bioinspired scaffolds were obtained by both single- and two-photon polymerization (2PP) processing. In particular, 2PP scaffolds represent a great advantage with respect to previous achievements based on traditional methods. 3D-structures were fabricated with lateral resolution of microns allowing an advanced control of pore microarchitecture of defined tensile strength and the inclusion of microbubbles with various functionalities. The mechanical, morphological, biochemical and functional characteristics are discussed. Moreover, the effects of the structured hydrogel scaffolds on the proliferation and differentiation of adult stem cells is critically analysed in view of the fabrication of portion of contractile cardiac muscle to be

obtained in vitro.

822 Microstructure formation in a quaternary Ti-Al-Nb-Ta alloy by massive transformation

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TiAl alloys are nowadays used as material for turbine blades in civil aero engines and partly replace Ni-base superalloys for this application. Nevertheless, their insufficient ductility especially at low temperature hampers their wider application. The so called convoluted microstructure is known to provide a favorable combination of strength and low temperature ductility in TiAl alloys. Unfortunately, to adjust this microstructure it is necessary to rapidly cool the material from higher temperatures to make the α phase undergo a massive transformation into γ phase. In this massively transformed γ phase α_2 lamellae in a convoluted configuration can be generated subsequently by heat treatment. The necessary cooling rates for the massive transformation are not feasible in industrial processes for the TiAl alloy compositions actually in use. Nevertheless, by alloying TiAl materials with heavy slowly diffusing elements it is possible to decelerate transformation kinetics to such an extent, that the massive transformation is accessible at reasonable cooling rates. In this study a Ti-45Al-4Nb-4Ta (at.%) alloy was investigated using in situ high-energy synchrotron X-ray diffraction. In situ experiments enable continuous monitoring of the phase evolution during heating and cooling. In particular the transformation start temperature, the critical cooling rate, and the undercooling required for the massive transformation, as well as for subsequent α_2 precipitation during annealing have been investigated. The microstructures developed after the massive transformation and after subsequent annealing have been characterised by EBSD.

823 Microstructural characterization and mechanical properties of stainless steel inlay welded dissimilar materials

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In several locations of the pressurized water reactors (PWRs), dissimilar metal welds using Inconel welding wires are used to join stainless steel pipes. Because of the existence of different materials and chemistry variation within welds, the mechanical properties, such as tensile and fracture properties, are expected to show spatial variation. For design and integrity assessment of the dissimilar welds, these variations should be evaluated. This present work was carried out to characterize the microstructure and mechanical properties of austenitic stainless steel AISI 316L to dissimilar welding of Inconel 82 and Inconel 52 fillers which were treated by ultrasonic nanocrystalline surface modification (UNSM) technique. The microstructure and hardness of the specimens were characterized by electron back-scatter diffraction (EBSD) and micro-hardness tester. The increase in hardness and refinement in grain size after UNSM treatment were found for the AISI316L/52 and AISI316L/82.

824 Fatigue deformation and crack growth behavior of Fe-Mn-C-(Al) TWIP steels

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Al, as an important alloying element, is added for adjusting the stacking fault energy of high Mn TWIP steels and its effects on the monotonic deformation behavior of these steels have been extensively investigated. However, how Al affects the fatigue properties of such steels remains unclear. In this work, the cyclic deformation and fatigue crack growth behaviors in two Fe-Mn-C TWIP steels with different Al additions (0Al and 3Al, wt. %) were investigated, by means of low-cycle fatigue tests and fatigue crack growth tests. The evolved microstructures and crack growth paths of fatigued samples were examined via microscopic observations and synchrotron x-ray computed tomography.

The results show a strong dependence of the responses of cyclic stress, fatigue crack threshold, and crack growth rate on Al addition. As compared with the 0Al steel, the 3Al steel, with a higher monotonic yield strength, demonstrates smaller initial cyclic hardening followed by larger cyclic softening at higher cyclic numbers, thus showing a decreased cyclic yield strength. Furthermore, the 0Al steel shows higher fatigue crack growth resistance in both the near threshold and Paris regimes than the 3Al steel. The differences in the cyclic deformation behavior and crack growth resistance observed in the two steels with different amounts of Al are discussed, in terms of roughness- and plasticity-induced crack closure, the planarity of slip and deformation twinning, based on microscopic observations of cyclically-deformed microstructures.

825 Evolution of *CSL boundaries of a Ni₃Al-base alloy in long term annealing treatments

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The effect of long term isothermal heat treatments on a wrought Ni₃Al-base alloy was investigated. The deformed grains of the alloy evolved to equiaxed ones due to the long thermal holds. Bimodal grains were observed in the recrystallized microstructure, which showed continuous growth along with the increasing of thermal duration. Characterization of electron back scattered diffraction (EBSD) revealed that annealing twins constituted the major part of coincident site lattice (CSL) boundaries. As thermal time extended, the volume fraction of the annealing twins decreased, however, less other SCSL boundaries were yielded. The characteristic evolution behavior of SCSL boundaries was discussed.

826 Interface of ultrafine grained Al/Mg multilayered disks prepared by high pressure torsion

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Al alloys and Mg alloys are the two lightest metallic structural materials. Apart from the low density and the corresponding high specific strength and specific modulus, Al alloys possess a high thermal conductivity, electrical conductivity and corrosion resistance; whilst pure Mg is one third lighter than pure Al and has good damping capacity. Al/Mg multilayered materials, when processed to obtain sound interfaces, may combine the advantages of both Al and Mg and find many applications in industry. In this paper, commercially pure Al and Mg were employed to fabricate ultrafine grained Al/Mg multilayered composites by high pressure torsion (HPT) at room temperature. The bonding quality was examined by OM, SEM+EBSD and FIB+TEM. Vacancy concentration was characterized by positron annihilation. Hardness distribution in the cross section of the interface was characterized by micro-Vickers hardness and Nano-indentation. A thin layer of $\text{Mg}_{17}\text{Al}_{12}$ was observed at the interface of Mg/Al disks in contrast to a very thick layer of Al_3Mg_2 and a relatively thin layer of $\text{Mg}_{17}\text{Al}_{12}$ at the interface of diffusion-bonded Mg/Al at high temperatures, e.g. 400 °C. This is attributed to the lower molar Gibbs free energy of Al_3Mg_2 than that of $\text{Mg}_{17}\text{Al}_{12}$ at room temperature and a very large number of vacancies and even vacancy clusters formed in Mg/Al disks during HPT at room temperature.

827 A statistical methodology to reconstruct nucleation pathways in the Fe-Cu system

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In precipitation strengthened ferritic alloys, the Fe-Cu binary system is a well-studied model system. Still, many unsettled questions remain about the early stages of bcc Cu precipitation, most of these with respect to the shape and composition of the critical and post-critical Cu precipitates. Since the critical nucleation states are hard to investigate by experimental methods, we propose a computational strategy to reconstruct precipitation pathways and identify the nucleation states making use of Monte Carlo simulations combined with Rare Event Sampling methods. The precipitation process is reproduced by Monte Carlo simulations with an energy description based on the Local Chemical Environment approach, applying efficient pair potentials, which are dependent on the chemical environment and Forward Flux Sampling technique. This method provides profound insight into the shape and the composition of the early precipitates and also the critical cluster size in dependency of the temperature and supersaturation.

828 Modelling of phase separation under electropulsing processing

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A model has been developed to study the materials separation under electric current processing. The phases with different electrical properties are reacted differently to the applied electric field. A force to drive the inclusion to move has been derived based on thermodynamic calculation of the multiphase system carrying electric current. Numerical calculation demonstrates an effect of electropulsing which is stronger than that of gravity.

The experiments based on the modelling and calculation have been introduced. The method has been applied successfully in the fabrication of super clean steel. References: R.S. Qin and A. Bhowmik, Computational thermodynamics in electric current metallurgy, Mater. Sci. Technol., 31 (2015) pp. 1560-1563. X.F. Zhang and R. S. Qin, Controlled motion of electrically neutral microparticles by pulsed direct current, Sci. Rep., 5 (2015) pp.10162. X.F. Zhang and R.S. Qin, Electric current-driven migration of electrically neutral particles in liquids, Appl. Phys. Lett., 104 (2014) pp. 114106.

829 Solidification behavior of aluminum-copper based alloy during controlled diffusion solidification

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Controlled diffusion solidification (CDS) is a novel and simple process that enables the formation of non-dendritic microstructure of primary Al phase through mixing two liquid alloys of different composition and temperature together. A quaternary alloy (Al-5.0Cu-0.35Mn-0.25Ti, wt.%), having a similar chemical component with ZL205A, was fabricated using controlled diffusion solidification (CDS) method with different mixing temperature. The mixing temperature of two liquids mostly affects the cast structure especially the primary Al phase. Results show that CDS can reduce the element segregation degree inside the grains. Microstructure evolution during solidification initiates from a primary nucleus firstly and then changed to a non-dendritic grain structure. The thermal analysis confirms the thermodynamic conditions for the formation of non-dendritic grain structure evolution.

830 Anti-bacterial nanocomposites by silver nano-coating fragmentation

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Silver nanocomposites are of great interest for several fields, ranging from packaging to biomedical applications, because of their known antibacterial properties. However, their use is strongly limited by technological issues: synthesis methods of silver nanoparticles are still difficult to control in terms of size, shape and aggregation. Moreover separation of nanoparticle aggregates should occur during part manufacturing by mixing with the polymer base but this

process is also troublesome. In the present study, a new process to fabricate silver nanocomposites with antibacterial properties is presented. Silver nano-films have been deposited by radio frequency (RF) sputtering on polypropylene (PP) substrates. Consequently the PP coated substrates were cut and inserted in a small scale polymer mixer to produce the nanocomposite in a single step, without the need of producing nanoparticles. In fact, nanoparticles originate by the fragmentation of the nano-coating. Microscopic observations of nanocomposites revealed silver nanoclusters of different sizes. Their antibacterial activity has been verified in accordance to ISO 22196. The antibacterial activity of the nanocomposite was detected for the *Escherichia coli* and *Streptococcus aureus* bacteria.

831 Electrodeposited molybdenum oxide films and patterned submicrometer motifs: structure and mechanical properties

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Transition metal oxides in thin-film form have received much attention owing to their promising applications in electrochromics, energy storage, sensing and non-volatile resistive random access memory devices, among others [1]. Due to their unusual chemistry produced by multiple Mo valence states (IV, V and VI) and a rich variety of crystallographic structures, molybdenum oxides are suitable candidates in many of these applications. In this work we analyze the influence of cetyltrimethylammonium bromide (CTAB) surfactant on the properties of molybdenum oxide films (1.5 μm -thick) electrodeposited onto indium tin oxide (ITO)/glass substrates. An electrolyte containing 0.2 M Na_2MoO_4 adjusted to pH = 1, 6 and 9 was used for the growth of these films. Key parameters such as film adhesion, morphology, degree of porosity, molybdenum speciation, and crystallographic structures were correlated with the mechanical properties of the films, namely hardness (H) and Young's modulus (E). Film adhesion improved, cracking was minimized and crystallinity increased upon adding 0.01 M CTAB to the electrolyte. As a consequence both H and E, measured by nanoindentation, were enhanced. Following a similar synthetic pathway, ordered arrays of molybdenum oxide submicrometer motifs, including pillars and stripes, were grown onto e-beam lithographed Au/Ti/Si substrates [2]. [1] N.A. Chernova, M. Roppolo, A.C. Dillon, M.S. Whittingham, Layered vanadium and molybdenum oxides: batteries and electrochromics, *J. Mater. Chem.* 19 (2009) 2526–2552. [2] A. Quintana, A. Varea, M. Guerrero, S. Suriñach, M.D. Baró, J. Sort, E. Pellicer, Structurally and mechanically tunable molybdenum oxide films and patterned submicrometer structures by electrodeposition, *Electrochim. Acta* 173 (2015) 705–714.

832 Computational and experimental analysis of hot ductility during continuous casting of micro-alloyed steel

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The vast majority of today's steel is produced on basis of continuous casting technology. Thermal gradients and mechanical loading due to roll contact and bulging expose the strand to severe thermal stresses. The most common continuous casting facilities utilize vertical casting, bending and straightening operations. Particularly, the latter process introduces additional mechanical loads on the strand. For steel, thermo-mechanical stresses are especially hazardous in the region of reduced ductility, typically between 1200°C and 600°C. In this work, we investigate the hot ductility of a micro-alloyed steel both experimentally on a Gleeble 1500 thermo-mechanical testing machine as well as computationally by means of the thermokinetic software MatCalc. One series of experiments is performed for linear cooling from 1320°C to testing temperature, a second series includes superimposed temperature troughs, which mimic the strand cooling profile in a more realistic way by taking into account the contacts between strand and rolls. Comparison between simulated and experimentally observed ductility are in good agreement. The loss of ductility can clearly be associated with the formation of deformation-induced micro-alloying precipitates.

833 Adsorption of an albumin subdomain on different crystallographic surfaces of anatase TiO₂: a molecular dynamics study

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The so called biomaterials of third-generation are materials designed to stimulate specific responses at molecular level. It is increasingly important to describe at atomistic level both the biomaterial surface chemistry, the adsorbed proteins and the interaction kinetics. An important and most abundant blood protein is albumin, and a largely used biomaterial is anatase TiO₂. In previous work, TiO₂ polymorphs (rutile, anatase, brookite) were studied. In this work, using the same methodology based on Molecular Mechanics (MM) and Molecular Dynamics (MD) methods, the adsorption of an albumin subdomain on different crystallographic faces of anatase is reported, considering the most stable (1 0 1) face, the (0 0 1) face exposing hydroxyl groups, the (0 0 -1) face exposing Ti atoms and the (1 0 0) face exposing oxygen and titanium atoms in a peculiar surface order. The results show that in the initial adsorption stage the albumin subdomain shows an interaction energy E_{int} comprised between 40 and 46 kJ/mol for the (1 0 1), (0 0 1) and (0 0 -1) faces. On the contrary, the E_{int} obtained for the hydroxylated (0 0 1) face amounts to 28 kJ/mol. After the MD simulation runs, in general some surface spreading of the modeled subdomain of this soft protein with parallel ordering of distant parts of the backbone was found. However, when the surface contains hydroxyl groups the overall structure remained globular. These results are in essential agreement with those obtained considering the adsorption on graphite, carbon SWNTs and poly (vinylalcohol) surfaces.

834 Phase field modeling of ordered kappa-carbide precipitate for various isothermal holding temperature

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The phase-field method is used to develop a new technique to simulate kappa- carbide precipitates in multicomponent low density steels. This technique generates the Gibbs free energy for both the disordered FCC and BCC phases and odered kappa-carbide from the single function formalism of both phases in the CALPHAD method without using conventional CALPHAD software. Elastic strain energy is also taken into account. The microstructure evolution for a quaternary Fe-Mn-al-C low density steel was simulated with three dimensional phase field modelling. The sequential evolution of the Mn concentration during the Growth and the interaction of precipitates were investigated. Six simulations were run for the temperature range within which kappa-carbide forms, namely 500 °C, 550 °C, 600 °C, 650 °C, 700 °C, 750 °C. The effects of isothermal holding temperature on the morphology and composition of precipitates were studied.

835 Joining of dual phase steel DP 600 – benefits of weld bonding

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Because of stringent emission and safety norms, it has become imperative for the automotive manufacturers to use advanced high-strength steels in passenger cars. Joining of these advanced high-strength steels, however, presents some special challenges. In the current work, joining of 1 mm thick sheets of dual phase steel DP 600 was investigated using resistance spot welding, adhesive bonding, and weld bonding - a hybrid technique that combines resistance spot welding and adhesive bonding. A commercially available one-part epoxy adhesive (heat curing type) was used for adhesive bonding. Lap joints produced using three these processes were comparatively evaluated using lap-shear, cross-tension and fatigue tests. The failure modes were analyzed in detail. The results show that weld bonding is a particularly attractive process for joining of dual phase steels. Keywords: Dual phase steel; Weld bonding; Resistance spot welding; Adhesive bonding

836 Softening kinetics of plain carbon steels containing dilute Nb additions

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Niobium (Nb) microalloying element addition to steel have been instrumental to the successful development of new steel flat products with enhanced property combinations i.e. pipeline steels

and automotive sheets. Significant progress has been made in the recent years in gaining a quantitative understanding of the complex interactions of solute and precipitates of Nb with recovery and recrystallisation which is basis of thermo-mechanical processing (TMCP) of these steels. However, the research and development on the use of Nb microalloying additions in carbon steel long products and its TMCP has been limited. There is anecdotal evidence within literature on effect of dilute additions of Nb to such steels resulting in improved mechanical properties and reduce manufacturing costs. The present study focuses on the effect of dilute additions of Nb (up to 0.020 %) to plain carbon steels of 0.20 and 0.80 wt % C respectively. The aim is to better quantify effect of prestrain and deformation temperature on the recrystallisation and precipitation behaviour in these steels. The extent of recrystallisation is characterised by a softening parameter calculated from a series of interrupted plain strain compression tests. The T_{5pct} and T_{95pct} , making the beginning and end of recrystallisation, respectively, are determined as a function of strain. The microstructural investigations are carried out using optical microscope and TEM to quantify the effect of precipitation on recrystallisation behaviour. In summary, the present study will succeed in creating a complete composition-temperature-strain processing window of the investigated steels for structural applications based on envisage dilute Nb concept.

837 Effect of nature of chemical linker on the formation of a zeolitic layer on zirconia substrates

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Zeolites possess a high stability, high specific surface area and pores tridimensional system that make them useful to formation of inorganic membranes. During membranes synthesis different parameters should be considered such as nature substrate and the method used in order to obtain a membrane according to its application field. In the present work the formation of a zeolitic layer on the functionalized surface of zirconia substrates was studied. Zirconia disks of ten millimeters of diameter were prepared. They were submitted a chemical functionalization with three different chemical linkers: polyethylenimine (PEI), polydialildimethylamine chloride (PDDA) and 3-aminopropyltriethoxysilane (gamma-APS). Subsequently the substrates were submitted to a seeding process, where their surface was grafted with zeolitic crystals corresponding to W zeolite. In order to promote the formation of zeolitic layer the substrates were submitted a hydrothermal treatment with a batch composition similar to that used in the W zeolite synthesis, at 150°C for 48 h. The crystallization products were characterized by XRD and SEM techniques. The results indicated that the chemical linker enhances the formation of a homogeneous zeolitic layer on the substrate and besides acts as structural directing agent allowing to crystallization of a different zeolitic phase to that used in the seeding process, the merlinoite. The morphology, crystalline phase and thickness of zeolitic layer formed on the surface of the substrate depend of the nature of chemical linker used and its interaction with the substrate.

838 Biocompatible ceramic-biopolymer coatings obtained by electrophoretic deposition on electron beam structured titanium alloy surfaces

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An area of major interest in biomedical engineering is currently the development of novel superior materials for medical implants. Research efforts are being focused on the investigation of surface modification methods for metallic prostheses due to the fundamental bioinert character of these materials and the possible ion release from their surfaces, which could potentially induce the interfacial loosening of devices after implantation. Electron beam (EB) structuring is a novel technique to control the surface topography (and hardness) in metals. Electrophoretic deposition (EPD) offers the feasibility to deposit at room temperature a variety of materials on conductive substrates from colloidal suspensions under electric fields. In this work single layers of chitosan composite coatings containing titania nanoparticles (n-TiO₂) were deposit by EPD on electron beam (EB) structured Ti6Al4V titanium alloy. Surface structures were designed following different criteria in order to develop specific topography on the Ti6Al4V substrate. n-TiO₂ particles were used as a model particle (can be substituted with gold or silver nanoparticles) in order to demonstrate the versatility of the proposed technique for achieving homogenous chitosan based coatings on structured surfaces. A linear relation between EPD time and deposition yield on different patterned Ti6Al4V surfaces was determined under constant voltage conditions, obtaining homogeneous EPD coatings which replicate the 3D structure (pattern) of the surface. A combination of both techniques can be considered as a promising surface modification approach for metallic implants, which should lead to improved interaction between the implant surface and the biological environment for orthopaedic applications.

839 Elaboration of nanostructured coatings by pulsed plasma spraying of liquid feedstock

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Growing needs in energy demand from industrialized and emerging nations compel many researches to improve the efficiency of energy management by focusing on the development of renewable energy sources and by tackling ecological concerns. For example, fuel cells, thermal barrier coatings in gas turbine industry or photo-catalytic coatings require advanced elaboration processes capable to manufacture nanostructured ceramic coatings. These materials must have specific service properties, like finely structured architectures and graded properties (porosity/chemical composition). Such refractory materials are deposited by cost-effective plasma spraying techniques possessing the advantage to treat materials with high rates (>kg/h) in high enthalpy medium (>10 MJ/kg). Ceramic nanostructured coatings can be now achieved whether nanopowders dispersed in a liquid are injected into arc plasma jet. Controlling electric arc instabilities confined in non-transferred arc plasma torch is therefore a key issue to get reproducible coating properties. Consequently, self-sustained pulsed arc plasma jet is under

study associated with a synchronous injection of droplets of solution precursor or containing nanopowders (TiO₂). By adjusting the injection timing relatively to time-dependent enthalpy variations, heat and momentum transfers from plasma to materials are expected to be efficiently controlled. The electrical features of such a pulsed arc are presented and the enthalpy modulation is studied by means of optical emission spectroscopy. The plasma treatment of droplets is also shown to be dependent on the injection timing. Samples of in-flight nanopowders and as-sprayed coatings are also characterized.

840 Fatigue in nanocrystalline, bimodal and ultrafine-grained nickel in respect of thermal and mechanical stability

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Motivated by the high strengthening effect of grain refinement, bulk nanocrystalline materials become more and more important in current studies and applications in material science. One main research focus lies on the stability of these structures regarding the undesired and distinct propensity of grain growth at slightly elevated temperatures or mechanical stresses. The problem of grain growth is the loss of the good mechanical properties. The reason for that is found in the reduction of surface energy which is especially high in nanocrystalline materials due to the big amount of grain boundaries. In the case of nanocrystalline, electrodeposited nickel, which is stabilized by the organic additive saccharin, grain growth occurs at 200°C, whereby some grains grow preferred (abnormal grain growth) and form a bimodal microstructure. Furthermore mechanical induced grain growth occurs in the stress field of a crack. The influence of this behavior in respect of fatigue and crack initiation and crack growth was investigated by crack propagation curves and SEM. The nanocrystalline samples showed thereby better crack propagation behavior than the bimodal ones. The underlying mechanism of the grain refinement and stabilization by additives like saccharin is known as solute-drag-effect which is based on the drag of impurity atoms on grain boundaries. A big disadvantage of this effect is the embrittlement of grain boundaries by segregation which seems to be the case after a heat treatment. Hence an alternative method is presented for production of a nanocomposite with higher stability against grain growth and minor grain boundary embrittlement.

841 Effect of prior austenite grain size on bainite formation: Faster or slower kinetics?

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Bainite microstructure forms the crux of several modern steels. With its ever growing significance in the field of structural steels, an in-depth understanding of the bainite kinetics is extremely important in order to select the right alloy and heat treatment during production. Furthermore, such an understanding would provide valuable insight into complex mechanism of bainite formation. One of the factors affecting the bainite kinetics is the prior austenite grain

size (PAGS). Although the effects of PAGS on the rate of bainite formation have been reported in the literature, the results are contradictory in nature. Adopting the displacive theory of bainite transformation, the rate of bainite formation is proportional to the rate of bainite nucleation, where the latter depends on the density of potential nucleation sites and the activation energy of bainite nucleation. In order to understand the contradictory results on the influence of PAGS, its effect on both the density of nucleation sites and the activation energy of bainite nucleation should be studied. In this work, experimental results and kinetic models are used to understand the effect of PAGS on bainite kinetics. Furthermore, the effect of PAGS on the density of nucleation sites and the activation energy for bainite nucleation are analysed. For the first time, a quantitative into the effect of PAGS and its apparent contradictory nature is reported.

842 Characterization of the oxygen pick-up behaviour of a nickel-based super alloy powder under different sintering atmosphere conditions using a combined TG-GC-MS technique

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The production of superalloy components through the use of powder metallurgy (PM) has gained increasing interest due the methods net shape capability. The production of net shape parts can reduce the buy-to-fly ratio, thus reducing material waste and production costs. Superalloys often contain alloying elements like Cr, Al and Ti which are very susceptible to oxidation, especially when the alloy is initially present in a fine powder form with a high exposed surface area. Even when sintering is performed in a nominally high purity inert atmosphere such as Ar, the parts can pick up oxygen through surface oxidation. This oxygen pick-up can be detrimental to the final properties of the part. In this investigation superalloy powders were studied using a newly developed combined thermogravimetry-gas chromatography-mass spectrometry (TG-GC-MS) technique, allowing for quantification of the material's oxidation or reduction under gas containing differing oxygen contents. The GC-MS was able to determine the O₂, H₂O and CO₂ impurities contained within the inert atmosphere and how these evolved gases change through reaction with the superalloy powders during heating. The TG was used to quantify the change in mass of the superalloy powders during heating. A quantitative relationship between the concentration of evolved gases in the atmosphere and the extent of oxygen pick-up in the superalloy powders was established. (Oral presentation preferred)

843 The role of structural contribution in grain boundary segregation and cohesion of Ti

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In this paper we perform a series of density functional theory (DFT) calculations to investigate segregation of 3d, 4d and 5d alloying elements to grain boundaries (GB) in bcc and hcp Ti. We study the effect of these elements on GB cohesion and analyze a role of structural contribution to the GB segregation and GB strengthening energies. The free surface and GB segregation

energies are analyzed by comparing results of DFT calculations to results of existing physical segregation models. Following the concept of low-alloying additions proposed in Ref [1], we suggest a list of the most promising alloying elements from the point of view of GB cohesion enhancement in both bcc and hcp Ti. [1] V.I. Razumovskiy, A. Y. Lozovoi, I. M. Razumovskii Acta Mater. 82 (2015) 369-377

844 Bridging the gap between ab initio and large scale studies – a Monte Carlo study of Cu precipitation in Fe

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In the present study, we investigate the reliability of efficient pair potentials in comparison to accurate ab initio potentials as energy descriptions for Monte Carlo simulations. As test scenario, we take the phase decomposition in binary $\text{Fe}_{1-x}\text{Cu}_x$. In a first effort, we predict thermodynamic equilibrium properties of bcc-rich Cu precipitates in an Fe-rich solution with a temperature and composition dependent Cluster Expansion (CE). For the CE, combined ab initio and phonon calculations for various configurations serve as input. Alternatively, we apply the Local Chemical Environment (LCE) approach, where the energy is described by computationally efficient pair potentials between atoms, which are calibrated on the first principles CE results. We observe that, although fundamentally different, both approaches provide similar information in terms of the equilibrium precipitate radius as well as interface constitution. The computational effort in the LCE approach is, however, significantly lower. We conclude that the CE is especially valuable in predicting energies of various ground state configurations for small systems, while the use of pair potentials is particularly useful in evaluation of cluster formation and evolution statistics for large-scale simulation cells.

845 Microstructural Changes During Creep Process of Friction Stir Welded AZ31B-H24

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Friction Stir Welding (FSW) was applied in the current study in order to butt weld AZ31B-H24 alloy plates. Creep tests were conducted both on the parent material and on the friction stir welded specimens. Optical microscopy, Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and aging experiments were applied in order to study the microstructure stability of the parent material together with that of the creep specimens. The microstructure of the AZ31B alloy was found to be unstable under creep conditions. In the case of friction stir welded AZ31B the material undergoes during FSW both recrystallization and grain growth, then the exposure to temperature during creep yields an extensive additional grain growth. On the other hand, twinning and twin-induced recrystallization occur as well during creep so that ultra-fine grains are being created concurrently.

846 On the influence of microstructure and thermally activated processes on anomalous yielding point phenomena during nanoindentation

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Small scale testing techniques, such as nanoindentation, are widely used for assessing local mechanical properties in order to shed more light into small scale deformation processes under ambient and non-ambient conditions. Especially at elevated temperatures, a careful experimental setup is required in order to ensure reliable and reproducible properties, since e.g. thermal drift issues can play a major role, and obscure the actual material response.

This work will focus on probing different materials exhibiting an anomalous but currently not well understood yield-point phenomena, such as found for the PLC-effect. Applying nanoindentation strain-rate jump tests to e.g. coarse-grained Al-Mg alloys, a well-developed stair case loading behaviour can be found. Increasing the Mg-content, the jumps get more precise and larger in size, simultaneously paired with a negative strain-rate sensitivity. By decreasing the grain size of the material, the serrated trend diminishes. However, the resultant hardness still shows some, but different and partly unclear yield-point phenomena. In a transient regime after each strain jump to a lower strain-rate, the hardness initially decreases immediately followed by an increase back to a lower but almost constant level strain rate dependent level, as expected for ultrafine grained materials. Moreover, we show that a similar, irregular behaviour during nanoindentation strain-rate changes is also found for other materials, such as Austenite or other Fe-based materials. Finally, the extent and trend of the measured yield-point phenomena are directly compared to each other and will be discussed in terms of alloying elements, microstructural, and thermal activated influences.

847 Anelastic behaviour of amorphous TiAl measured by in-situ electron scattering

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Metallic glasses exhibit a number of superior mechanical properties such as high strength and high elastic limit that are a consequence of the amorphous nature of the structure. Therefore, high interest exists in the characterisation of the structure of amorphous materials and the correlation to the mechanical properties. Due to the lack of structural order metallic glasses show also a time-dependent elastic behaviour. This anelasticity is a consequence of topological instabilities leading to local anelastic deformations under stress. It is the aim of the present study to investigate the anelastic behaviour of an amorphous TiAl thin film by comparing macroscopic and microscopic strain measurements during tensile deformation in-situ in the transmission electron microscope.

TiAl films were synthesized by co-deposition of Ti and Al on a Silicon wafer by DC Magnetron Sputtering. Photolithography and reactive Ion etching techniques were used to co-fabricate MEMS based tensile testing stages with freestanding thin films [1]. Tensile tests were carried out in a Philips CM200 microscope. The special design of the samples allows macroscopic strain and stress measurements. Microscopic strain tensor on atomic level was obtained from electron scattering images by tracing the shift of the maximum of the first broad diffraction halo

during tensile loading. The anelastic contribution measured by the difference between microscopic and macroscopic strain is compared with strain-rate dependent stiffness measurements. [1] W. Kang, J. Rajagopalan, M.T.A. Saif, *Nanosc. and Nanotechn. Letters* 2 (2010) 282. C. E. and C. R. acknowledge financial support by the Austrian Science Fund FWF: [I1309].

848 Innovative surface treatments of titanium alloys for biomedical applications

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Biomedical engineering is an advanced technology based on an extremely complex development of advanced biomaterials. Since the first Consensus Conference in Chester (UK) on Definitions in Biomaterials of the European Society for Biomaterials, in 1986, biomaterial was defined as 'a bioinert or bioactive material used in a material advice, intended to interact with biological systems, restore functions of natural living tissues and organism in the body'. In this way, passive metallic materials (as titanium alloys), a broad spectrum of bioceramics, even biopolymers and all combinations of these biomaterials are used for numerous medical devices owing to their high biocompatibility. For example, titanium alloys can be employed for the femoral stems in the total hip joint replacement (trh) or for dental applications. Among the different clinical aims of an implant, a high osseointegration is required and crucial.

In order to prevent the alloys from the aggressive body environment, surface modification of implants are employed to render them protection from both wear, corrosion and even tribocorrosion. In addition to the surface treatments, new implant materials are also being fabricated with biocompatible alloying elements to reduce the toxic effects of the alloying elements. These presentation describes the methodologies that could be adapted to overcome some of the factors leading to implant failure. It gives a panorama and shows that the different processes can increase noticeably the performance of the alloy as orthopedic and dental implant. It also gives prospects for the development of new possible ways for enhancing the biosecurity of such material.

849 Nanostructured transition metal oxides: Application in conversion and storage of energy

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Breakthroughs in the field of conversion and storage of energy are intimately related to the progress in materials research. In particular, materials obtained using the potential of nanotechnologies should give rise to new advances. The properties of transition metal oxides are very attractive due to their numerous applications in the environmental domains (electrochemistry or photovoltaics for instance...). In order to remain compatible with low temperature processes on plastic substrates, we develop syntheses of nanostructured titanium oxides at moderate temperatures (<200°C). Solvothermal process allows us to adjust the size and variety of titanium dioxides by tuning pH or by using organic solvents. Low temperature processes are particularly well adapted to monitor the size, the allotropic variety and the shape

of oxide and gives us the opportunity to optimise materials for energy application as powders but also as colloidal solutions. The solvent is selected to monitor the physico-chemical properties of the obtained solutions. The integration of nanomaterials requires the elaboration of films. They can be deposited by printing processes or by electrodeposition, without annealing at high temperature. Some examples of oxide thin film deposited by solution processes will illustrate the potentials of our approach in the field of organic bulk heterojunction solar cells.

850 Laser pulse simulation of high energy transient thermal loads on plasma sprayed W for NFR

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W is a plasma-facing material candidate for applications in future nuclear fusion reactors (NFR). Transient thermal loads of high energy on plasma sprayed W have been simulated by the use of single laser pulse. Using the high power Nd we carried out the experiments: YAG/Glass laser operating in first harmonic (wavelength $\lambda = 1064$ nm). The pulse parameters were: energy $E \approx 8$ J, pulse duration $\tau \approx 15$ ns, focal spot size diameter $\Phi = 200$ μm corresponding to a fluence of 1.7×10^{12} W/cm². The damage produced by the laser pulse focused on the W surface has been then investigated by scanning electron microscopy (SEM) observations. The main effect of a single laser pulse is to induce weight loss with the formation of a crater corresponding to the spot central area. Melting, and successive re-solidification of W, give rise to a typical morphology with metal drops decorating the crater wall and rim. In a more external area thermal stresses lead to surface roughening and an extended network of cracks, which often follow the grain boundaries. Furthermore, it was observed that pores present in the original material become preferred sites for weight loss. Therefore, in addition to high purity and small grain size, low porosity is a fundamental requirement for W armours in tokamaks.

851 Study on the formation of stray grains during directional solidification of Nickel-base superalloys

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Ni-base superalloy single-crystal turbine blades are widely used in gas turbines for aircraft propulsion and power generation as they can be subjected to high service temperature and show high mechanical properties due to the almost total elimination of grain boundaries. Particularly in presence of complex geometry shapes, rare grains nucleating apart from the primary grain, become a serious problem in directional solidification, when characterized by high-angle boundaries with the primary grain, extremely brittle due the elevated amount of highly segregating elements and the absence of grain boundary strengthening elements. It is of fundamental importance analysing the physical mechanisms of formation of stray grains, to understand which thermo-physical and geometrical factors highly influence their formation and to find possible ways to reduce the impact of the problem. In this paper constrained dendrite growth and heterogeneous grain nucleation theories have been used to model the formation of

stray grains in directional solidification of Ni-base superalloys. The study allows to derive the preferred locations of stray grains formation and the role played by the most affecting factors: (i) geometrical: angle of primary grain dendrites with withdrawal direction and orientation of the primary grain with respect to the side walls, responsible for the formation of volumes where the stray grain undercooling is lower than the undercooling of the columnar dendrite tip; (ii) process and alloy: thermal gradient ahead to the solidification front and alloy composition, influencing the columnar dendrite tip undercooling; (iii) wettability of foreign substrates, on which the stray grain undercooling strongly depends.

852 A model for strain hardening, recovery, recrystallization and grain growth with applications to forming processes of nickel base alloys

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An ensemble of N spherical grains is considered, each of which is characterized by its radius r_i and by a hardening variable a_i . The hardening variable obeys a Chaboche-type evolution equation with a hardening term and terms for dynamic and static recovery. The evolution equation for the grain radius contains the usual contribution of the grain boundary energy and, in addition, a term for the stored energy associated with the hardening variable. The grain growth law is modified by the Zener pinning force exerted by particles on the grain boundaries. Particles may grow or disappear during the temperature cycles occurring in forming operations; this is described by an evolution equation for the particle radius derived from Onsager's thermodynamic extremal principle. New grains are created by recrystallization in grains whose stored energy density exceeds a critical value, and the nucleation rate is given by the difference between the actual and the critical stored energy density. This set of coupled first order differential equations in time is integrated numerically by an Euler forward scheme. The model parameters are adjusted using stress-strain curves measured in compression tests, as well as measured recrystallized volume fractions and grain size distributions after the test. The capabilities of the model are demonstrated on forming processes of nickel base alloys, for which it is important to predict the microstructure resulting from a complex temperature and deformation history.

853 Development of a parameter window for fibre-laser beam welding of aluminium-lithium alloy without filler material

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In the aircraft industry, laser beam welding is concurring with riveting, due to its high process velocities, flexibility and efficiency, and the chance to avoid corrosion nuclei like the holes of riveted structures. Most welds in aluminium alloys are performed with the use of filler wire, to overcome the hot crack sensitivity and to fill up material losses which occur during welding. The aim of this work was to investigate the weldability of 3.2mm thick AA2198 sheets when

autogenously welded in butt joint configuration without the use of filler wire, to compare it with the weldability of AA2139 and to analyse the mechanical properties. An Yb:YAG fibre laser, with a maximum output of 8kW, was used for welding both alloys. Advanced metallurgical examinations were performed to study the hot cracking behaviour and mechanical tests were used to analyse its influence on the resulting mechanical performances of the joints in terms of microhardness and UTS. To improve the hot cracking behaviour, different welding conditions were investigated. During the study was also found that a major influence on porosity was not the type of shielding gas or its flow rate but the power density on the surface of the specimen. For both materials crack-free welds were obtained with high joint efficiency. In the case of AA2198 a slight level of porosity could not be avoided.

854 Thermo-mechanical processing advanced high-strength steels: atom probe microscopy guided materials design

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Motivated by the increasing environmental demand for stronger steels that achieve dramatic weight savings in engineered structures, this paper will summarise recent research on the design of advanced high strength steels (AHSS). The critical enabler has been atom probe microscopy, where subnano- to nanoscale resolution of the chemistry and crystallography has guided selections of steel composition and thermo-mechanical treatment. Our focus is on steels exhibiting a reverse austenite transformation to obtain an austenite-ferrite duplex microstructure. We are seeking to extend the performance and practicality of third-generation (G3) AHSS utilizing an ultrafine grain size. Recent results using high pressure torsion on a TWIP steel to achieve nanostructured grain sizes will also be discussed.

855 Low temperature toughness stability increasing of X65 and X70 steel using austenite evolution models

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The research was aimed on improving the stability of low temperature toughness of X65 and X70 strips for linepipe produced by Thin Slab Direct Rolling (TSDR). The reason of unfavorable results of impact toughness and drop-weight tear tests is nonuniformity of final structure. The study includes analysis of structure at different stages of TSDR process, developing of recrystallization and grain size models, adaptation of the models on a laboratory rolling mill, creating and industrial testing of new program tool for processing technique improvement. It was determined that coarse initial grains and partial recrystallization during rolling are causes of the structural nonuniformity and a strong spreading of tests results. With the new program tool specific processing parameters were used for achieve of uniform final structure. As a result, the spreading of tests results was considerably reduced and product yield was raised.

856 Numerical and experimental investigation on dissimilar friction stir welded butt joints made of AA7020-T651 and AA6060-T6

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This work deals with the FSW joints composed of 2 hardened alloys AA7020-T651 and AA6060-T6. First of all, an experimental study has been carried out to obtain satisfactory joints in an industrial context with welding speeds up to 1400 mm/min. A microstructural analysis has been done for each joints. The mechanical properties has been also studied by means of hardness measurements, residual stress analysis, and tensile tests. Overall, the results show that friction stir welding can lead to satisfactory mechanical properties which can be better than the ones obtained by standard MIG welding. To understand the formation of the joints, a finite element model has been developped to simulate the the material flow around the pins. The numerical approach includes the complex shapes of the pins to investigate the periodic effects inside the joints. A comparison with some experimental obervations shows the interest of the numerical simulation for understanding periodic phenomena such as onion rings.

857 Effect of previous grain size on recrystallization texture and the formability of a Nb ferritic stainless steel

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The ferritic stainless steels are materials used in several segments due to the excellent combination of mechanical properties and corrosion resistance. The mechanical properties of these alloys are strongly dependent on the microstructural characteristics and crystallography texture. The aim of this experimental study is to investigate the roles of the previous grain size of the hot rolled sample on the development of the microstructure, texture and formability of ferritic stainless steel. The main elements of chemical composition of the steel under investigation were 16.0 %Cr, 0.021 %C, 0.024 %N and 0.35 %Nb. Coarse and fine grains samples were cold rolled up to 90% thickness reduction and annealed at 880 °C with soaking time of the 24 s. The texture measurements were performed by Electron Backscattered Diffraction (EBSD) in longitudinal section. The formability was evaluated by the R-value and planar anisotropy (Δr) in tensile tests. The final microstructure after annealed was more homogenous for smaller initial grain size sample. This condition was favorable to develop gamma-fiber, with sharpness intensity in $\{111\}<121>$ components. The highest R-value and smallest planar anisotropy was obtained for a $\{111\}/\{001\}$ ration around 5.37. On the other hand, coarser initial grain size sample had showed a heterogeneous microstructure and texture, performing badly in mechanical tests (anisotropy).

858 The behaviour of graphitized steels in machining processes

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Graphitized steels are claimed to perform excellent in machining processes. They therefore can be considered as environmental friendly alternatives to widely used Pb-alloyed steels. Due to liquid metal embrittlement and in-situ lubrication Pb improves machinability in a narrow tool-chip interface temperature window corresponding to low machining speeds. Although graphite inclusions are also supposed to generate in-situ lubrication, the mechanism and the corresponding optimum working zone is not very clear. The present work applies a new test methodology (including in-situ tribology, analysis of material flow and chip formation, tool lifetime studies) to investigate the effect of graphite inclusions in turning and drilling operations. Pb-alloyed low carbon free-cutting steel and Pb-alloyed case hardening steel were used as reference steels. The obtained results were discussed against the background of existing industrial production processes.

859 A new approach for the spectroscopic detection of different pH-values

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Fermentation based bioreactors are the state of the art biotechnology tool for cultivating microorganisms like bacteria or algae. New approaches aim for the constant miniaturization of such bioreactors in the framework of personalized medical care or high throughput experiments. Such small scale devices pose great challenges for measurement technology. The objective of the present work was the development of a micro-pH meter for the determination of the pH-value within bioreactors with a volume of up to 200 µl in total. Lower detection limits could be determined correlating the pH-value with the spectroscopic analysis of bromothymol blue in a micro-sized-channel structure utilizing glass fibers, enabling measurements in sample volumes down to the range of picoliters. For the characterization of this setup a spectrophotometric calibration was carried out with the indicator at different pH-values to detect specific absorption maxima. For the determination of acidic pH-values the absorption maximum is set at a wavelength of 433nm and for alkaline pH-values at 617nm. Determining the pH-value within micro-sized 150µl-bioreactors a different illumination system consisting of a RGB-sensor and a LED light source was utilized. This setup improved the handling of the measurement system for practical applications. Phenol red was successfully applied as the pH indicator for this second setup. The operating software for the measurement technology was developed using LabVIEW 2013, enabling data acquisition, sensor calibration and light source control via an Arduino-Board. Furthermore, it aims for the enhancement of data quality and detections limits as well as for the improvement of the sensor system.

860 DFT simulations of dislocations with mixed character in BCC metals

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The body-centered cubic metals represent a technologically highly relevant material class including Fe and the refractory metals W and Mo. A major drawback of this material class is given by the brittle-to-ductile transition which for some metals, e.g. W occurs above room temperature. Traditionally, a dominant role in this connection is assigned to the $\frac{1}{2}\langle 111 \rangle$ screw dislocation which, due to a non-planar core, has low mobility and limits plastic deformation. Recently, however, also certain dislocations of mixed character with planar core have been proposed to possess Peierls stresses not much smaller than the one of the screw dislocation. Hence, these dislocations might play an equally important role to explain the limited plastic deformation of bcc metals. In this talk we will present simulations for dislocations of mixed character in bcc metals. The focus is laid on the atomistic structure of the core, in particular its width, and the Peierls barrier. The simulations are based on DFT and treat the dislocations either in periodic dislocation arrangements or within a Peierls-Nabarro approach. We will focus on the trends observed for different bcc metals, most notably Fe, Mo, W and also discuss the impact of alloying for W. We will compare the Peierls barrier of mixed dislocations to the one of $\frac{1}{2}\langle 111 \rangle$ screw dislocations and discuss the implications for the thermal activation of dislocation glide. These findings contribute to an improved understanding of plastic deformation and the ductile-to-brittle transition in this metals.

861 Overview of some innovative coatings for electrical applications at Schneider Electric

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Surface coatings are widely used in electrical industries for connectors applications and electrical contacts parts mainly to upgrade the contact substrate properties such as corrosion resistance, electrical conductivity, frictional properties or solderability. For many years, Schneider Electric has been involved in developing new coating solutions for specific applications with the aim to fulfill engineering requirements with an optimized manufacturing cost. In this framework, an overview of innovative coatings developed by Schneider Electric will be presented. A first highlight will be made on localized conductive coatings made by electroplating or thermal spraying on copper or aluminum substrates, while a second part will be dedicated to the development of composite deposits. A comparative analysis of these different techniques will be drawn in terms of technical requirements like coating thickness, substrate geometry, deposited materials in parallel with manufacturing considerations like cost breakdown, ease of industrialization and environmental aspects.

862 Development of a 2.25%Cr steel grade T/P P23 reinforced with micro/nano-carbide particles prepared by self-propagating high-temperature synthesis

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A 2.25%Cr steel grade T/P P23 was reinforced by 1 wt. % of (Ti,Nb)C carbide. Carbide particles up to 20 micron in size were obtained by self-propagating high-temperature synthesis. Casting ingots of reinforced steel were prepared incorporating these carbides in a induction melted steel grade T/P. Microstructural observations of as cast material revealed a fine eutectic solidification structure with the presence of martensite laths inside the dendrites and carbide particles in the interdendritic spacing. This suggests the partial dissolution of the carbides. Compared with the unreinforced steel, an increase of hardness values was observed that is associated to microstructural features. The castings were laminated to obtain compression samples that were tested at strain rates in the range 1 to 20 s⁻¹ and temperatures between 950 and 1250°C to simulate the processing conditions. Stability maps were constructed to determine the optimal processing temperature at a given strain rate.

863 Doping nanocrystalline alloys to improve strength and toughness

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Doped interfaces can have unique structures and, in some cases, thermodynamically-stable interfacial complexions can form. In this talk, the use of such features to alter mechanical behavior will be discussed. Molecular dynamics simulations are used to show that disordered complexions can act as high-capacity sinks for dislocations, and to identify the processing conditions which promote the formation of such boundary structures. Experimental validation of this concept is provided by high resolution transmission electron microscopy in conjunction with energy dispersive x-ray spectroscopy, showing segregation of Zr to the boundaries of Cu-Zr alloys created with mechanically alloying and providing evidence for the formation of amorphous films. Microcompression and in-situ bending experiments are then used to quantify the effect of disordered complexions on mechanical behavior, showing that both strength and ductility can be controlled with segregation engineering, giving a potential path for producing structural metals with optimized mechanical properties. Finally, efforts to create bulk nanocrystalline materials with these unique boundary features will be described.

864 Optimization of design and development of advanced TMCP steel plates using physical simulation

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Continuous development of new and optimization of existing steel grades is of fundamental importance to meet the further increasing demands on plates for pipes and structural applications. Challenging requirement profiles like plates with higher thickness, higher strength, improved weldability and higher toughness also at lower temperatures can be achieved by the utilization of TMCP technology. A precise understanding of the metallurgical mechanisms and parameters controlling the microstructural evolution during processing provides the basis for a tailored and optimized design. Physical simulation allows studying microstructural phenomena in a fast and efficient way and thus can play an important role in the development process. Selected results are presented and illustrated with recent applications of high-strength heavy plates.

865 Deformation behavior analysis of the single-phase ferritic steel using by the digital image correlation method and crystal plasticity fast Fourier transform method

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Mechanical properties of polycrystalline material depend on hierarchic structure. On the polycrystalline materials, resolved shear stress of each grain is different during deformation. And, binding force acts on each grain because displacements across the grain boundary must be continuous. For this reason, elastic-plastic deformation behavior of polycrystalline material is inhomogeneous. It is important to understand microscopic deformation for understanding microscopic deformation. In this study, inhomogeneous deformation in the single-phase ferritic steel has been investigated by the digital image correlation method and the fast fourier transform based micromechanical modeling. On the basis of these results, the influence of the characteristic of deformation inhomogeneity on the mechanical property of single-phase ferritic steel is discussed. Interstitial-free steel was employed. Local plastic strain during tensile deformation was measured by the digital image correlation (DIC) method. Local grain orientation obtained by SEM-EBSD method. In addition, Three-dimensional structure of each grains measured by the DIC method was observed by serial sectioning. And, crystal plasticity simulation was carried out base on 3D structure obtained from serial sectioning and SEM-EBSD. After comparing the local strain map obtained from DIC method with that from crystal plasticity simulation, it showed a good match, but it had a margin of error in part. From the results of the serial sectioning, the error was found to be caused by the influence of the grain shape and surface.

866 Structural rejuvenation for improved properties in metallic glasses

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Relaxation state in metallic glasses is regarded as one of the important parameters originating from the intrinsic random atomic configuration since various properties such as mechanical properties strongly depend on it. Recently, the authors have reported that the cooling rate just above glass transition temperature, T_g fixes the relaxation state finally [1] and have succeeded to control it by simple post annealing around the corresponding temperature region followed by the appropriate cooling. Additionally, we have confirmed that the controlled relaxation state also correlated to the fundamental properties such as local structure as well as mechanical properties by experimental and simulation studies. [2,3] In this presentation, we evaluate the rejuvenated $Zr_{55}Al_{10}Ni_5Cu_{30}$ bulk metallic glass (BMG) by low temperature annealing at $1.07T_g$ from the viewpoints of thermodynamics, mechanical properties and molecular dynamics (MD) simulation studies. The results obtained in the present study propose a novel processing method for improvement of properties of metallic glasses and provides useful information on the application of BMGs. [1] J. Saida et al., Met. Mater. Trans. A, 42 (2011) 1450. [2] J. Saida et al., Appl. Phys. Lett., 103 (2013) 221910. [3] M. Wakeda et al., Sci. Rep., 5 (2015) 10545.

867 Quantitative evaluation of aging embrittlement cracking susceptibility in weld metal of heat-resistant alloys

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The present study focused on the quantitative evaluation of the aging embrittlement cracking susceptibility in the weld metal of heat-resistant nickel-base alloys (617, 263, 740) and stainless steels (316H, 347H, 321H, 800H). The laser confocal microscope equipped with the high temperature tensile testing machine was used to investigate the aging embrittlement cracking behavior under the operation temperature, and enabled the quantitative evaluation of the aging embrittlement cracking susceptibility to measure the critical crack opening strain by in-situ observation during tensile testing. The tensile test was carried out at 700 °C using the U-notched specimen. The tensile specimens were aged at 700 °C for various times prior to testing. An aging embrittlement crack initiated at the notch root and propagated along grain boundary in the weld metals. The critical crack opening strain decreased with an increase in the aging time for all alloys. Microstructural observation revealed that carbides were precipitated in all weld metals during aging, and that gamma' phase was additionally precipitated in the 617, 263, 740, 800H weld metals with progress in aging. Precipitation of carbides and gamma' phase resulted in the decreased critical crack opening strain due to the intergranular embrittlement and the transgranular hardening. The effect of carbon content on the aging embrittlement cracking susceptibility was investigated. The critical crack opening strain was ameliorated by reducing the carbon content in the weld metal. It follows that the aging embrittlement cracking susceptibility could be improved by controlling the balance between intergranular and transgranular strength at the elevated temperature.

868 Age hardening behavior of Al-Li alloys produced by sand mold casting

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Al-Li alloys have higher specific strength and modulus than other conventional aluminum alloys, it is focused as a good material for weight reduction of aerospace-related fields. In recent years, investigations have been conducted on the wrought products, such as rolling sheet and extruded bar. However, since the Al-Li alloy are highly active and hard to cast, there has been limited research on Al-Li alloy for sand mold casting. In this study, age-hardening behavior of Al-2.5mass%Li alloys cast into sand mold were investigated by OM, SEM, TEM and micro-vickers hardness measurement.

869 Solid-state bonding of 5052 aluminum alloy/316L stainless steel by using organic salt formation/decomposition reaction

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In recent years, weight reduction of transporters have been needed for the preservation of the global environment. Therefore, sophisticated assembly of the 5052 aluminum alloy and 316L stainless steel has been attracting attention. Moreover, 5052 aluminum alloy has the high specific strength and 316L stainless steel has the high corrosion resistance and high heat resistance. In this study, the effect of the metal salt generation bonding technique on the strength of a direct-bonded 5052 aluminum alloy / 316L stainless steel interface was investigated. The specimen of 5052 aluminum alloy and 316L stainless steel surfaces were modified by boiling in organic acid. In this bonding process, direct bonding was performed at a bonding temperature of 733 ~ 773 K under a pressure of 20 MPa (for a bonding time of 900 s). Analysis of the product were performed by FT-IR to clarify the metal salt is generated after surface modification. After solid-state bonding, tensile test were performed by Instron universal testing machine. Observation of interfacial microstructure and fractured surfaces were performed by SEM. As a result, a high bond strength joint at low temperature and low deformation was obtained.

870 Syntheses of novel hydrides under high pressure and high temperature

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High-pressure and high-temperature synthesis is a powerful method to fabricate novel hydrides because hydrogen becomes extremely reactive to form novel hydrides under high pressure. In situ synchrotron radiation X-ray diffraction measurements are effectively used to explore

synthetic conditions and to investigate the reaction mechanisms. Theoretical calculations based on first-principles calculations enable us to predict the thermal stability and crystal structure of the target hydrides before the high-pressure experiments, which leads to the rapid discovery of the novel hydrides. Synthetic studies on aluminum-based interstitial hydrides and lithium-containing complex hydrides will be presented. These results demonstrate that the high-pressure technique is useful for discovering novel hydrides.

871 Thermo-mechanical processing of a beta titanium alloy

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Beta titanium alloys feature favorable properties with the possibility of attaining high strength along with decent ductility levels. In this study, a Beta-c titanium alloy was subjected to cold deformation assisted with various heat treatment schedules. Microstructure investigations along with mechanical behavior of the processed samples are presented. The flow stress and elongation levels were shown to be strongly dependent on the cold work amount, and strength levels up to 1600 MPa were achievable after aging treatments. Prior cold deformation was shown to be instrumental in attaining desired precipitation characteristics.

872 Nanostructure-driven control of defects in GaN grown by the Na flux method

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GaN is one of the most efficient energy-saving materials for realizing high performance power devices and optical devices such as light-emitting and laser diodes. Defect-free and large size GaN single crystals are strongly demanded as a freestanding substrate for the devices. Solution growth techniques, such as the ammonothermal method and the Na flux method, have been developed for the growth of GaN with millimeter-scale thickness. In particular, the Na flux method combined with flux composition control and coalescence growth techniques has successfully achieved the growth of bulk GaN with an extremely low threading dislocation density. In this work, we demonstrate the Na flux growth techniques based on an effective way of reducing threading dislocations in GaN. Transmission electron microscopy clearly reveals details of the structures in the vicinity of the interface between the Na-flux-grown GaN and the template GaN grown by metalorganic chemical vapor deposition. We elucidated that the reduction of threading dislocations is essentially due to the blockage of dislocation propagation by nanometer-scale voids formed at the interface during the Na flux growth as well as lateral and collective dislocation propagation above the interface. The cause of nano-void formation, the role of nano-voids in controlling the dislocation behavior, and the mechanism of lateral and collective dislocation propagation are discussed.

873 High temperature tensile behavior in Si-bearing near alpha titanium alloy

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High temperature yield strength and dynamic recrystallization in Ti-1100 were investigated. Ti-1100 is one of near alpha titanium alloys and contains Si for improving high temperature mechanical properties. Ti-1100 exhibits martensitic transformation by quenching into iced brine after solid solution treatment. Hereafter specimens subjected to quenching into iced brine and to cooling in air after solid solution treatment are called IBQ specimen and AC specimen, respectively. Yield strength of IBQ specimen is larger than that in AC specimen at room temperature because IBQ specimen is in martensitic phase with high dislocation density. In contrast, at 700°C yield strength of IBQ specimen is smaller than that in AC specimen. This indicates that the decrease in the yield strength in IBQ specimen due to temperature rise is larger than that in AC specimen. This fact may arise from competitive effect of solid solution strength by Si and precipitation strength by silicide. After tensile test at high temperature, IBQ specimen exhibits morphological change from lath structure to equiaxed structure, but AC specimen does not. It is indicated that dynamic recrystallization occurs during the tensile test of IBQ specimen due to high dislocation density. Effect of silicide on the dynamic recrystallization was investigated using two specimens: one included more silicide precipitates and the other less. The former specimen shows smaller recrystallized grains than the latter. It is indicated that the specimen including more silicides exhibits smaller recrystallized grains.

874 Thermal spray coating application onto low temperature polymer substrate

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Functional coatings onto polymers are of great interest for different domains such as aerospace and medical industries. Due to the high thermal load of the spray process and the poor coating adhesion properties of the polymer substrates it is a challenge to apply such coatings. In this work we report the results of an advanced plasma spray process which allows to apply metallic and ceramic coatings on to high and low temperature polymer materials such as PEEK and Polyethylene (PE). This study reports the experimental characterisation of the strength and adherence of titanium metallic coatings on PEEK and PE substrates. The physical and chemical microstructure of the substrate were extensively examined to prove that the coating process does not detrimentally affect the material, especially for the temperature sensitive PE. These results provide guidelines for the design of polymer component with new surface functionality as for example orthopaedic implants for which such a titanium coating is used to enhance anchorage of bone tissues.

875 Anodizing of Al alloys in tartaric, boric and sulfuric acids mixture

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Al alloys have been widely used in automotive and aircraft industries for many decades due to its light weight. AA 6022 alloy was used in automotive body panels. On the other hand, AI-8Fe was considered as high strength material to be used at elevated temperatures.

In this study, non-chromate anodizing process was carried out in a solution contains tartaric, boric and sulfuric acids to produce corrosion protective and strongly adhered oxide film on both AA 6022 and AI-8Fe alloys. Anodic oxide coatings were formed on the surface and lead to enhancement of the anti-corrosion property of Al alloys.

876 Manufacturing of ODS RAFM Steel: Mechanical and Microstructural Characterization

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Ferritic ODS 14Cr steels reinforced by means of Yttrium oxide nanoclusters represent one of the options for future structural applications in high nuclear Generation IV reactors. Due to their high tensile properties and resistance to irradiation damage, Oxide Dispersion Strengthened Steels (ODS-S) have been suggested for nuclear fusion applications. The present paper describes the experimental procedure of mechanical alloying, canning and hot extrusion adopted to produce ODS rods. The effect of variations in the processing parameters are also discussed. Hot extrusion has been successfully applied to produce a batch of about 10 kg of ODS steel. Full size ASTM E21 and E8 specimens have been tested from room temperature up to 800 °C. The microstructure characterization of the manufactured materials has been carried out by transmission electron microscopy. Ultimate tensile stress higher than 1350 MPa have been obtained in the as-extruded material and higher than 1100 MPa in samples annealed for 4 hours at 800 °C.

877 Elasticity and plasticity of earth's mantle minerals under pressure

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Understanding deep planetary interiors requires a multidisciplinary approach involving geophysical observations (e.g. seismic wave propagation), geochemical analysis of rocks, and studies of Earth materials properties at relevant P and T. Seismic wave variations in the Earth's interior can be used to derive temperature and compositional heterogeneities, and the deformation and flow state in deep regions. Laboratory measurements of the elasticity (e.g., sound velocities and elastic constants) and plasticity (e.g., strength and textures) of candidate materials remain, however, scarce. Here, I will discuss advances in determining the elasticity

and plasticity of mantle phases along the MgO-SiO₂-Al₂O₃-H₂O system. Specifically, I will present Brillouin scattering and X-ray radial diffraction studies of SiO₂ stishovite and hydrous phases (the so-called "alphabet" phases: Phase A, E, Superhydrous B and phase D) in diamond anvil cells that shed light into the dynamics of subducting slabs and the deep water cycle. The results elucidate the seismic signature of hydration at depth and show that hydrous phases display lower seismic velocities, higher anisotropy and lower strength than anhydrous phases, and therefore they accommodate most of the strain in the slab and easily develop textures. The results, combined with seismic observations from slabs in the Pacific, suggest substantial hydration of slabs penetrating below the transition zone (> 700 km depth). The implications for the large-scale dynamics and evolution of the lower mantle will be discussed.

878 Functionalization of the anodic 3D nanostructures by atomic layer deposition for energy applications

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Atomic Layer Deposition (ALD) has shown a great ability to conformally coat porous structures exhibiting a high aspect ratio. We report three examples of nanostructured electrodes fabricated using anodic oxidation in combination with ALD. The targeted applications are in the fields of electrocatalysis, microbatteries and photoelectrochemical water splitting.

Direct Ethanol Fuel Cells offer significant advantages due to ethanol non-toxicity and renewability and its high power density. TiO₂ have been successfully used as replacement of C as catalyst support because it exhibits a good chemical stability and it can enhance the activity of the catalyst. After a brief reminding on the TiO₂-nt fabrication and properties, the ALD of Pd nanoparticles into TiO₂-nt array and their electrochemical properties will be presented.

A 3D nano-architected composite negative electrode has been fabricated in order to be used in microbatterie. It consists of TiO₂-nt coated by a thin SnO₂ film grown by ALD. Such composite 3D structure increases therefore the active area, improves the kinetics of the system, facilitates the ion exchange at the electrode/electrolyte interface and, better accommodates the volume expansion induced by the Li insertion within the Li_xSn alloy. The last example consists of producing a nanostructured photocathode for water splitting in which the photogeneration and transport of the charge carriers do not occur in the same material. In order to create tailored 3D nanostructures, p-NiO and i-Sb₂S₃ thin films have been successively grown into nanoporous alumina by ALD.

879 Shape recovery of polymeric matrix composites by IR heating

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Shape memory composite (SMC) structures are of keen interest for aerospace applications. In previous works, the authors have studied SMC lab-scale deploying prototypes manufactured using two carbon fiber composite layers with a shape memory polymer interlayer. The prototypes were produced in the opened configuration and subsequently closed in the

memorizing step. The initial deployed configuration is recovered by heating the prototype. Memorization and recovery phases were performed by means of conventional heating (by hot air gun or heater plate). In this study, the authors evaluate the SMC heating through the use of an infrared lamp. Time, temperature and recovery percentage are measured during and after tests. A square plate was produced on purpose and recovered after different memory steps. This study is especially aimed to future space applications in which the deployment (recovery) phase is being initiated only by exposure to solar radiation. As a result of IR heating tests, initial configuration can be successfully recovered without failures.

880 Recrystallization kinetics and texture evolution of Nb stabilized ferritic 430 stainless steel cold rolled and isothermal annealed

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The ferritic stainless steel type 430 stabilized with Nb, with and without annealing after hot rolling, was cold rolled and subjected to isothermal annealing at temperatures 650, 700, 750, 800, 900 and 1000°C for times ranging between 10 to 86400 s. The quantification of the recrystallized fraction was performed by JMAK model and by microhardness measurements along the annealing time. The evolution of the texture was analyzed via EBSD technique. The recrystallization temperature occurred at 750°C. The Avrami exponents were between 0.8 to 1.2. The nucleation rate and grain growth decreased continuously with time. There was an increase in intensity of the component (111)[1-21] and (111)[0-11] of gamma fiber and a decrease in intensity of the deformation alfa fiber. The rotated cube component, feature of the hot rolled steel, decreased with annealing time. The results showed that occurred the grain refining after recrystallization, resulting in a final average size of 12.5 µm. A recrystallization kinetics model of the steel type 430, stabilized with Nb, which takes into account the nucleation and growth processes was obtained. In order to complement the analysis about the recrystallization kinetics, the effects of annealing temperature and time were researched by measurements of Grain Orientation Spread (GOS) parameter in the EBSD analysis software. In comparison with annealing time, annealing temperature had greater influence in the recrystallized fraction variation and softening behavior of the samples.

881 Thermal Effect during Electromagnetic Pulse Welding Process

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The magnetic pulse welding (MPW) is a solid state joining process, that can be successfully utilized to join dissimilar materials. This advantage attracted manufacturing industries to fabricate hybrid materials to attain materials with a combination of multiple attributes. The high speed impact during the welding process causes many interfacial phenomena, which have been reported in various research studies. Combined high speed collision, eddy current and Joule

heating due to the presence of electrical contact resistance cause noticeable heating. This heating is particularly significant at the localized region of the interface compared to other regions of the workpiece due to dissipation of plastic work and Joule heating during the welding. The induction heating due to eddy current affects the entire workpiece that is prominent before the collision. Coupled thermo-mechanical with integrated electromagnetic 3D simulations are carried out to investigate the heating during the MPW process caused by the combined induction heating, Joule heating and plastic dissipation. A case study of MPW, consisting in one turn coil combined with a field shaper, is used to investigate the welding process. The simulations were performed using LS-DYNA, which has the capability of using both finite and boundary elements to solve the thermo-mechanical problem during an electromagnetic forming. The predicted temperature distributions show a good agreement with the expected phenomena at welded interfaces of the samples. Minimizing the heating effect by identifying the influencing factors could be helpful to optimize and control the quality of the magnetic pulse welded parts.

882 Re-melting technique with high intense pulsed plasma beams applied for surface modification of steel. Own investigations.

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Among different methods used in surface engineering the re-melting techniques are widely applied. The techniques using high intensity pulsed plasma beams (HIPPB) are relatively new ones. The plasma beams were generated in a Rod Plasma Injector (RPI) operated in the Deposition by Pulsed Erosion (DPE) mode. The plasma pulses contained both ions/atoms of electrodes material and those of working gas. The applied pulse energy densities were sufficient to melt the near surface layer of steel and active elements were introduced to the melted material. Heating and cooling processes were of non-equilibrium type. The aim of this work was to investigate the changes of steel surface properties (morphology, elemental composition, presence of identified phases) and functional properties (wear and high temperature oxidation resistance) after alloying their near surface layer with chosen active elements. Samples of unalloyed and austenitic stainless steels were irradiated with short (us scale) intense (energy density 3.0 J/cm^2) pulses. The near surface layer - thickness in μm range - was melted and simultaneously doped with active element like nitrogen, cerium and lanthanum. Initial and modified materials were characterised using different investigation methods including non-destructive nuclear techniques. The wear tests and high temperature oxidation processes were carried out. The most important obtained results were: (i) austenitic structure was present in steel after HIPPB modification processes and (ii) modified surface layers showed significant improvement of tribological properties and increase of high temperature oxidation resistance as compared with initial material.

883 Effect of preparation conditions of TiB₂ particle dispersed Al composites on microstructure

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Pure Aluminum (Al) has been used as high performance heat sink, electrical line and power supply line because of its light-weight, good thermal and electrical properties and high cost performance. On the other hand, the higher performance heat sink and electrical line at room and high temperature are required because of increasing power density and higher integration of semiconductor and LED. The use of Al matrix composites is one of suitable solution, because these parts need multi functional properties. In this study, TiB₂ particles were selected as the dispersant in Al matrix because of its good thermal and electrical conductivity, and good mechanical properties. At first, the mixed powder of Al and TiB₂ particles was obtained from six kinds of mixing process, which parameters are conventional ball milling, milling with ultrasonic vibration, planetary and ball milling with wet process and dry process. And then 20 vol% TiB₂/Al composites were obtained from spark plasma sintering. The dense composites with over 98% in relative density with highly dispersed TiB₂ particles were obtained from wet mixing process. But the tensile strength of the composites prepared from dry mixing process is higher than that of wet mixing process. The thermal conductivity of the composites is about 120 W/mK, and the obvious difference between the wet process and dry process was not observed.

884 High speed X-ray stress measurement with a monolithic SOI pixel detector

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This study is on X-ray stress measurement with a two-dimensional detector which acquires image data of a Debye-Scherrer ring and then analyzes them with a theory, so-called the $\cos\alpha$ method. One of the authors, Sasaki, developed a new machine in 2011 which is based on the $\cos\alpha$ method and showed that, compared with the ordinary machines, the weight, occupied space, and measuring time is about 1/20, 1/15, and 1/10, respectively. The accuracy of the stress was also found to increase because of the fact that the acquisition number of X-ray diffraction data is about 70 times more than that of the ordinary method. Sasaki has used an image plate (IP) as a two-dimensional detector until now. It takes about 30 to 60 seconds for X-ray exposure and about 60 seconds for the data analysis. In this study, a monolithic SOI pixel detector (SOI) was used instead of an IP in order to reduce the measuring time. The SOI detector is one of the two-dimensional X-ray detectors developed by Arai et al. and is made from a silicon wafer. The authors used the SOI detector and succeeded in measuring the stress of a steel with CrK α radiation in two seconds. This measuring time is about 1/60 that of the IP-use method. It is also possible to acquire a Debye-Scherrer ring in 20 milliseconds, which is about 1/6000 the time of the IP-use method. The outline of IP-use and SOI-use X-ray stress measurements as well as their comparison is shown with some experimental data in this paper.

885 Physical modeling of chosen metallurgical processes

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Today physical modelling is commonly used tool for modeling metallurgical processes. It can be applied both to the steel metallurgy and non-ferrous metals metallurgy processes. It gives the opportunity to determine the hydrodynamic conditions of the processes. Although, the flow of mass and gas is not totally presented by this modelling, these kinds of research are very often and willingly used. This is because there is really hard to conduct experimental research in industrial conditions. Typically as a modeling agent water is used, so the physical modeling is not as expensive as the one carried out in industrial conditions. To obtain representative research from physical modeling the physical models has to be build according to the strict rules coming from the theory of similarity. The results obtained from the experimental test on the physical model, after verification, can be transferred to the real conditions. Article shows the chosen results coming from physical modeling of the steel production process. In the Institute of Metals Technologies of Silesian University of Technology the appropriate test stand was built to simulate the steel flow and mixing in the ladle. The visualization results will be presented. Additionally, the test stand to simulate processing condition during aluminium refining was also built. The exemplary results will be presented for different flow rate of gas, rotary impeller speed and different shapes of impellers. All presented results will be discussed and presented the perspectives of further research.

886 Quantitative evaluation of creep curve in compression by strain acceleration and transition objective index

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It has been well accepted that the creep characteristics of alloys and compounds are mainly evaluated by the minimum creep rate or the steady state creep rate. Almost the same strain-rate, however, can be evaluated even when creep curves show different shape in primary and tertiary stages in different conditions. The minimum or the steady state creep rate is one of indicative values, but not reflects entire aspects of evolution of deformation at a condition. Authors believe analysis in shape of creep curve should be considered for detailed understandings of creep deformation and one of the authors has proposed a method of quantitative evaluation of creep curve based on the evaluation of strain rate change and its strain dependence. The method provides information of shape change of creep curve during deformation objectively, and a method of reconstruction and extrapolation of creep curve from a part of creep curve. Authors have found that this method is applicable in many alloys especially in solid solution alloys in tensile creep. In this presentation, examples of evaluation and extrapolation of creep curve in compression are presented with comparison of tensile creep. Materials presented will be UFG magnesium and texture controlled Ti-Al alloys. Some creep curves are quoted from references. It is concluded that the extrapolation of creep curves in compression are similar to the extrapolation in primary stage in tensile creep, and imaginal minimum creep rates are reasonably extrapolated from part of creep curve in compression.

887 Effects of particle size on fabrication of Al-TiO₂ functionally graded materials by centrifugal mixed-powder method

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As one of processing methods of functionally graded materials (FGMs), centrifugal mixed-powder method has been proposed. The centrifugal mixed-powder is the casting method combined of centrifugal casting and powder metallurgy. This processing method has advantage that fine solid-particles, whose wettability with matrix melt is low, can be composed into metallic material. However, effects of particle size on microstructure and mechanical properties of the FGMs fabricated by the centrifugal mixed-powder method are unclear. In this study, two kinds of Al-TiO₂ FGMs are fabricated by the centrifugal mixed-powder method. One has large TiO₂ particles with diameter of 150 micron - 250 micron, and the other one contains small TiO₂ particles with diameter of 0.35 micron. In case of the Al-TiO₂ FGMs with large TiO₂ particles, the TiO₂ particles are homogeneously dispersed in Al matrix. On the other hand, TiO₂ particles in the Al-TiO₂ FGMs with small TiO₂ particles are distributed along grain boundary of Al matrix. Moreover, Vickers-hardness of the Al-TiO₂ FGMs with large TiO₂ particles is higher than that of the Al-TiO₂ FGMs with small TiO₂ particles. Since Al powders in the mixed-powder containing small TiO₂ particles are surrounded by the TiO₂ particles, the Al powders can be hardly melted by heat of Al melt at casting process. As a result, the Al-TiO₂ FGMs sample with small TiO₂ particles has low hardness. Therefore, it is found that large TiO₂ particles is more suitable for fabrication of the present Al-TiO₂ FGMs samples comparing with small TiO₂ particles.

888 Usability of Ti6Al4V powder via hydride-dehydride process for selective laser melting process

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In this study, we investigated a usability of a hydride-dehydride (HDH) powder for selective laser melting (SLM) process. Feedstock powders of the SLM for metals are generally recommended using spherical powder because of ensuring homogeneous flowing and packing conditions of powders in the SLM process. However, the spherical powder made by gas or plasma atomization is expensive and the yield of fine spherical powder after atomizing is low. Additionally, there are some materials which are difficult to produce the spherical powder. Then, investigation usability of non-spherical powder is required to expand a field of application of the SLM process. To investigate usability of HDH Ti6Al4V powder, we have characterized and compared properties of powders and SLM fabricated products between Ti6Al4V powder via gas atomization and HDH process. Flowability and packing density of the HDH powder were lower than the atomized power. Coating powder surface of the atomized powder was homogeneous, on the other hand, that of the HDH powder was not homogeneous, for example cracks or waves were found on the surface. Then, we have added lubricant to the HDH powder to secure the flow ability of it. In the result, relative density of SLM fabricated products made by both powders become same.

889 Effect of Cu and Ag addition on mechanical properties in Al-Mg-Ge alloys aged at different temperatures

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In this research, the effect of both 0.2% Cu and 0.19% Ag additions on Al-0.45% Mg-0.21% Ge alloys that have Mg/Ge ratio (at.%) of 2 is investigated. Alloy was fabricated using casting process. Alloy was subjected to homogenization treatment at the temperature of 623 K for 21.6 ks. Then, hot extrusion was conducted till the thickness of 1.5 mm. Solution treatment was carried out at the temperature of 873 K for 3.6 ks. Aging behavior at 473 K and 523 K was investigated by means of Vickers microhardness tester, and transmission electron microscopy (TEM).

890 Reactive plasma depositions of gallium nitride thin films on amorphous substrates and their properties

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Gallium nitride (GaN) is one of the transparent and semiconducting materials. It has a hexagonal wurtzite-type crystal structure and direct energy band gap of about 3.4 eV. Therefore, GaN is transparent for the visible light and also can emit near-ultraviolet light. Indium nitride (InN) has also the wurtzite structure, and its energy band gap is about 0.7 eV. Alloys between GaN and InN ($\text{In}_x\text{Ga}_{1-x}\text{N}$) keep the wurtzite structure and their energy band gaps continuously vary from 0.7 to 3.4 eV. Therefore, $\text{In}_x\text{Ga}_{1-x}\text{N}$ thin films have been utilized for the highly efficient short-wavelength light-emitting devices until now. They are also expected as materials for highly efficient multi-junction solar cells recently because they can efficiently absorb almost of sunlight of wide wavelength region. Usually, the nitride thin films are grown epitaxially on single crystalline substrates such as sapphire. Metal-organic vapour phase epitaxy or molecular beam epitaxy methods are generally used for the epitaxial growths. On the other hand, amorphous substrates are rarely used for their depositions. In addition, reactive evaporation or reactive sputtering methods are rarely used for the depositions though the methods are suitable for the low temperature and large area depositions. In this study, GaN thin films were deposited on a glass substrate or silicon wafers on which amorphous silicon dioxide layers of various thicknesses were pre-deposited. Some properties of the films such as crystallinities, optical properties, and electrical properties were investigated.

891 Relationship between microstructures and mechanical properties of temper heat treated metastable austenitic stainless steel

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Good strength-ductility relationship in metastable austenitic stainless steels is brought by active strain induced martensitic transformation, and their mechanical properties are generally adjusted by temper rolling. However, it is sometimes difficult to obtain desired mechanical properties by the temper rolling because the processing temperature is close to the transformation temperature and slight fluctuation of transformation temperature leads to significant change in volume fraction of the martensite phase. In this paper, “temper heat treatment” followed by strong cold rolling was examined in place of temper rolling. With this process, considerably improved strength-ductility relationship can be obtained by extremely refined microstructures mainly as a result of the reverse transformation from martensite to austenite. Effects of temperatures in temper heat treatment on microstructures and mechanical properties were investigated using annealed AISI 301L sheets with 20 μm or larger grain diameter. They were cold rolled at 80% reduction and heat treated at temperatures between 873 and 1273 K for 30 s. Specimens for tensile tests and microstructure observations were taken from the heat treated sheets of 0.2 mm in thickness. Obtained results are as follows:

1. Hardness after heat treatment decreased as temperature increased.
2. Microstructures were composed of a lot of extremely fine grains whose diameter was 1 μm or smaller. Reverse transformation completed during heating to 1073K and remarkable grain growth was recognized in the specimens heated to temperatures above 1073K.
3. It is confirmed that strength-ductility relationship was far better than that in the temper rolled sheet manufactured from the same initial materials.

892 Weighted individual crystallographic orientations capturing a given texture

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Simultaneous Electron Back Scatter Diffraction (EBSD) and Energy Dispersive X-ray Spectrometry (EDS) experiments provide spatially referenced information about the mineral phase, the crystallographic orientation (Kikuchi patterns), and meta data quantifying the reliability of the data. Given today's speed of the experimental equipment, a typical EBSD dataset comprises several ten to hundred thousand individual crystallographic orientation measurements. Their statistical distribution in $\text{SO}(3)$ is usually represented in terms of an orientation density function (odf). The odf can be estimated with non-parametric kernel density estimation, i.e., the superposition of radially symmetric bell-shaped kernel functions centered at the measured crystallographic orientations. The total number of orientation measurements acquired with EBSD is far too large to be used in numerical ab-initio simulations. To run simulations in a reasonable computational time, the initial sample size N has to be diminished by orders of magnitude yet preserving the texture, i.e., characteristics of the odf estimated using the full dataset. This problem can be tackled in terms of designing a set of weighted individual

crystallographic orientations of much smaller size N' honoring a given odf in some sense. We compare approaches to the construction of weighted crystallographic orientations in the spatial domain $SO(3)$ or in frequency domain applying least squares and maximum likelihood, respectively, preserving either the overall shape of the initial odf or its first Fourier coefficients. The methods we devise can easily be realized numerically with our free and open source Matlab toolbox MTEX.

893 Simulating cosegregation of carbon and oxygen in molybdenum with DFT

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In several high-temperature applications molybdenum and its alloys are favored materials due to their excellent properties at elevated temperatures, e.g. high melting point, high creep strength, low vapor pressure and low coefficient of thermal expansion. One drawback of these materials is the brittleness and intergranular fracture mode near room temperature caused by the reduced cohesive strength of grain boundaries (GB). For this problem the segregation of interstitial solute elements, such as carbon and oxygen, is crucial. We present atomistic simulations of GBs in molybdenum based on density-functional theory (DFT), where we investigate the effect of C and O segregation on the cohesive properties of the GBs. First, the segregation is treated separately for the solutes and in a second stage we also look onto cosegregation phenomena, where elements interact with each other. These interactions are either repulsive or attractive, so an element can be either depleted or accumulated (clustering) at an interface leading to large changes in GB cohesion. By modeling not only single solute segregation but interactive cosegregation and clustering of C and O, we elucidate processes which are closer to real alloys. The changes in GB cohesion are evaluated by ab-initio fracture simulations, where the GB is subjected to uniaxial loading. With these simulations, we try to give suggestions regarding the detrimental effect of O and other impurities at GBs in Mo and how to improve the cohesive properties.

894 New insights into high-temperature polymer electrolyte membrane fuel cells using electron microscopy techniques

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The increasing need for energy supply and reduction of environmental pollution has intensified the research on fuel cells during the last years. One promising candidate are high-temperature polymer electrolyte membrane fuel cells with polybenzimidazole (PBI)-based membranes. The performance and degradation behavior can be improved by adding inorganic fillers into the membrane and by replacing the commonly used carbon support by oxides. In our work we analyze the different components as well as whole membrane-electrode assemblies in depth by different TEM techniques and correlate the results to the synthesis routes and performance. We investigated PBI-based membranes containing nanoparticles which were formed in-situ by a sol-gel reaction [1]. The TEM studies revealed that the nanoparticles are homogeneously

distributed and consist of amorphous silica. EDS linescans showed that neither P nor Cl are segregated to the particle – PBI interface. With these findings we were able to explain the properties as well as the degradation behavior [1]. Tungsten oxide support materials were analyzed after different operation times and modes including start-stop-cycles [2]. After deposition three dimensional networks of few nanometer thin, crystalline Pt rods form on the WO_{3-x} support with sizes of up to about 1 μm . The Pt rods decrease in size during operation and Pt is diffusing into different areas of the fuel cells, including the membrane [2].

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895 Influence of different pH and fluoride addition on the corrosion behavior of the sintered CoCr alloy ceramill sintron compared to the cast alloy girobond Nb

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Objectives: In addition to conventionally casting, dental metallic framework can be manufactured using different CAD-CAM technologies (selective laser melting, milling, sintering). The milling of porous CoCr blanks followed by sintering under protective gas is a new 2012 introduced dental technology called Ceramill Sintron. For this new material so far, there exist few studies on the corrosion behavior. The aim of this study was to investigate the influence of different pH values as well as fluoride additions on the corrosion behavior of the sintered CoCr alloy compared to the cast condition by electrochemical corrosion measurements. **Materials & Methods:** The CoCr alloys Ceramill Sintron (66Co-28Cr-5Mo) and Girobond Nb (62Co-25Cr-5Mo-5W-1.2Si-0.3Ce) were used. From each alloy 6 samples were prepared without further subsequent heat treatment. As electrolytes 0.9% saline solutions with pH 5, pH 3 and 0.02 M NaF were prepared. After grinding and cleaning the samples, electrochemical tests were done according to ISO 10271 consisting of OCP-measurements over 2 hours followed by anodic polarization starting from -150 mV~E_{corr} to 1 V. **Results:** The most pronounced differences between both alloys could be seen among the parameters E_{corr} and i_{corr}. The i_{corr} values at pH 5 and pH 3 were 86.9 ± 4.9 and 85.6 ± 14.6 for Ceramill Sintron and 60.7 ± 8.8 and 12.9 ± 1.5 nA/cm² for Girobond Nb, which were increased by adding fluoride to 6 and 29% as well as 25 and 74%. **Conclusion:** Based on this study Girobond Nb showed a good acidic resistance at low pH-values. Ceramill Sintron showed stable corrosion resistance in all electrolytes.

896 Manganese effect on Q&P CMnSi steels

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The so called Quenching and Partitioning (Q&P) steels belongs to a family of third generation of Advanced High Strength Steels where increase strength can be attained at significant

ductility values (as high as 1200MPa and 20% respectively). Their chemical composition must be carefully fitted to obtain the desired microstructure (absence of precipitates and bainites) at the industrial cooling facilities. The present study is focused on analyzing the effect of Mn amount on two experimental steel compositions, specially designed for Q&P, 0.15C-2.5Mn-1.5Si and 0.15C-3Mn-1.5Si without significant contribution of Al. 2-Step Q&P thermal treatments were performed at laboratory scale in a quenching dilatometer Bähr DIL805A/D. The fractions of retained austenite and amount of Carbon in the austenite were evaluated by X-ray diffraction techniques. The mechanical properties of the Q&P samples were evaluated, and they showed a wide range of strength, ductility and strain hardening values from 1000 to 1200 MPa, from 15 to 22 % and from 0.05 to 0.17, respectively. The 0.15C-3Mn-1.5Si steel shows systematically the largest mechanical values. Based on these properties an optimal Q&P parameters, for both compositions, were selected to perform the treatment on larger area samples, A50, in a salt bath installation. All the mechanical properties and % of retained austenite were compared with literature and discussed as a function of the Q&P parameters and treatment processes.

897 Liquid composite moulding: A widely used group of FRPC processing techniques, but still a challenging topic

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Liquid Composite Molding (LCM) techniques are widely used technologies in order to manufacture fiber reinforced plastic (FRP) composites using near-net-shaped preforms consisting of single reinforcements, e.g. woven textiles or multiaxial fabrics. All LCM process variants have in common to impregnate and saturate dry reinforcing structures with a liquid thermoset resin system. The challenge during LCM process development and mold designing is the prevention of potential error sources for further FRP production. Race tracking zones and air inclusions are two major issues which need to be avoided in order to ensure an excellent FRP quality. No matter of the processing technique the knowledge about preform transmissibility, i.e. permeability, to the liquid flow during the saturation phase is of major importance. The knowledge about the filling and flow behavior during FRP processing is responsible for the process efficiency and process success. In-plane and out-of-plane permeability characterization is of great interest. Especially industry is interested in precise permeability values for numerical mold filling simulations in order to support the process development and the mold design. Industrial work is also carried out for filling strategies and textile development as well as textile improvement. The paper presents different LCM processing techniques and discusses the advantages and disadvantages as well as the linked challenges during FRP processing. Furthermore, the different permeability characterization methods and systems and moreover influencing factors on the filling behavior are presented. Finally the significance of accurate and reliable permeability values according to numerical filling simulations and their validity are discussed.

898 Feedstock development for enhanced control of the direct ink write additive manufacturing process

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Direct Ink Write (DIW) of elastomeric materials is an additive manufacturing (AM) process that allows for the manufacturing of soft structures with a broad range of properties. Previous work manipulating the internal structure of parts printed using commercial silicone adhesive has demonstrated modification to the part's stress/strain response. For instance, parts have been built that respond with a negative stiffness in shear while under compression by layering the filaments in a cubic structure that becomes unstable and buckles at high enough strains (Duoss et al. 2014). Maturation of this technology requires the development of an AM specific feedstock that has well understood processing parameters. We focus on the resin development, where the chemistry and printed structure can work together in defining the final part's properties. The feedstock rheology must be defined by two critical characteristics; the resin must be shear-thinning enough to be extruded through a ≤ 600 micron nozzle, but possess a yield stress that will prevent the part from flowing under its own weight before curing. Our resin is developed by starting with a PDMS of known molecular weight and structure, to which a networking filler can be added until the material is made printable. To characterize the resin's rheology, a parallel disk rheometer was employed to identify the criteria required for a successful ink. Once printed, a suite of analytical techniques are used to probe the interaction of the chemistry, fillers, and structure, and their impact on the material's mechanical and thermal response.

899 Simulation of roll bonding and further rolling of roll bonded material including bond strength development

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Roll bonding is one of the most used method for a production of metallic materials compounds. Beside surface treatment and several annealing steps the main parts of this technology are the processes of roll bonding and further rolling of the layered structures. On the one hand, differences in mechanical und thermal properties of the alloys used for each layer are the reason for several technological problems. On the other hand, small tolerances are expected concerning variations in layer thickness, bond strength and the maximum allowed residual stresses. The influence of adsorption layers and surface roughness is known from industrial experience and laboratory trials. A general theoretical investigation of their influence on optimum process conditions are still missing. In the given presentation a multi - scale cladding and bond strength model is presented, which is based on the N- layer model LayCladd for material flow and for bond strength computations on film theory of adhesion, contact and fracture mechanics as well as thermodynamically considerations concerning surface tension and the effect of adsorption layers on it. The used equations are an extension of the model presented by Bay and Zhang. Based on a coupled computation of bonding process and bond strength development parameter identification is performed concerning the dependence of the final bond strength on initial material state and process conditions. Several aspects of the bond strength model were

evaluated in laboratory trials. A comparison of theoretical and experimental results is given.

900 Experimental and theoretical EELS study of rhenium borides

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Rhenium borides and other group 6 borides are extensively studied as many of them have interesting physical properties [1, 2]. One approach to understand the structure–property relations in these compounds is to systematically characterize the interatomic bonding.

Here, we studied the boron K–edge of Re_3B and ReB_2 by electron energy loss spectroscopy, EELS, and compared the results to those obtained from density functional theory–based calculations. Samples were synthesized either by arc–melting, in an induction furnace or in a multi–anvil press at 1373 K and 12.5 GPa. Powder diffraction was employed to ensure that the samples were single phase. Samples were mechanically ground and placed on lacey carbon on a copper grid. We used a FEI Tecnai F30. From the experimental spectra, a background was subtracted using the Gatan Digital Micrograph software. The experimental data were compared to theoretical spectra obtained using density functional theory [3]. The agreement between theory and experiment is very good and the distinct shapes of the K–edges are correctly reproduced by the DFT calculations. This then allows to predict the K–edge spectrum for Re_7B_3 and to quantitatively interpret the experimentally observed data. Acknowledgement

FS and DS are grateful for financial support from the DAAD. We thank LINAN for access to electron microscopy facilities and Ignacio Becerril and Ana Iris Peña for technical support at the IPICYT. References: [1] Friedrich et al., *Materials*, 4:1648–1692, 2011. [2] Juarez-Arellano et al., *Solid State Sci.*, 25:85–92, 2013. [3] B.Winkler et al., *J. Phys. Condens. Matter*, 25, 2013.

901 Artificial extracellular matrices based on cross-linkable polysaccharides for tissue regeneration

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The extracellular matrix (ECM) of mammalian cells represents a complex biopolymer network providing mechanical support to the cellular constituents and regulating many important biological functions like cell growth, wound healing, and fibrosis. Water-soluble polysaccharides and especially glycosaminoglycans (GAG) are versatile starting materials to generate gel-like materials mimicking the native ECM. Chemically or photo-chemically initiated network formation and structuring is a promising approach to create artificial ECM usable in soft tissue reconstruction and drug delivery. Based on dextran, hyaluronan, and chondroitin sulfate biopolymers we prepared polysaccharide and GAG derivatives with defined degree and pattern of crosslinkable substituents. In addition, synthetic routes to cross-linkable GAG with multiple substituents (e. g. both growth factor sequestering sulphate and cross-linkable (meth) acrylate groups) were elaborated. The synthesized macromers were structurally

characterized using conventional analytical techniques (IR, NMR spectroscopy, molecular weight determination using GPC, elemental analysis). Hydrogel formation was studied using different radical forming initiators and the resulting gels were tested with regard to their mechanical properties and their cytocompatibility by an established live/dead viability test and a WST-1 cytotoxicity assay. Cytocompatible hydrogels of sufficient mechanical stability and with controllable rate of biodegradation could be obtained. Different scaffold fabrication techniques including light-based additive manufacturing processes and cryogel formation have been used to evaluate the potential of these polysaccharide (meth) acrylate macromers to manufacture tailored hydrogel scaffolds able to provide, similar to the native ECM, both support and biological activity to cells.

902 Influence of different welding processes on the mechanical properties of structural steel S960

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High-strength structural steels are widely used in many industrial sectors, such as shipbuilding, offshore constructions, cranes and pipelines. They offer high strength with sufficient toughness and good forming capabilities. Regarding the weldability of S960 and high-strength low-alloy steels (HSLA) in general there is a lot of ongoing research. Especially the toughness properties of the weld region, i.e. weld metal (WM) and heat affected zone (HAZ) are of special interest. The microstructure of the HAZ is mainly influenced by the heat input of the welding process, while in the WM the used filler metal is also of great importance. In the present work different fusion welding processes as well as different filler metals are compared on 8 mm thick sheets of S960TM structural steel. The welding processes include electron beam welding (EBW), laser beam welding (LBW), laser hybrid welding (LHBW), plasma welding (PW), tungsten inert gas welding (TIG) and gas metal arc welding (GMAW). The influence of different energy densities and the use of inert or active shielding gas on the microstructure and mechanical properties, especially toughness, is discussed. For mechanical characterization tensile, impact and hardness testing was carried out. The microstructure was analyzed via light- optical microscopy using different etching techniques.

903 Miniaturized laser power sensor via rapid prototyping

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The power of lasers is subjected to considerable temporal changes, especially for high-power lasers for material processing. For the setting of processing parameters, it is therefore necessary to know the currently available optical power as precisely as possible. This has generated the need for laser power sensors. In this work, thermocouples are used, which are processed with cleanroom technology on flat substrates. For this purpose, a lift-off process line has been developed for the purposes of rapid prototyping based on photolithography, i.e. rapid

phototyping. The goal here was a cost-effective process route. The result is a functioning, rapid phototyping process chain, enabling to manufacture thin film structures as thermocouples fast, cost-effective, and with minimal efforts regarding media, time and energy. Other than vacuum and a UV light source no further media are required. Moreover, the mask size is flexible. In the process, structural widths of up to 50 μm and 250 μm height could be manufactured reproducibly. Just the positions of the structures on the mask were subject to fluctuation, i.e. the positions of the structures on average vary 5 μm horizontally and 30 μm vertically. For the purposes of rapid prototyping, this process chain is - in comparison to retail / serial process routes - very flexible for different mask designs, sufficiently accurate for manufacturing reproducible prototypes, and low cost avoiding expensive process materials.

904 Development of the strongest welding consumables

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The need of constructions with reduced weight and the requirement of carrying higher loads increase the demands on high strength steels. Developing high strength filler materials with acceptable toughness is an essential task for the realization of these ultra-high strength steel designs. The development of filler metals has reached now its limitation at a yield strength of 960 MPa. In order to increase the strength and reach an adequate toughness level the usage of micro-alloying elements is considered as an alternative concept compared to the conventional solid solution strengthening. These micro-alloying elements can influence grain growth behavior during solidification and cooling and increase therefore the toughness. Furthermore they can promote the formation of precipitates which result in a strengthening effect. For evaluation of the influence of different alloying elements, trial alloys of metal-cored wires were produced and tensile and notched impact samples of all-weld metal were machined. The results are presented and the most effective alloying concept for increasing the strength and maintaining the toughness is shown. Furthermore first results from comprehensive microstructural characterization are presented. Thereby atom probe tomography was applied in order to reveal the atomic-scale microstructure. These investigations should help to understand the structure-properties relationship of high strength welding consumables.

905 Influence of corrosive conditions on the mechanical performance of flow drill screw joints between light metals

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The major research project Next Generation Car (NGC) concentrates the expertise of the German Aerospace Center (DLR) in ground based traffic for a sustained and safe mobility of tomorrow. A tool for the automatic selection of joining technologies for multi material design (MMD) is being developed within the NGC project. A particular function of this tool is to consider the increased demand MMD structures place on corrosion resistance. In this paper, the influence of corrosive conditions on the mechanical performance of Flow Drill Screw (FDS)

joints is investigated in greater detail. Different combinations of light metals such as high strength/stainless steel, aluminium and magnesium alloys served as the test material. The joint strength, under quasi-static and cyclic loading, of FDS joints was measured before and after six weeks of corrosion climate change testing. Furthermore metallographic sections of the samples were compared in order to evaluate the stage of surface, galvanic and crevice corrosion. To classify the effect of progressing corrosion on the mechanical properties of FDS joints the following factors are taken into account: corrosion resistance of the materials, joining parameters, geometry of the joint, as well as corrosion protection by coating. For all material combinations there is an apparent change in both the fatigue strength and the failure behaviour after corrosion testing. These results confirm that environmental conditions have a significant impact on the structural durability of MMD structures during the lifetime of a vehicle.

906 Grain boundary hardening and dislocation pile-ups: The effect of misorientation

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A classical way of explaining the Hall-Petch relationship is the analysis of a double dislocation pile-up along the grain diameter. The solution of a singular integral equation describing the stress field of infinite straight and parallel dislocations leads to a $d_g^{-1/2}$ relationship. If slip is transmitted from one grain to the next upon reaching a critical stress level characteristic of the grain boundary, the Hall-Petch formula is recovered. This work reviews the problems associated with this approximation and provides calculations on the Peach-Köhler force between slip systems in misoriented grains. It has been pointed out that the approximation of the problem by an array of infinite parallel dislocations in a single slip plane perpendicular to the grain boundary is overly simplified. Large deviations from the classical result can be found by making small modifications to the simplifying assumptions. It has been shown that if various pile-ups in parallel slip planes are considered, the connection of the stress field with the Hall-Petch relationship is lost. Also, by considering the shear required to move the dislocations into their positions, the model breaks down for medium to small grain sizes. The Peach-Köhler force, which activates slip in the neighbouring grain, shows a strong dependence on the misorientation between the grains and, due to the inclination between the slip systems, generates localised stress concentrations which show strong differences from what is considered in the parallel pile-up model but coincide with available TEM observations of slip transfer at the boundaries.

907 Understanding chemical reactions of small molecules at extreme conditions by means of high pressure pair distribution function analysis

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Chemical reactions at high pressures and temperatures are of interest because extreme conditions can lead to reaction pathways which result in novel compounds with interesting properties [1]. Reactions of molecules from the system C-H-N-O are of particular interest for

example in astrophysics, condensed-matter physics or chemistry [2]. Products of those chemical reactions are not necessarily crystalline. The analysis of the pair distribution function (PDF), which is obtained from powder X-ray diffraction data, is a powerful method for the determination of structural parameters of disordered, amorphous and nanocrystalline materials [3]. In this study, the PDF and spectroscopic techniques were employed to study urea and its derivative carbonylhydrazide at extreme conditions. We induced chemical reactions in laser heated diamond anvil cells (DAC) at pressures up to 16 GPa. The colourless reactants transformed to coloured (reddish and grey) phases, which showed strong luminescence. PDFs of the products were obtained from X-ray diffraction data measured at the Extreme Conditions Beamline P02.2 (PETRA III, Hamburg, Germany) after heating as well as during pressure increase and decrease in order to study the stability field of the products. The PDFs changed significantly upon opening the DAC indicating that the reaction products were unstable at ambient conditions. The authors gratefully acknowledge financial support from the DFG (project RA 2585/1-1) and the BMBF (project 05K13RF1). [1] R. Bini, M. Ceppatelli, M. Citroni, V. Schettino, *Chem. Phys.*, 398, 262 (2012). [2] F. Datchi and G. Weck, *Z. Kristallogr.*, 229, 135 (2014). [3] T. Proffen and H. J. Kim, *J. Mater. Chem.*, 19, 5078 (2009).

908 Phase decomposition of a single-phase nanocrystalline CoCrFeMnNi high-entropy alloy

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An equiatomic CoCrFeMnNi high-entropy alloy was subjected to severe plastic deformation using high-pressure torsion. After this process the alloy remained a true FCC solid solution down to the atomic scale with a grain size of ~ 50 nm leading to an increase in tensile strength to 1950 MPa and a hardness of 520 HV. Isochronal (1 hour) annealing treatments led to a hardness increase with a maximum hardness of 630 HV at about 450 °C before softening set in at higher temperatures. Isothermal anneals at 450°C revealed a hardness rise to a maximum of 910 HV after 100 hours. In order to clarify the unexpected annealing behavior, comprehensive microstructural analyses of selected microstructural states using transmission electron microscopy and 3-dimensional atom probe tomography were employed. The changes in mechanical properties could be related to the formation of nanoscale phases and clusters embedded in the nanocrystalline high-entropy alloy matrix stabilizing the microstructure against grain growth. Further investigation of a nanocrystalline equiatomic CoCrNi alloy in regards to thermodynamical stability, mechanical properties and microstructural features during annealing treatments was also performed. A comparison of the results gives new valuable insights into the phase stability of single phase high entropy alloys.

909 High-pressure synthesis of skutterudite-type thermoelectric materials

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New thermoelectric materials $Mm_xCo_4Sb_{12}$ (Mm =mischmetal) were synthesized using high-pressure techniques. Although the binary skutterudite compound $CoSb_3$ shows excellent thermoelectric properties, the lattice thermal conductivity is too high. However, the filling of the voids partially in the binary skutterudite structure with rare-earth (R) ions can reduce the lattice thermal conductivity. The R ions are located inside the oversized cages, formed by twelve Sb ions, and the undersized ions are believed to show random motion around the equilibrium positions. Thus, it produces a large phonon scattering, reducing the lattice thermal conductivity. Owing to the reduced thermal conductivity, partially filled skutterudite compounds $R_xCo_4Sb_{12}$ show excellent thermoelectric performance. Recently, however, the use of rare earth metals has serious problems about cost and supply amount. Therefore, we focus on Mm as the void-filling ions. Mm is an alloy of rare earth elements in various naturally occurring proportion. Mm is low-cost compared with individual rare earths because the extraction processes is unnecessary. A preparation of high-quality samples for the antimony-based skutterudite compounds is quite difficult because the high vapor pressure of Sb and impurity phases tend to be formed easily. High-pressure synthesis technique is one of the useful methods to prepare high quality samples of skutterudites. Furthermore, the limit of R filling in $CoSb_3$ can be expanded. In this study, we have tried to synthesize $Mm_xCo_4Sb_{12}$ samples with high filling rate of Mm under high temperature and high pressure by means of a Kawai-type multi-anvil high-pressure apparatus.

910 Materials tuning of titania nanotubes for enhancing physical-photochemical multifunctions

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Titania nanotube (TiO_2 nanotube, TNT), which has nanometer-scale tubular structure with a diameter of around 10 nm, has excellent physicochemical and photochemical properties due to the synergy of its wide-bandgap semiconductor properties, unique nano- and crystalline structures. It is, however, responsible to UV light due to its bandgap energy of around 3.3 ~ 3.4 eV. Development of visible-light responsible TNTs is thus desired to enlarge the practical application, which is one of or motivations for this research. We have modified the TNT to realize high performance environmental and/or energy nanomaterials. One of our strategies lies in doping elements to TNT. Metal-doped TNTs have been synthesized by a simple solution processing based on chemical treatment of raw TiO_2 powders in alkaline solution. Various ions such as Cr, Sm and Ru have been doped TNTs. Obtained M-doped TNTs exhibited optical adsorption peaks in the visible light region, and the bandgap energy decreased depending on the doped elements. Organic dye removal test in aqueous solution revealed that the small amount of metal doping enhanced the visible light photocatalytic performances. On the other hand, we have recently succeeded to synthesize visible light responsible nanostructured titania by simple chemical treatment of TNT at room temperature. TNT surfaces have been modified through the chemical treatment, and resulted in decrease in bandgap energy down to 2.4 eV.

The modified TNT exhibited excellent photocatalytic degradation performance for organic dye under the visible light (>420nm). Detailed materials processing, nanostructures, surface chemistry and photochemical properties will be discussed.

911 Quantitative microstructural analysis for age-hardenable Cu-based alloys using extraction technique

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Extraction technique has been employed to quantitatively analysis the precipitation behavior of age-hardening Cu-based alloys, such as Cu-Ti and Cu-Ni-Sn alloys. When the alloys were submerged in a nitric acid solution at 273 K, only the Cu solid solution matrix was dissolved chemically to leave precipitates as insoluble residue. Subsequent structural and compositional analyses of the insoluble residue directly reveal the fraction and composition of the precipitates: in the case of Cu-4 mol% Ti alloys aged at 723 K, fine needle-shaped α -Cu₄Ti particles (a tetragonal structure), with Ti content of 36 mol%, form continuously in the Cu matrix phase in an initial stage of aging. During prolonged aging, coarse cellular components composed of the terminal Cu solid solution and the stable β -Cu₄Ti (orthorhombic), with the Ti content of 21.0 mol%, form in grain boundaries, consuming α -Cu₄Ti particles. Thereby, the amount of α -Cu₄Ti particles in the specimen shows a maximum of approximately 1.7 vol% at 24 h aging, and then the amount of β -Cu₄Ti particles increased instead and reached to more than 18 vol% after 480 h. Based on the extraction technique, a precise partial phase-diagram and TTT diagram for the Cu-based alloys can be proposed.

912 Friction surfacing of Alloy 625 on AISI 4140: Effect of process parameters on coating geometry and microstructure

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Friction Surfacing is a solid state coating technique used to add material onto a surface as a repair method or to realize material combinations with different properties of the surface and the bulk of a component. The process poses several advantages for dissimilar configurations since melting of the processed materials is avoided. In this study, Alloy® 625 was deposited by Friction Surfacing onto carbon steel AISI 4140 substrate using various process parameters aiming to evaluate the effect of process parameters on coating morphology, hardness and microstructure. In addition, a comparison between original stud microstructure and the one resulting from the Friction Surfacing process is presented. The proposed material combination is destined to be used in aggressive corrosion environments, where due to the substrate material's low corrosion resistance, coating integrity and thickness are of utmost importance. The results show that increasing traverse speeds generated thinner coatings, whereas increasing rotational speeds generated coatings with lower width. The stud material suffered full dynamic recrystallization and formed a fine-grained coating with submicron grains and increased

hardness near the coating-substrate interface. These results are related to the non-homogeneous strain distribution during the process which resulted in regions with different grain morphologies, confirmed by EBSD analyses. In addition, FS led to a microstructure free of precipitates known to be detrimental and coatings with improved thickness and ductility are expected. It is possible to conclude that FS is a potential technology for depositing nickel alloys with good interface quality and improved mechanical properties.

913 Development of joining method for zircaloy and SiC/SiC composite tubes by using diode laser

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Silicon carbide fiber reinforced silicon carbide composite (SiC/SiC composite) is one of the promising candidates for various next generation nuclear components such as fuel cladding for the fission reactor and blanket structures for the nuclear fusion reactor due to many superiorities: high-temperature thermos-mechanical and chemical stability, radiation tolerance, etc. As for the fuel cladding for the light-water reactor, SiC/SiC composite is expected as a replacement of Zircaloy, where it is necessary to develop the method for plugging SiC/SiC composite tube. In this study, as one possible design, SiC/SiC composite and Zircaloy tubes were joined based on the caulking method. The inner face of Zircaloy tube and the outer surface of SiC/SiC composite were machined in order to fit two tubes, and then the diode laser beam irradiation was circumferentially applied to the outer surface of Zircaloy tube. In addition, in order to improve the weldability of SiC/SiC composite and Zircaloy, the nano-powder of titanium was inserted into a gap between SiC/SiC composite and Zircaloy. Although only the surface of Zircaloy tube was melted by the diode laser irradiation, all proportional solid solution of zirconium and titanium was produced at the inner face of Zircaloy tube and then SiC/SiC composite and Zircaloy was partially well joined. The microstructural observations will be discussed in order to examine an airtightness of this joint.

914 On the strength effects in hydrogenated palladium subjected to HPT processing

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It has been recently shown that in hydrogenated SPD (severe plastic deformation) processed Palladium [1] the densities of lattice defects (vacancies and dislocations), were significantly higher than in case of non-hydrogenated Pd. Very recently the authors showed that this enhanced densities of defects cause a tremendous increase in strength [2]. Although the results [2] tend to be in line with previous investigations by Chen et al [3] concluding that all the strength increase arises from increased dislocation density, the strength part from vacancy agglomerates could not be fully excluded. In the present study, isochronal micro hardness measurements were undertaken to provide both a selective defect annealing as well as strength changes associated with the annealing of defects induced by HPT (high pressure torsion) in

hydrogenated Pd. The concentrations of annealing defects were measured by a combination of XPA (X-ray Bragg Profile Analysis) and DSC (differential scanning calorimetry) [4]. The lecture presents first results on specific strength contributions arising from vacancy agglomerates and dislocations, and gives ideas how to understand them. Financial Support by Austrian Science Fund (FWF): project number T512-N20 is gratefully acknowledged. [1] D. Setman, et al. Materials Science Forum 584-586 (2008) 355–360. [2] K. Werbach, et al. HySEA Conference, Oct. 2014, Rio (Brasil) [3] Y.Z. Chen, et al. Scripta Mater. 68 (2013) 743-746 [4] D. Setman, et al. Metall. Mater. Trans. A, 41A (2010) 810-815;

915 Spatio-temporal behaviors of atmospheric-pressure dielectric barrier discharge plasma jets for reactive interactions with materials

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Non-equilibrium atmospheric-pressure plasmas have attracted great interests as low-temperature plasma sources for a variety of materials processes including nano-surface modifications and nano-materials production in liquid as well as biological applications and medical treatments referred to as "plasma medicine". For development of innovative science and technologies for nano-materials processes and plasma medicine, it is of great significance to understand basic characteristics in terms of spatio-temporal behaviors and reactive particle generation in atmospheric-pressure plasmas. So far a series of investigations have been carried out for investigations on frequency dependence of atmospheric-pressure discharge generation in a wide range of discharge-power frequency from kHz region with high-voltage DC pulses to VHF. In this presentation, these studies are extended further to investigation of basic characteristics regarding spatio-temporal behaviors of atmospheric-pressure plasma jet and reactive interactions with materials. Dynamic behaviors of atmospheric-pressure dielectric-barrier discharge plasma jets have been investigated using an intensified CCD (ICCD) camera, in which O and He spectrum lines were selectively detected through optical band-pass filter. The fluid dynamic behaviors of the plasma jet have further been studied using Schlieren method for investigation of the gas-flow effects on discharge and correlation of with radical formation in liquid. Acknowledgements: This work has been carried out partly under the auspices of The Grant-in-Aid for Scientific Research on Innovative Areas "Plasma Medical Innovation" from The Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

916 Anodization behaviour of friction stir processed aluminium surface composites

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Multiple-pass friction stir processing (FSP) was employed to impregnate TiO₂ (rutile) powders into the surface of an Aluminium alloy. The surface composites of Al-TiO₂ were then anodized in a sulphuric acid electrolyte. The effect of anodizing parameters on the resulting optical

appearance was studied. Microstructural and morphological characterization was performed using scanning (SEM) and transmission electron microscopy (TEM), and X-ray diffraction (XRD). The surface appearance was analysed using an integrating sphere-spectrometer setup which measures the diffuse and total reflectance of light from the surface. Compared to samples without TiO₂, surface appearance after anodizing of samples with TiO₂ changed from dark to greyish white upon increasing the anodizing voltage. This is attributed to the localized microstructural and morphological differences around the TiO₂ particles incorporated into the anodic alumina matrix. The TiO₂ particles in the FSP zone were partially or completely amorphized during the anodizing process, and also electrochemically shadowed the anodizing of underlying Al matrix.

917 Kinetics of submicrocrystalline structure formation in a Cu-Cr-Zr alloy during large plastic deformation

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The grain refinement and kinetics of submicrocrystalline structure formation in a Cu-0.3%Cr - 0.5%Zr during large plastic deformation were investigated. The fraction of high-angle boundaries and the fraction of ultrafine grains were used to estimate the kinetics of grain refinement and submicrocrystalline structure evolution during large plastic deformation. The multidirectional forging (MDF), equal channel angular pressing (ECAP), and high pressure torsion (HPT) were used as methods of large plastic deformation. Comparative analysis showed that the grain refinement process occurred faster during HPT in comparison with MDF and ECAP. The fraction of ultrafine grains achieved 0.8 after 3 HPT turns, while the one reached 0.35 and 0.05 after MDF and ECAP to a total strain of 4, respectively. The modified Johnson-Mehl-Avrami-Kolmogorov equation could be applied to determine the kinetics of grain refinement in copper alloy during large plastic deformation as a function of true strain.

918 The effects of interfacial heat transfer coefficient on the microstructure of high-pressure Die-cast magnesium alloy AM60B

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This paper describes the details of a quantitative experimental and numerical study on the influence of solidification conditions including the apparent interfacial heat transfer coefficient (IHTC) between the die and solidifying metal. Multiple runs of the commercial casting simulation package, ProCAST™, are used to model the mold filling and solidification events employing a range of IHTC values. The simulation results are used to estimate the centreline cooling curve at various locations through the casting. The centreline cooling curve, together with the die temperature and the thermodynamic properties of the alloy are then used as inputs to compute the solution to the Stefan problem of a moving phase boundary, thereby providing the through-thickness cooling curves at each chosen location of the casting. Finally, the local cooling rate is used to calculate the resulting grain size and skin thickness via previously established relationships. A comparison of the predicted and experimentally determined grain

size profiles enables the determination of the apparent IHTC, which, in this study, was approximately $12000 \text{ W/m}^2\cdot\text{K}$. Additional useful observations from the numerical study suggest that the IHTC has a significant influence on the skin thickness and grain size in both the skin and core regions of the casting, while the effect of die temperature is limited to influencing the skin grain size only.

919 Concept of nano technology in ayurveda

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Archeological sites in India have provided abundant information about the prevalence and use of metals in ancient times. Ayurveda, the traditional system of medicine in India likewise used metals and minerals judiciously in the form of medicine. The therapeutic utility of these substances, especially in dire conditions, was attributed to its instant action, lesser posology, better palatability and the ability to use it across patients of variable constitution. The popularity of these medicines was such that over time, it evolved and flourished as a separate specialization in Ayurveda viz. Rasa Shastra. Sequential complex procedures have been described to convert the metals and minerals into simple forms that can be easily digested into the human body. The procedures include cleansing (Shodhana) killing (Marana) etc. Several studies have demonstrated that these processes change the crystalline structure, reduce particle size and increase the surface area and bioavailability. Hence they can be administered in lower dose and a direct action on the target cell may be expected. It is believed that metals that have been subjected to appropriate procedures are finally converted as Nanoparticle. Research has thus far revealed few of the strengths of such nanoparticle use in Ayurveda. However, there have been conflicting reports of toxicity or poisoning by metal and mineral drugs of Ayurveda. Concerted efforts by multiple agencies, Institutes and policy makers is needed to review, experiment and analyse this facet of Ayurveda that has immense potential in optimising health care delivery.

920 Improving intergranular corrosion and stress corrosion cracking resistance in a high-nitrogen austenitic stainless steel through GBCD optimization

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Grain boundary engineering (GBE) is a practice of improving resistance to grain boundary failure of materials through increasing the proportion of low Σ coincidence site lattice (CSL) grain boundaries (special grain boundaries) in the grain boundary character distributions (GBCD). The GBCD of Fe-18Cr-18Mn-0.63N high-nitrogen austenitic stainless steel cold rolled and then annealed were analyzed by electron back scatter diffraction (EBSD), meanwhile, the effects of GBCD optimization on the intergranular corrosion (IGC) and the intergranular stress corrosion cracking (IGSCC) resistance were investigated by oxalic acid electrolytic corrosion experiment, ferric sulfate-sulfuric acid test and C type ring test. The results show that the optimization process of GBE in the conventional austenitic stainless steel cannot be well applied to the present high-nitrogen austenitic stainless steel. The fraction of low Σ CSL grain boundaries could increase from 47.3% for the solid solution treated high-nitrogen

austenitic stainless steel specimen to 83.3% for the specimen cold-rolled by 7% and then annealed at 1423 K for 10 min. The second phase precipitation obviously decreases and the IGC and IGSCC are remarkably improved in the specimens after GBCD optimization. These special boundaries of high proportion effectively interrupt the connectivity of general high angle grain boundary network, thus achieving GBCD optimization and improving the IGC. The specimen owning special boundaries of high proportion is resistant to IGC, thus inhibiting the initiation of stress corrosion cracks for the high-nitrogen austenitic stainless steel.

921 Multiaxial stress states of Zr-base bulk metallic glasses by elastic-plastic finite element analyses

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Multiaxial yield condition of Zr-base metallic glasses was first investigated by elastic-plastic finite element analyses. The proposed constitutive law which is based on Drucker-Prager (DP) yield function and the free volume theory has two kinds of parameter set at room temperature and a high temperature close to glass transition temperature by fitting to the experimental data. Regardless of the temperature, the coefficient of mean stress dependence in DP criterion is obtained as 0.09 and the elastic limit as the yield point of metallic glass provides a good agreement to the prediction by DP condition. The shear banding behaviors around the defects such as a void or notches were then simulated under plain strain condition and it was found that crossing between two shear bandings coming up at the very narrow base between two deep notches was contributed to release the rapid stress drop after yielding. The block laminated by the amorphous and the crystalline phases was also calculated and the nucleation of shear bandings in both layers were strongly interrupted each other.

922 Kinetic simulation of long-term precipitate evolution in heat-resistant alloys

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There is an urgent need to improve the thermal efficiency of fossil fuel power plants by raising the operation temperature and pressure in order to reduce CO₂ emission as well as the cost of fuel. Plant operation at higher temperatures inevitably requires the development of heat-resistant alloys with a higher creep strength. There has recently been an increasing interest in applying advanced heat-resistant alloys to fossil fuel power plants in order to raise their operation temperature. The important role of precipitation in the achievement of good creep properties of heat-resistant alloys has long been recognized. One of the most effective ways for improving the creep properties is to uniformly distribute fine precipitates with a good long-term stability at elevated temperatures. The experimental investigation of the long-term precipitate evolution behavior in heat-resistant alloys have not often been performed, although it is important in understanding the creep properties. In addition to experimental approaches, there have recently been a few attempts to simulate the precipitation kinetics in heat-resistant alloys. The purpose of this study is to simulate the long-term precipitate evolution in various heat-

resistant alloys such as ferritic/martensitic and austenitic steels and nickel alloys using a numerical model based on the classical nucleation theory and evolution equations derived from the thermodynamic extremum principle that maximizes the dissipation rate of the total Gibbs energy of the system. The simulation results, such as the precipitation sequence and the precipitate size, will be compared with experimental observations.

923 Crystallographic features of states near the state boundary between the C- and A-type orbital-ordered states in $\text{Sr}_{1-x}\text{R}_x\text{MnO}_3$ (R=Nd, Sm)

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The simple-perovskite manganites $\text{Sr}_{1-x}\text{R}_x\text{MnO}_3$ (R=Nd, Sm) have been reported to exhibit fascinating electronic states for e_g electrons in Mn ions. In $\text{Sr}_{1-x}\text{Nd}_x\text{MnO}_3$, there exist the C-type orbital-ordered (COO) state for $0.20 \leq x \leq 0.38$ and the A-type (AOO) state for $0.40 \leq x \leq 0.48$. Their crystallographic feature is that, in addition to the Jahn-Teller distortions for C- and A-type orbital orderings, the R_{25} -type rotational displacement of oxygen octahedra is involved in their crystal structures. However, the detailed features of a state change between these two states have not been understood sufficiently. Thus, the crystallographic features of prepared $\text{Sr}_{1-x}\text{R}_x\text{MnO}_3$ samples with $0.4 \leq x \leq 0.49$ have been examined by x-ray powder diffraction and transmission electron microscopy. In $\text{Sr}_{1-x}\text{Nd}_x\text{MnO}_3$, for instance, x-ray powder diffraction profiles measured in our experiment showed that the tetragonal $I4/mcm$ and orthorhombic $Imma$ states were, respectively, present for $0.40 \leq x \leq 0.46$ and for $0.46 \leq x \leq 0.49$ at room temperature. From obtained electron diffraction patterns, the $I4/mcm$ state could be identified as the COO state with a nanometer-scaled banded structure. The banded structure was found to consist of an alternating array of two tetragonal bands with different c/a values. On the other hand, the $Imma$ state involves only the R_{25} -type rotational displacements about two $\langle 100 \rangle_c$ directions in the pseudo-cubic notation. Because there is the same rotational-displacement pattern in the AOO state, the $Imma$ state may be regarded as its precursor state.

924 Fabrication of high porosity mullite foams and their properties

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Already, we are developed high porosity alumina foam. However, alumina has high thermal conductivity about 36W/mK at room temperature, and it need to achieve to high porosity to decrease thermal conductivity to for application of refractory bricks. Therefore, high porosity mullite refractory brick is developed using GS (Gelation of Slurry) method that is already developed for production of high porosity metal foam. Applying this method to production of mullite foams, the ceramics foams from 90 to 97.% porosity can be produced. They have compression strength from 0.1 to 1.0MPa, and these strengths are almost proportional to square of the densities. Also, their thermal conductivities are proportional to densities and obey to Ashby-Glicksman model. Its thermal conductivity is about 0.05W/mK when density is 0.1 g/cm³. The high porosity mullite foams achieved enough thermal insulating properties for

refractory brick.

925 Difference of development of local structure with high-pressure between early and late transition metal oxides

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Crystal structure predominantly affects for function of material as well as composition. Then, to investigate effect of external field such as pressure and temperature for local environment around atoms is useful to control character of a material. Already, such investigations have been established as three kinds of Crystal Chemistry of material with ionic, covalent, or metallic bonding. However, Crystal Chemistries of materials with competition between the bonding natures is still unsettled. In this presentation, we will introduce about pressure induced phase transition sequence of simple ionic compounds, such as AMX_3 and AX_2 , including early or late transition metal elements, and then we will discuss about different structural phase transition sequence for pressure between the early and late transition metal elements based on nature of covalent bonding since decrease of interatomic distance usually enhances covalent character between the two atoms/ions.

926 Fluctuation of position and energy of a fine particle in plasma nanofabrication

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We are developing plasma nanofabrication, namely, nano and micro scale guided assembly using plasmas [1]. We manipulate nano and micro objects using electrostatic, electromagnetic, ion drag, neutral drag, and optical forces. The accuracy of positioning the objects depends on fluctuation of position and energy of a fine particle (= each object) in plasmas. Here we evaluate such fluctuations and discuss the mechanism behind them. In the first experiment, we grabbed a fine particle in plasma using an optical tweezers. The fine particle moves in a potential well made by the optical tweezers. This is a kind of Brownian motion and the position fluctuation can be caused by neutral molecule collisions, ion collisions, fluctuation of charge on the fine particle, and fluctuation of electric field. Among these possible causes, fluctuation of electric field may be main one. In the second experiment, we deduced interaction potential between two fine particles during their Coulomb collision. We found that there exist repulsive and attractive forces between them. The repulsive force is a screened Coulomb one, whereas the attractive force is likely a force due to a shadow effect, a non-collective attractive force. Moreover, we noted that there is a fluctuation of the potential, probably due to fluctuation of electric field. These position and potential energy fluctuations may limit the accuracy of guided assembly using plasmas. [1] M. Shiratani, et al., J. Phys. D 44(2011)174038.

927 Magneto-electric switching of interfacial spins toward magnetic recording/memory

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Owing to the recent development of IT, the huge amount of digital data has been treated worldwide. In order to meet the continuing requirements, magnetic storage/memory devices which possess low power consumption and high integration simultaneously, are highly desired. In contrast to conventional current-driven or magnetic-field-driven devices, voltage-driven ones offer a new architecture to meet the above requirements. Among the several methods of the voltage-driven magnetization control, we have been investigating the voltage-driven control of antiferromagnetic spins based on a magneto-electric effect. Cr_2O_3 is a conventional magneto-electric antiferromagnet. While the magneto-electric effect of Cr_2O_3 was observed in the middle of 1950s, the pioneering work that the ferromagnetic magnetization can be controlled by the coupling of exchange bias and ME effect shed a light to this technique again. In addition, the recent development achieved the ME effect in the ferromagnet/ Cr_2O_3 stacked film. In this presentation, we overview the recent progress on the magneto-electric magnetization control in all-thin-film system including Cr_2O_3 layer, in particular, two switching mechanisms: ME-field cooling [1,2] and isothermal switching [3], pulsed-voltage driven switching [3]. We also present the direct imaging of the antiferromagnetic domains using a focused soft X-ray and the coupling between antiferromagnetic and ferromagnetic domains [4]. [1] K. Toyoki, Y. Shiratsuchi et al., Appl. Phys. Express 7, 114201 (2014). [2] K. Toyoi, Y. Shiratsuchi et al., J. Appl. Phys. 117, 17D902 (2015). [3] K. Toyoki, Y. Shiratsuchi et al., Appl. Phys. Lett. 106, 162404 (2015). [4] Y. Shiratsuchi et al., submitted.

928 Fabrication of composite structures of Nd-doped laser crystals and diamond crystals by use of the room-temperature-bonding technique

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Thermal effects such as thermal lens and thermally induced birefringence prevent higher-power operations in solid-state lasers. The composite lasers, which consist of laser-ion-doped and undoped materials, are effective because the heat generated in the doped region can be removed into the undoped segment. We have recently fabricated composite Yb:YAG/YAG and Nd:YAG/YAG using the room-temperature-bonding (RTB) technique. The RTB is a versatile technique for bonding variety of materials without any degradation of crystal qualities, which is accomplished after the activation of the surface atoms by irradiating Ar atom beams to etch the surface layers, the whole process of which is carried out in a vacuum at room temperature. Since the RTB enables one to bond materials with different thermal-expansion coefficients, we tried to fabricate Nd:YAG/diamond and Nd:YVO₄/diamond composites because diamond is an ideal material for a heat spreader with its thermal conductivity as high as 2000 W/mK. We have succeeded in fabrication and laser oscillation of those composites. The output power of the non-composite Nd:YAG and Nd:YVO₄ saturated at the pump power of 19 W and 10 W, respectively, and the crystals were broken. However, the Nd:YAG/diamond composite did not show saturation even at 30 W pump power, and achieved higher output power than the non-

composite Nd:YAG. The maximum power from the Nd:YVO₄/diamond composite was also higher than that from the non-composite crystal, although the composite was damaged probably due to the anisotropy of the thermal expansion coefficients.

929 Physical simulation of industrial hot rolling of steels

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The use of physical simulation to study industrial hot rolling of steels has been a successful tool to provide metallurgical understanding and improve hot rolling schedules. Simulation work performed in a laboratory scale provides important metallurgical information about the full-scale rolling process. During hot rolling, the study and prediction of the roll force during processing is of primary importance to develop new products and to improve processing conditions. In addition, the final properties of the hot rolled product are strongly dependent on the rolling conditions. In this work, simulation of hot rolling of steels has been performed by means of hot torsion testing performed on a Gleeble simulation system in hot torsion mode. Tested schedules match the same conditions used to produce heavy gauge steel plate in industrial scale. The results show excellent correlations between full-scale industrial rolling and small-scale simulation by torsion tests.

930 Migration of interfaces in low carbon steels at low temperatures

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The most fascinating combinations of mechanical properties reached nowadays in low carbon steels are obtained after careful design of microstructures containing phases formed or manipulated at relatively low temperatures. These microstructures contain retained austenite, finely dispersed in a harder matrix consisting of bainite and/or martensite. The design of carbide-free bainitic steels and Quenching and Partitioning (Q&P) steels is based on these strategies. In the former type of steels, the carbon enrichment of the austenite is a consequence of the mechanism controlling the formation of bainite. On the other hand, in Q&P steels martensite-austenite grain assemblies are subjected to isothermal treatment to induce the partitioning of carbon from the martensite into the austenite. In both processes, interphase boundaries are observed to migrate. This work analyses the mechanisms controlling this interface migration in the two different cases based on theoretical modelling, phase field modelling and molecular dynamics. The role of carbon diffusion within the phases and across the different interphases, the coherency of the interphases as well as the mechanisms by which substitutional atoms rearrange to form the new lattices will be analysed. Results will be compared with experimental observations from our own research and from literature aiming to better understand these processes.

931 Physical properties of aluminum-carbon composites fabricated by semi-liquid route

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Aluminum-matrix composite materials reinforced with carbon-based features (Al/C) such as carbon fibers, diamond particles, or graphite flakes, have long been recognized as promising materials for the thermal management of electronic packages. These materials have driven massive research efforts since they potentially offer a desirable combination of high thermal conductivity, limited thermal expansion, limited weight impact, and acceptable cost at the industrial scale. Aluminum is however well known for suffering from immediate oxidation upon exposure to the air environment. Therefore, the sintering process of aluminum powders always has to make up with the existence of an aluminum-oxide (Al_2O_3) layer at the surface of the aluminum particles to be sintered. However, most of classical hot pressing methods let the Al_2O_3 layer intact. Therefore, an alternative way to improve the densification of Al/C composites through hot pressing could be interesting. In this study, a mixture of aluminum (Al) powders and binary aluminum-silicon (Al-Si) powders (11.3 at. % Si) is used as the matrix (95 vol.% Al – 5 vol.% Al-Si) for the fabrication of Al/C composites through hot pressing. The reinforcements either consist in carbon fibers, diamond particles, or graphite flakes. The sintering process, typically carried out at 600°C, relies on the fact that the Al-Si binary powders exhibit a melting point located around 580°C, while the Al powders remain solid until 660°C. Thus, a liquid Al-Si phase is induced in the material upon sintering, which critically enhances the densification process of Al/C composites. The Al/C composites as-processed promisingly combine low density, high thermal conductivity, and low thermal expansion.

932 Influence of the scan speed on the microstructure of AlSi10Mg processed by additive manufacturing

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The aeronautic industry seeks constant improvement of manufacturing techniques in order to improve the mechanical performances to cost ratio. Thus, additive manufacturing is seen as a promising process. Due to the infancy of the process, the influence of the process parameters such as laser power, layer thickness or scanning strategy on the microstructure requires further investigation. In the present project, bulk samples 10x10x10 mm³ in size made of AlSi10Mg are processed by Laser Beam Melting (LBM). The microstructure of the samples is characterized for different scanning speeds (165, 186, 212 and 236 mm/s). The size of the microstructure decreases as the scanning speed increases and thus the hardness increases. Electron Backscattered Diffraction (EBSD) images allow to map the crystallographic orientation of the various samples. The columnar grains seem to present a specific crystallographic orientation and are surrounded by equiaxed grains in a pattern that can be related to the hatch space. In addition, the presence of defects and porosities is evaluated by X-ray microtomography and compared according to the process parameters used. In parallel, lattice structures performed by LBM have been analyzed with the same characterization tools and compared with the microstructure obtained for the bulk samples and related to the thermal conditions.

933 Development of porous metallic femoral stems

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The biomechanical compatibility of orthopedic implants in terms of bone ingrowth and mechanical compliance is strongly enhanced when the implants integrate porous materials. In this communication, these benefits are prompted through the design, simulation, manufacture and testing of porous femoral stem models.

The design of the porous femoral stem starts with the definition of its outer skin by reproducing the shape and overall dimensions of a Stryker Secure-Fit Max femoral stem to be implanted in an artificial femur model from Sawbones. In-house algorithms are then used to generate inside the outer skin two types of porous structures: stochastic graded porous structure and diamond-type unit-cell porous structure. A model of the bone-implant assembly is developed next using the finite element method and a multiscale approach. The porous stems containing stochastic and regular porous structures are then fabricated using selective laser melting, an additive manufacturing process allowing building complex components directly from their 3D models. Finally, mechanical tests inspired from daily activities, such as walking, are conducted on intact artificial femurs. The tests are then performed on the identical femur models implanted with the porous stems and its fully dense commercial counterpart. The results of both series of tests are compared to assess which implant design (fully dense, porous stochastic or porous regular) allows the implanted femur to preserve its intact stiffness. The experimental results are then compared to the numerical predictions. Once validated, such numerical tools can be efficiently employed for structural optimization of the porosity distribution within the femoral stem.

934 Modelling fracture behaviour of high strength low alloy steel with microstructural FE model and crystal plasticity

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High strength steels are used in many engineering applications requiring combination of high strength and low weight, such as cranes, transportation and machine construction. Microstructure of these high strength steels is typically mostly, if not fully, martensitic. These materials usually fail by fracturing, more specifically brittle fracture, especially in low temperature applications. In this study we focus on the microstructural features affecting crack initiation. Properties affiliated with toughness are critical for safe use exploiting the high strength in applications and for consistent design rules for these modern steel grades.

The material is characterized with HR-SEM EBSD imaging and nanoindentation testing. The EBSD data of the base material and the heat affected zone in welding are utilized in image based finite element models. A crystal plasticity material model is calibrated to the material on the basis of the nanoindentation tests and polycrystalline homogenization. The model is verified by comparing the stress-strain response of the model to experimental results. Different size scale microstructural features (grain, packet etc) from the EBSD images are incorporated in the models and the effects of these features on brittle fracture are investigated. The results provide structure – property correlations for high strength steels, as well as insights with respect to their

systematic simulation driven development. On the basis of the findings, fracture mechanical methods for evaluation of cleavage initiation in high strength steels are discussed with respect to formulation of design guidelines.

935 Development of a self-healing thermal barrier coating system for prolonged lifetime

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In a large European research project called SAMBA, industry and universities work together on the development and improvement of a unique self-healing thermal barrier coating (TBC). A TBC is applied in gas turbine engines for propulsion and electric power generation to enhance the gas turbine engine efficiency by allowing higher operation temperatures, saving fuel and reducing CO₂ emissions. By using self-healing TBC, small cracks leading to delamination are repaired, thereby prolonging the lifetime of the coating by 20-25%. The new ceramic coating consists of a layer of yttria-stabilized zirconia, including small particles consisting of MoSi₂. Addition of these small particles allows for self-repair of the coating. Upon fracture, the silicon is oxidized and fills the crack with SiO₂. Subsequently, the SiO₂ reacts with the ceramic coating forming ZrSiO₄. This mechanism postpones failure of the TBC system. However, since oxygen can readily diffuse through the YSZ matrix and the porous nature of the coating, MoSi₂ tends to oxidize prematurely. To prevent this, the healing particles are encapsulated with a shell of alumina (Al₂O₃). With this approach, the healing mechanism will become active only when a crack penetrates the alumina shell. This self-healing concept will be realized through a combined theoretical, experimental and modelling approach. The project is carried out by Delft University of Technology and her partners, which include Forschungszentrum Jülich, University of Manchester, Institut National Polytechnique de Toulouse, Research Center RSE in Italy, Flame Spray Technologies in the Netherlands, Alstom Switzerland and GKN Aerospace in Sweden.

936 An extended mean field model for coupling discontinuous dynamic RX and post-dynamic RX

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Dynamic Recrystallization (DRX) is known to be one of the main phenomena that control microstructure evolution during hot working of austenitic stainless steels. The mean-field models for Discontinuous DRX (DDR_X) have demonstrated for years their ability to reproduce stress and grain size evolution during hot deformation. However most of them fail to predict the grain size distribution, which is necessary to couple DDR_X models with Post-Dynamic RX models. That failure is a consequence of mean field assumptions. Indeed each grain in the model have the same behavior as the same hardening and recovery parameters describe them. Thus, all grains follow the same path in the diameter dislocation-density space. In this paper, an alternative model is proposed; it is based on a stochastic approach enable to deal with the main advantages of mean field models without the last limitation. To highlight the efficiency of this

new model, experimental work was made on 304L steel (high purity alloy with 0.05 wt % Nb). Samples were deformed to high strains by hot torsion test in order to characterize the rheological behavior and microstructures within the range 950°C – 1150°C at strain rates of 0.003, 0.01 and 0.1 s⁻¹. Post-dynamic tests were also made by torsion tests with isothermal holding time after deformation. Simulation results are compared to experimental data of dynamic and post-dynamic recrystallization and to level-set based full field PDRX predictions.

937 Measurement of residual stresses in linear friction welded in-service Inconel 718 superalloy by neutron diffraction

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In this research, linear friction welding (LFW) of Inconel 718 superalloy was investigated. Welds with virgin (V) and in-service (IS) material obtained from an aeroengine were carried out. The residual stresses that evolved during the LFW process were characterized using neutron diffraction. The results suggests that the LFW process produces high tensile stresses across the weld interface for both V-V and V-IS as-welded samples. A post weld heat treatment was effective in reducing the residual stresses in the V-V samples, but not in the V-IS samples. In this research, linear friction welding (LFW) of Inconel 718 superalloy was investigated. Welds with virgin (V) and in-service (IS) material obtained from an aeroengine were carried out. The residual stresses that evolved during the LFW process were characterized using neutron diffraction. The results suggests that the LFW process produces high tensile stresses across the weld interface for both V-V and V-IS as-welded samples. A post weld heat treatment was effective in reducing the residual stresses in the V-V samples, but not in the V-IS samples.

938 Superplastic Properties of the Friction Stir Processed Al -Mg- Sc-Zr Alloys

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The research comprises microstructure analysis and the influence of the friction stir processing (FSP) on the superplastic properties of the Al-4,5Mg alloy with the additions of the various combinations of Sc and Zr. FSP causes the formation of the recrystallized microstructure with fine and equiaxed crystal graines. At optimal processing conditions of FSP, very fine crystal graines are formed, with an avarage size under 1,5 µm. The grain size of FSPed alloys dependes upon the tool rotation rate and the contents of Sc and Zr. In the Al-Mg-Sc-Zr alloys, Sc and Zr are present in various formations, as Al₃(Sc,Zr) particles that influence on the size of crystal grains and thermal stability of the microstructure at elevated temperatures. The investigated FSPed Al-4,5Mg-0,4Sc-0,2Zr corresponded to the criteria for microstructure and the thermal stability that are required for superplastic forming. The tensile tests were carried out with samples that were FSP treated. At various tool rotation rates from 95 to 475 rpm and same tool traverse rate 73 mm/min and with samples of conventional rolled alloy at initial strain rates of

1×10^{-3} to 1 s^{-1} and at forming temperatures from 350 to 500 °C. The elongations of FSPed alloy, treated with tool rotation rates up to 235 rpm, were all greater in comparison to the rolled alloy. The FSP alloy Al-4,5Mg-0,4Sc-0,2Zr has extreme superplastic properties with elongations up to $\approx 1900 \%$ without failure. The investigated FSPed alloy Al-4,5Mg-0,4Sc-0,2Zr corresponds to the criteria for high strain-rate superplasticity (HSRS), and low temperature superplasticity (LTSP).

939 Mathematical modelling of steel quenching

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Mathematical modeling of phase transformations and hardness distribution in non-monotonic quenched steel specimen was developed based on the results of simple experimental test i.e. Jominy test. The hardness in specimen points was estimated by the conversion of cooling time results to hardness by using both, the relation between cooling time and distance from the quenched end of Jominy specimen, and by using the Jominy hardenability curve. Microstructure composition and other mechanical properties were predicted based on predicted as-quenched hardness and characteristic cooling time. The cooling curve at the specimen point was predicted by numerical modeling of cooling by using the finite volume method. Developed numerical model for computer simulation of quenching was also experimentally verified. Limitations of proposed numerical model were found out as well. It has been shown that proposed numerical model can be successfully applied for purposes of simulation of quenching of carbon and low alloyed steel specimens.

940 A plasma-based surface treatment as an alternative to chromate-based conversion coating for Al alloys

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Nowadays, clad aluminum alloys are often protected by a chromate-based conversion coating (CBCC) generated using toxic chromate compounds. It is therefore necessary to identify alternative protective coatings. In this context, we have developed a multilayer combining, among others, plasma polymer films (PPF) and conventional polymers layers. The key feature of this multilayer is the grafting of a conventional polymer on the alloy surface by using the free radicals present in the as-deposited PPF as initiating sites for a radical-based polymerization reaction. It is therefore necessary to get a complete understanding on the generation and stabilization of the PPF radicals. This work aims to contribute towards the understanding of the free radicals generation mechanism and on their stabilization by comparing the plasma polymerization of different precursors namely isopropanol, benzene and cyclohexane. In situ FTIR spectroscopy and a combination of XPS and chemical derivatization measurements are used to quantitatively evaluate the plasma and thin films chemistry, respectively. Grafting experiments with 2-ethylhexyl acrylate (EHA) allows to cross-check the relevance of the XPS results. Our results reveal that, for isopropanol PPF, the surface density of free radicals is about $\sim 1.6 \cdot 10^{14} \text{ spin/cm}^2$ and depends strongly on the injected power in the

plasma. On the other hand, a significant effect of the presence of resonant structure in the plasma polymer on the radical stability is highlighted. Finally, the surfaces were used to grow EHA layer and the resulting multilayers have been tested for their protective properties revealing promising behaviors.

941 The effect of grain boundary segregation on embrittlement and magnetism in metallic systems

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Intergranular brittle fracture is closely related to the chemistry of grain boundaries. Unfortunately, the values of the segregation energy of a solute at grain boundaries and surfaces calculated by various authors sometimes differ by more than one order of magnitude. We summarize available data on interfacial segregation and embrittlement of various solutes in nickel and iron and critically discuss their reliability. We demonstrate that theoretical approaches are limited by the size of the computational repeat cell used for the calculations of the segregation energy. On the other hand, the change in the grain boundary cohesion (strengthening/embrittling energy) may be obtained with a reasonable accuracy.

To illustrate the effect of impurities on magnetism, we present a systematic *ab initio* study of segregation of 12 non-magnetic sp-impurities (Al, Si, P, S, Ga, Ge, As, Se, In, Sn, Sb and Te) at $\Sigma 5(210)$ grain boundary (GB) and (210) free surface (FS) in fcc ferromagnetic cobalt and nickel. In nickel, most of the above impurities nearly kill or substantially reduce the magnetic moments at the FS and, when segregating interstitially, also at the GB so that they provide atomically thin magnetically dead layers which may be very desirable in spintronics. Reduction of magnetic moments at the $\Sigma 5(210)$ GB in fcc ferromagnetic cobalt is, in absolute values, very similar to that in nickel. As there is very little experimental information on GB segregation in nickel and cobalt, most of the present results are theoretical predictions which may motivate future experimental work.

942 Materials processing for fluorescent probes in the second biological window

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The wavelength range between 1000 and 1700 nm has been attracting much interest for researchers who is researching on biophotonics and called as the “second biological window.” The major reason is the high transparency in biological tissues in the SBW. Since the optical loss is mainly caused by scattering, the longer wavelength for biophotonics has been a trend. Compared to the currently used wavelength, the authors has proved that one can achieve ten times deeper observation depth, several cm, for fluorescence bioimaging. Currently known fluorescent materials are quantum dots, carbon nanotubes and rare-earth doped ceramic nanoparticles. For bio-functionalizing those inorganic materials, novel processing is required. The paper will review the materials processing development for the fluorescent probes in the SBW.

943 Property improvement of (ferrite + austenite) duplex lightweight steels by TRIP and TWIP mechanisms

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The reduction in vehicle's weight has been devoted to reduce vehicle's exhaust emission and to improve fuel efficiency. The most efficient method is the use of materials lighter than conventional ones, such lightweight steels. Here in this study, new high-strength (ferrite + austenite) duplex lightweight steels containing a low-density element of Al, which exhibit tensile elongation up to 95% as well as high tensile strength (785 MPa), are presented. The enhanced properties are attributed to the simultaneous formation of deformation-induced martensites and deformation twins as the TRIP and TWIP mechanisms are working simultaneously beyond the true strain of 30% according to the relatively low stability of austenite. The present lightweight steels have outstanding properties of strength and ductility, easy manufacturing process, and costs of alloying elements as well as reduced specific weight for automotive applications.

944 Re-shaping our thoughts on thermoelectric higher manganese silicides

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In this presentation, we highlight the specific features of the crystal structure and the microstructure of higher manganese silicides based alloys (HMS) and outlined the discrepancies that arise from the number of different approaches applied to perform the difficult task of identification of HMS phases and characterization of the related transport properties. Through a strategy coupling solid state chemistry and metallurgy, our work revisits the knowledges on HMS and reveals the relationship between the phases, the microstructure and the manufacturing process. In addition, the production of grain oriented and highly pure HMS materials allows us to evidence the isotropy of the transport properties of HMS. Finally, this study suggests the relationship between grain boundary texture and segregation in doped-HMS, opening new directions for enhancing thermoelectric properties.

945 Variant selection in α/β Ti alloy

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Mechanical properties of α/β Ti alloys are improved through complex thermomechanical treatments. In particular, the β -forging modifies the α -phase precipitation. In this work we investigated the influence of the forging strain on microstructure development and its consequences on mechanical properties. Textures have been measured by neutron and X-ray diffractions. SEM/EBSD investigations and image processing have been used to understand the effect of forging on the α -phase precipitation. During forging in the β domain, two fibers

appear: and parallel to the forging direction (FD). These fibers are reinforced with the deformation. α nucleation is impacted by the forging microstructure, their orientation are determined by the Burgers orientation relationships (BOR). Up to medium deformation almost all possible α variants appear in the final microstructure. On contrary, after high deformation, variant selection is induced by β texture and microstructure, and explains the development of the fiber parallel to FD.

946 Preparation and characterization of nanostructured (Zn,Al) layered double hydroxides

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The term of layered double hydroxides (LDHs) is used to entitle synthetic or natural lamellar hydroxides with two or more kinds of metallic cations in the main layers and hydrated interlayer domains containing anionic species. LDHs, which are also called anionic clays, are mineral and synthetic materials with positively charged brucite type layers of mixed metal hydroxides. Interchangeable anions located in interlayer spaces provide the positive charge of brucite type layer. LDHs are very interesting for their intercalation properties, because they are able to host even complex organic molecules. Namely for this reason they can be applied in many different fields, such as catalysis, water treatment, biomedical science and nanocomposite material engineering. Two types of (Zn, Al) LDHs were prepared by a simple one-step hydrothermal process at room temperature using Zn salt precursors sputtered on Al foils, which act as a reactant and substrate. The properties of LDH nanoplatelets were analysed by surface-sensitive techniques. X-ray photoelectron spectroscopy (XPS) and ultraviolet photoemission spectroscopy (UPS) have been used to investigate four types of the samples, representative of the two hosted anions (Cl^- and NO_3^-) and two times of growth (6 and 24 h). The structure of LDHs was studied by X-ray diffraction (XRD), whereas their surface morphology was analysed by Scanning Electron Microscopy (SEM).

947 An appraisal of direct quenching for the development and processing of novel super-high strength steels

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The University of Oulu in collaboration with its industrial partners has set its long term vision for developing novel super-high strength steels and the possible approaches are direct quenching with or without tempering (DQ, DQT) or combined with partitioning (DQP). Depending on the carbon content and the processing variant, both strip and plate products have been targeted for various applications such as high-strength structural and high-hardness abrasion-resistant purposes. For DQ/DQT steels, the ASTM A255 approach for predicting the hardenability was considered inapplicable for boron-containing steels. Fresh attempts were made to develop new hardenability models through non-linear regression analysis by dynamically varying both the boron factor and multiplying factors of most elements in the alloy factor. While tempering enables precipitation of finer carbides in heavily dislocated laths for

enhanced strength, partitioning at or above the quench stop temperature facilitates transfer of carbon from supersaturated martensite to the remaining austenite thus partially or fully stabilizing it down to room temperature. Based on preliminary Q&P simulations, a novel TMR-DQP processing route has been designed that is amenable for industrial hot strip production not only for tough ductile structural steels, but also for hard abrasion-resistant steels. These developments, however, do not come without problems or industrial challenges. For instance, the bendability of DQ martensitic-bainitic ultrahigh-strength steels can be very anisotropic and required significant modification of subsurface microstructure for improving it. The paper will highlight recent advances made in the direction of DQ processing to realize tough, ductile super-high strength steels and the associated challenges.

948 Low temperature surface hardening of stainless steel; the role of plastic deformation

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Thermochemical surface engineering by nitriding of austenitic stainless steel transforms the surface zone into expanded austenite, which improves the wear resistance of the stainless steel while preserving the stainless behavior. As a consequence of the thermochemical surface engineering, huge residual stresses are introduced in the developing case, arising from the volume expansion that accompanies the dissolution of high interstitial contents in expanded austenite. The presentation will address two aspects of the role of plastic deformation on the case developing during low temperature nitriding. In practice, plastic deformation of metastable austenitic stainless is the rule rather than the exception. Plastic deformation of metastable austenite leads to the development of strain-induced martensite. Experimental work has demonstrated that the presence of bcc martensite leads to a thicker case, while the precipitation of CrN is promoted. On the other hand, for plastically deformed stable austenite, or austenite stabilized by a high temperature solution nitriding treatment, the case depth is insensitive for the degree of plastic deformation, and high dislocation density does not appear to promote the precipitation of CrN. Modelling of the composition and stress profiles developing during low temperature surface engineering from the processing parameters temperature, time and gas composition is a prerequisite for targeted process optimization. The evolution of composition- and stress-profiles over the developing case was developed can be simulated starting from the fundamental thermodynamic, kinetic and crystallographic data. It is shown that is necessary to include stress-induced diffusion as well as elastic-plastic accommodation of the composition-induced strains and interstitial strengthening.

949 Microstructural influence on low-cycle fatigue properties of high-manganese Fe-Mn-C steels

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This study aims to investigate the effect of initial microstructures on fatigue properties of high-manganese austenitic steels. By changing the amount of pre-strain and pre-straining

temperature, various microstructures containing deformation-induced twins, epsilon-martensite phase, and so on were developed. Tensile and fully-reversed strain-controlled fatigue tests were conducted using plate-type samples. The presence of small amount (lower than 20%) of epsilon-martensite phase exhibited higher value of fatigue life revealing well-deflected and branched fatigue crack propagation path, while high fraction of epsilon-martensite phase showed brittle fracture-surface and inferior fatigue life. The twin boundary demonstrated positive effect on fatigue properties, regardless of fraction of twin boundary. This effect of microstructures on fatigue characteristics was interpreted based on a fatigue crack initiation, propagation and cyclic hardening and softening behaviors.

950 Effect of heat treatment on the microstructure evolution of Ti-6Al-3Sn-3Zr-3Mo-3Nb-1W-0.2Si titanium alloy

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Ti-6Al-3Sn-3Zr-3Mo-3Nb-1W-0.2Si (BTi-6431S) alloy is a novel two-phase high temperature titanium alloy for short-term using at 650~700°C. In this study, BTi-6431S alloy ingot was prepared by vacuum arc re-melting, and hot rolled into 16-mm-diameter bars. The effects of heat treatment on the microstructure of BTi-6431S alloy bar were investigated through optical microscopy (OM), X-ray diffraction (XRD), electron probe microanalysis (EPMA) and transmission electron microscopy (TEM). The results show that the microstructure of as-received BTi-6431S alloy bar is composed of equiaxed primary alpha phase and transformed beta structure with 2-μm-thickness secondary alpha phase lamellas. After solution treatment at beta region and water quenching, the microstructure consists of single orthorhombic martensite phase. When annealing at temperatures from 850°C to 980°C, the content of primary alpha phase decreases while transformed beta structure increases with the annealing temperature. After 960°C/2h/AC annealing treatment, BTi-6431S alloy shows excellent high-temperature tensile properties and stress rupture property. The values of ultimate tensile strength (UTS) and elongation (EL) at 650°C reach 750MPa and 29%, respectively.

951 Characterization of a degradable Zn based alloy

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Zinc alloy is considered a degradable material candidate of great potential for medical implant application. In comparison with Mg alloy, Zn alloy has higher strength and better corrosion properties. In the present study, we prepared a series of Zn-Mg-Ca alloys and investigated their mechanical and corrosion properties. The results showed that the Zn alloy exhibited considerably high strength after extrusion and the strength varies with the increase of alloying element amount. Good anti-corrosion properties in simulated body fluids were also obtained in the Zn alloy. Microstructures of the alloy were examined to reveal the effect of the alloying elements on the properties.

952 Effect of powder outgazing conditions on mechanical and microstructural properties of oxides dispersed strengthened steel foreseen for nuclear applications

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Oxides Dispersed strengthened (ODS) stainless steels are foreseen for fuel cladding tubes in the coming generation of fission sodium cooled nuclear reactors. In spite of a body-centered matrix, those steels present a convenient creep behavior thanks to very fine oxides dispersion. Those grades are currently obtained by Powder Metallurgy (PM). After mechanical alloying with the oxide, the powder is commonly consolidated as seamless tube by Hot Extrusion (HE). Before this operation the powder is canned and outgazed in order to reduce the interaction with nitrogen and oxygen of ambient air. The control of the mechanical properties after extrusion is a key issue for this grade regarding service conditions. Therefore, effect of processing conditions must be taken into account. This study focuses on microstructural and mechanical characterization of three 14wt%Cr ODS material elaborated with various outgazing conditions before HE. Those materials are tested in term of tenacity (using Charpy impact tests) and micro hardness. In the same time microstructural characterization by X-Ray tomography reveal the occurrence of unexpected large phases whose densities is correlated with mechanical properties. EPMA characterizations are driven in order to prospect the composition of those phases. Precipitates on Prior Particles Boundaries (PPB) are also studied as they play a crucial role on the coalescence of creep damage voids. Finally, effects of outgazing conditions are summarized regarding both microstructural and mechanical properties of consolidated materials.

953 Recent advancements in arc welding consumables for III+ generation fission nuclear power plants

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The III+ generation fission nuclear power plants make extensive use of well established metallic materials for major pressure retaining equipments and pipework subjected to high temperature load, corrosive service and neutron irradiation, coherently with the evolutionary approach undertaken for these designs. Arc welding continues to be the key technology for successful nuclear components manufacturing with improved safety margins and extended lifetime. While existing low alloyed steels, austenitic stainless steels and nickel alloys continue to dominate these applications, many current designs make extensive use of larger and thicker walled components that need longer PWHT after welded manufacturing. This complicates the retaining of strength, good toughness, ductility of the weldments or corrosion resistance of the cladding layers over the much longer expected lifetime. At the same time, level of impurities in proprietary specifications was made more stringent than the baseline given in ASME or RCC-M codes. In this overview the current requirements and challenges are presented alongside the latest technology for arc welding filler consumables. Recent developments are given for low alloyed steel, austenitic steels and overlay alloys used in nuclear hardware fabrication.

954 Determination of the boron and oxygen K-edge in orthoboric acid by electron energy loss spectroscopy

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Orthoboric acid, H_3BO_3 , is an interesting material, not only due to its relevance as a neutron absorber and other technical applications, but also because of the unusual ladder of interatomic interactions. In crystalline H_3BO_3 , molecules with strong covalent intramolecular bonds form layers by forming weak intermolecular hydrogen bonds. These layers are held together by even weaker van der Waals interactions. There have been proposals for temperature-induced order/disorder transitions in crystalline H_3BO_3 as a result of freezing-in of proton rearrangement motion in hydrogen bonds [1]. Here, we characterise the bonding in H_3BO_3 by electron energy loss spectroscopy, EELS. The quality of crystals of synthetic H_3BO_3 was checked by scanning electron microscopy using a FEI Quanta 200 and 250. Electron energy loss spectra were obtained for the boron and oxygen K-edges employing a FEI Tecnai F30. After background subtraction employing the Gatan Digital Micrograph software, the spectra were compared to theoretical results obtained using density functional theory [2]. The agreement between the experimental and theoretical data is very good. This then allows us to use the quantum mechanical model for a quantitative interpretation of the bonding pattern in crystalline H_3BO_3 , which we will discuss in our contribution. Acknowledgement: DS and FS are grateful for financial support from the DAAD. We thank LINAN for access to electron microscopy facilities and Ignacio Becerril and Ana Iris Peña for technical support at the IPICYT.

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955 Nb-microalloying in next-generation automotive sheet steels

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Extensive efforts are underway worldwide to develop new steels with substantial fractions of retained austenite, for lightweight automobile manufacturing and other applications requiring improved combinations of strength and formability. It is likely that microalloying can provide product enhancements in these emerging products, such as Q&P, TBF, medium-Mn TRIP, etc. and this paper examines the expected behavior of niobium using inferences based on published AHSS literature and principles of Nb microalloying. Some benefits of Nb in terms of microstructure refinement and precipitation strengthening have been reported, and selected experimental results are shown here for Q&P simulations. The potential influences of Nb are complex due to the sensitivity of Nb dissolution and precipitation to chemical composition and processing; differences in the expected role of Nb are pointed out with respect to different product forms produced via hot-rolling or annealing after cold-rolling, and microstructures with or without substantial quantities of primary ferrite. Some issues that warrant further examination are identified, as a deep understanding of Nb microalloying and other fundamental behaviors will be needed to optimize the performance of these next-generation steels.

956 Development of new heat resistant austenitic alloys hardened with intermetallic sigma phase

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Two newly developed austenitic stainless steel concepts are presented, both of them using the precipitation of σ -phase. One is a 32Ni-27Cr steel, designed for 700 °C the other an alumina forming austenite with 25Ni-19Cr-3Al designed for lower temperatures and highly corrosive conditions. The paper will discuss the scientific concept and related microstructure development of the two alloys. The precipitation of intermetallic σ -phase together with B-stabilised $M_{23}C_6$ and Nb (C,N) is the microstructural concept of the 32Ni-27Cr steel as for the 25Ni-19Cr-3Al alloy additionally g' is present. As the precipitation of sigma phase has to occur in the austenite grain and not at the grain boundaries one major task is to avoid the transformation of grain boundary $M_{23}C_6$ to sigma. This is done by the addition of boron, which is incooperated into the carbide lattice by forming $M_{23}(C,B)_6$. It is important to mention that the incooperation of boron also changes the $M = Cr, Mo, Si$ ratio, which will also have an effect on the transformation kinetics. Furthermore, results of corrosion investigations under coal fired conditions and from creep tests are shown.

957 Experimental validation of the CALPHAD approach applied to multi-component alloys

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The last 10 years have seen significant growth in the exploration, design and development of multi-component alloys (MCAs, also called high entropy alloys, multi-principle element alloys and complex-concentrated alloys). A major motivation behind this field is the discovery of new alloys of practical benefit and new scientific principles. The vast range of hyper-dimensional composition space is both the greatest benefit and the greatest challenge, since conventional approaches are inadequate for exploring and characterizing the immense range of alloys. For this reason, the CALculation of PHase Diagrams (CALPHAD) approach is being explored to predict equilibrium phases in MCAs. CALPHAD is well-established for conventional alloys, but available databases are not optimized for MCAs. This study seeks to experimentally validate the current CALPHAD approach when applied to MCAs. Five alloy compositions were produced by vacuum arc melting and characterized with SEM, EDS, and XRD in the as-cast condition and after equilibrium heat treatments of 500hr at 1000°C and 1000hr at 750°C. Phases detected in the experimental alloys were compared with the CALPHAD predicted equilibrium phases. When considerations are taken, the current CALPHAD approach is adequate in predicting elevated temperature equilibrium phases for MCAs.

958 Evaluating athletic performances with a real time location and tracking system

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The precision in localization of the UBISENSE Real Time Location System was assessed for evaluating the possibility of using the system in the detection of movements and position of athletes on the field during the sport performance. The system was implemented in an indoor lab space measuring 11 by 7 meters. Four sensors were installed on the roof at a height of 3 meters above floor. Various sampling rates were used with a single location tag. UBISENSE is using UWB localization technology based on the triangulation of time of arrivals of UWB pulses at the different antenna sensors. Precision varied in position and speed determination being position resolution in the range of 15 to 50 cm while speed precision was in the figure of 6% at slow actual speed of 0.18 m/s up to 20% at fast speed of 1 m/s. This was mainly due to sampling frequency in the location system. In conclusion, as long as speed of the athlete is not at stake, the position can be measures with quite good resolution and sampling frequency so to permit the evaluation of sport performances of the athlete during the exercise or activity. One very attractive feature of the system is that the athlete must be tagged with a very low-weight and quite comfortable active tag which doesn't need to remain in field of view on the contrary to video tracking systems.

959 The use of vibrotactile stimulation for improving manual tasks in Parkinson's disease patients

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In spite of the potentially harmful effects of vibrations on the human body, a new path was recently opened for the use of these mechanical means in the therapeutic field. The stimulation of proprioceptive and exteroceptive sensitivity is the main target in both peripheral (diabetes type 1 and type 2) and central (stroke, Parkinson's disease multiple sclerosis) nervous system disorders, particularly for the recovery and maintenance of functional state. Our experimental apparatus consists of a virtual reality system "LEAP Motion" which involves the patient in the execution of visuo-manual tasks in a virtual environment while receiving vibrotactile stimulation. We also used a modular 36 channels EEG system and a vibratory stimulation system able of delivering vibratory stimuli perpendicular and tangential to the body surface area. And 'the study evaluation of motor performance and the ability to perform the tasks of visuomotor task assigned, in the presence and absence of vibratory stimulation and in real time, evoked potentials in the cortex or other levels of the nervous system, studying potential models of elicitation of the somatosensory system. The vibration frequency extended from 5 to 200 Hz and with accelerations between 0.3G and 1.5G with displacement amplitude of about 0.5 mm applied on the affected limb hand. In this regard, our study took into account patients with Parkinson's disease and in particular evoked potentials N18 N20 N24 N30 particularly related to tactile stimulus, and indicative of the level of perception and processing in the brain of the Parkinson's patient.

960 Near infrared device for tissue inflammation evaluation

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A photoplethysmographic (PPG) near infrared (850nm) system has been developed for the non-invasive assessment of superficial tissues (skin) swelling and redness. The PPG signal is treated to avoid ambient light and to extract the DC (direct current) and AC (alternate current) components. These components are then subtracted from the same components acquired on a reference site on the same subject. The resulting measures are used in a model able to classify different states of inflammation of the tissues. Numerical results permit to follow inflammation state and to evaluate medical and physiotherapy interventions. To avoid artefacts due to compression of the skin by the instrument, a pressure sensor has been embedded on the optical sensing head of the system, thus validating measures only if they are taken within the same range of applied pressure on the skin. This is important to get comparable PPG measures. Heart rate and haemoglobin oxygen saturation measures are a by-product output of the system and they can be exploited for other purposes or they can be taken into account for a better evaluation of the inflammation state. Although the system is still a research prototype and no real model is available about how inflammation affects the optical properties of the skin, the very first qualitative results show a strong sensibility of the system to skin alterations due to acute inflammation.

961 A novel model for diffusion-controlled precipitation reactions based on the extended volume concept: Analysis the model and applications

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Recently a new model for diffusion-controlled precipitation reactions based on the extended volume concept was derived. The new elements in the model focus primarily on the treatment of soft impingement. The new model leads to an analytical equation describing the relation between the fraction transformed, α , the reaction time, t , and the reaction exponent, n , as:
$$\alpha = \{ \exp(-2(k_1 t)^n) - 1 \} / (2(k_1 t)^n) + 1$$

In this presentation, the model derivation is reviewed. The model is compared to a range of new and old data on diffusion-controlled reactions including precipitation reactions and exsolution reactions, showing generally a very good performance, outperforming classical and recent models. The model allows new interpretation of existing data which, for the first time, show a consistent analysis, in which reaction exponents, n , equal values that are always consistent with transformation theory. New analysis methods are derived which allow determination of the reaction exponent, n , in reactions. Consistent with the interpretation, determination of n for a large number of reactions shows that n is always $1\frac{1}{2}$ or $2\frac{1}{2}$. This provides a consistent analysis where reaction model and n value are linked in a consistent way. The latter indicates that generally during a reaction with growth in 3 dimensions there are only 2 modes: either the nucleation rate in the extended volume is constant or it is negligibly small. A new approach to the interaction of diffusion-controlled growth and nucleation is proposed to rationalise these findings.

962 Heat treatments and hot forming of titanium aluminide alloys studied by in situ synchrotron radiation experiments

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Multiphase gamma-TiAl based alloys exhibit excellent high-temperature strength combined with low weight. This makes them ideal candidates for replacing the twice as dense Ni base super-alloys in industrial and aviation gas turbines. An important step towards the serial production of TiAl parts is the development of suitable hot-forming processes and subsequent heat treatments. With conventional analysis techniques thermo-mechanical processing and heat treatments can only be studied after processing. This means the real high temperature material conditions are often masked by alterations as lower temperature phase transformations or recrystallization. We use a quenching and deformation dilatometer modified for working in the HZG synchrotron radiation beamline HEMS at PETRA III, DESY. Combined with a fast detector, this setup enables the direct study of rapid high temperature and quenching processes. We have recorded the evolution of the phase constitution directly during quenching with cooling rates up to 100 °C/s. This results in the compilation of continuous cooling transformation (CCT) diagrams which provide information necessary for adjusting suitable microstructures for different applications. Another example is the direct observation of the formation of crystallographic textures in different phases during hot forming. This enables the determination of a process window which results in the formation of an optimal reduced texture from a technical point of view.

963 Advanced evaluation of fatigue phenomena using non-destructive testing methods

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The comprehensive characterization of the change in metallic materials' microstructure due to an applied load is of prime importance for the understanding of basic fatigue mechanisms. If those mechanisms are to be understood to a much greater extent, advanced fatigue life calculation methods being far away from linear damage accumulation models, have to be realized providing more than "classic fatigue data" only. Among others the physically based fatigue life calculation method named PHYBAL has been developed over the recent years. This short-time procedure allows the efforts in experimentation to be reduced by more than 90 % and therefore offers the possibility to take further fatigue relevant parameters into account. This opens the procedures for generating a S,N-curve to a multidimensional dataset. To just name a few examples, the influence of temperature, loading conditions, geometry as well as thermal and mechanical ageing processes on the fatigue behaviour can now be calculated in accordance to a process being straightforward leading to an important step with regard to improving the efficiency in monitoring components. In that context non-destructive testing parameters are increasingly considered to characterize a metallic material's microstructure allowing more precise information to be obtained regarding the actual damage condition and the integrity of a component. The paper will address the high capability of non-destructive testing techniques for

the evaluation of damage evolution processes also with respect to mechanism based fatigue as well as residual life calculations.

964 In-situ experiments for the study of advanced welding processes using high-energy X-rays

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Laser beam welding (LBW) and friction stir welding (FSW) are processes that have many advantages for industrial production; however, they still have to be optimized or developed for some modern high-temperature or lightweight alloys. Specific questions about the development of phase content during fast heating and cooling can only be addressed with time-resolved in-situ experiments, avoiding artefacts from quenching. Therefore, in-situ experiments were designed for these welding processes, employing high-energy X-rays from a synchrotron source that provide material penetration capability as well as time resolution for such studies. One example is LBW of intermetallic TiAl alloys. An LBW chamber employing a fibre laser was developed for use with high-energy X-rays from a synchrotron source. The chamber includes a furnace for heating the sample up to 900 °C before LBW. Bead-on-plate welding experiments were carried out which focused on the solid-liquid and solid-solid phase transformations in a Ti-45Al-5Nb-0.2C-0.2B alloy. Details of the solidification pathway could be revealed by the experiments. Another example is FSW of steel where the occurring phase transformations can be studied with diffraction. A FSW machine with a state-of-the-art welding head was constructed for use with high-energy X-rays. Three different steels (S355, 1.4462, 1.4410) were welded at different welding speeds. The experiments delivered detailed insight into the time development of the ferrite and austenite content of small gauge volumes at different locations relative to the welding tool. The sample environments were designed and used together with partners from the Institute of Materials Science of HZG.

965 Discontinuous precipitation of the complex intermetallic phase Nb₂Co₇ from supersaturated Co solid solution

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The binary system Co-Nb contains the very particular intermetallic phase Nb₂Co₇ that despite its complex monoclinic crystal structure can be strongly deformed at room temperature by hammering without fracture. The phase forms during cooling in a very slow peritectoid reaction from Co solid solution and NbCo₂ Laves phase at 1086 °C. As-cast material of ideal Nb₂Co₇ composition (i.e. Co-22.2at.%Nb) exclusively consists of the two phases Co and NbCo₂ and does not contain any Nb₂Co₇. Only after long-time heat treatments, the metastable two-phase mixture transforms to single-phase Nb₂Co₇. However, the kinetics of formation of this phase is very different if it is directly precipitated from supersaturated Co solid solution. The Nb solubility in the Co solid solution strongly depends on temperature increasing from values near 0 at room temperature to a maximum of 5.5 at.% at 1239 °C. By cooling Co-Nb alloys with 1,

2, and 4 at.% Nb from inside the single-phase Co field, supersaturated Co solid solutions were produced and annealed under various conditions. Nb₂Co₇ was found to form in a rather quick discontinuous precipitation reaction and this process seems to be coupled to a change of the crystal structure of the Co matrix from face-centered cubic to hexagonal-close packed. Detailed investigations of this process were performed applying a combination of differential scanning calorimetry, X ray diffraction, scanning electron microscopy, electron back-scatter diffraction, and transmission electron microscopy experiments.

966 The effects of intercritical annealing temperature and initial microstructure on the stability of retained austenite in a 0.1C-6Mn steel

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The effects of the intercritical annealing temperature and initial microstructure on the stability of retained austenite were investigated for a 0.1C-6Mn (wt.-%) steel. Medium-Mn transformation-induced plasticity (TRIP) steels exhibit a strong dependence of their mechanical properties on the variation of intercritical annealing temperature. This behavior is strongly linked to the amount and stability of the retained austenite. Thus, interrupted tensile tests were used to examine the effect of annealing temperature on the stabilization of the retained austenite. Detailed microstructural investigations were employed to elaborate the effects of its chemical and mechanical stabilization. Furthermore, the final microstructure was varied by applying the batch annealing step to an initial non-deformed and deformed microstructure respectively. Retained austenite stability along with resulting mechanical properties of the investigated medium-Mn TRIP steel was significantly influenced as the amount and morphology of the respective phases altered as a consequence of both initial microstructure and applied intercritical annealing temperature.

967 Microstructure refinement in the CoCrFeNiMn high entropy alloy under plastic straining.

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The so-called high entropy alloys, usually defined as multicomponent alloys from at least 5 principal elements with nearly equimolar concentrations, have recently attracted considerable attention due their attractive properties. The equiatomic CoCrFeNiMn alloy, for example, has single face centered cubic solid solution structure, and demonstrates remarkable strain hardening capacity, good ductility even at cryogenic temperature, and exceptional fracture toughness. Due to high ductility, the alloy can be easily plastically deformed. In these work, we present results on microstructure evolution in the CoCrFeNiMn alloy during plastic straining at different conditions. The alloy was sheet rolled at room and cryogenic temperatures, and uniaxially compressed at room temperature and temperatures of 600-1100°C with different height reductions. Scanning and transmission electron microscopy, including EBSD analysis,

was widely employed to characterize microstructure of the deformed alloy. It was revealed that at elevated temperatures microstructure development is mainly governed by discontinuous dynamic recrystallization. At room and cryogenic temperatures, mechanical twinning and shear banding plays play dominant role in microstructure refinement. Extensive refinement of the microstructure down to nano-scale level occurs as the result of rolling with reduction of 80%. Structure refinement results in increase of ultimate tensile strength from 440 MPa in initial homogenized condition to 1200-1500 MPa after rolling. Peculiarities of microstructural evolutions during plastic straining at different conditions are discussed. Specific attention is paid to debatable occurrence of mechanical twinning at room temperature.

968 Influence of small Cu addition on the crystallization behavior of soft magnetic FeCoBSiNb bulk metallic glass

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By adding 0.5 at.% to the $\text{Fe}_{36}\text{Co}_{36}\text{B}_{19.2}\text{Si}_{4.8}\text{Nb}_4$ bulk metallic glass (BMG), fully amorphous rods with diameters up to 2 mm can be cast. The addition changes completely the crystallization mechanism. Instead of the typical Fe_{23}B_6 -type metastable phase, the new Cu-added glass shows two glass transitions-like events, separated by an interval of more than 100 K, in between which a bcc-(Fe,Co) solid solution is formed. The precipitation of bcc-(Fe,Co) grains changes continuously the composition of the remaining amorphous matrix. Time-resolved X-Ray diffraction in transmission configuration using synchrotron radiation allowed the full investigation of the mechanism of phase formation upon heating. The quantitative analysis of the variation of the lattice parameter upon heating revealed that the precipitated phase contains mostly Fe, therefore, during the heating, the matrix is continuously enriched in Co. The soft magnetic properties are changed as well by the precipitation of the cubic ferromagnetic crystals. The work will present in details the crystallization behavior and the crystallization kinetics, together with the variations of the soft magnetic properties.

969 Novel ESPI measurement prototype for analyzing biological samples from cell culture technique

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An essential part of cell cultivation via cell culture technology is the determination and monitoring of culture parameters. Such parameters refer to the vitality or mutual mutations of the cell culture, while the actual number of living cells in each batch indicates the correct growth rate rather than a stagnation or an overgrowth of the cell culture. Today such parameters are determined by applying light microscopy methods or by staining specific constituents of the cells. Commonly such methods are a stressful procedure for the studied cells. Most applied dyes are toxic over a certain period of time and thus are used in low concentrations only when

necessary. Within this work a new kind of measurement device prototype was designed to address these problems. This device is based on the electronic speckle pattern interferometry (ESPI). ESPI is an optical high resolution method united with a photonic analysis system which is capable of analyzing deformations and oscillations. In this approach the combination of a greatly modified microscope with ESPI allows the determination of cellular deformation (i) at highest magnifications, (ii) with high lateral resolution, (iii) contact free, non-invasive and non-destructive and (iv) in vitro is presented. A co-developed cultivation system allows to monitor the culture parameters in real time minimizing the stress for the cell culture. Since No additional substances are needed. The presented prototype is automated to a large extent and can be operated by a special control- and regulation system (CRS) based on a microcontroller development board (Arduino Mega).

970 Microstructural instabilities in Co-Co₃Ti based superalloys

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Alloys may be derived from the Co-Ti binary system that possess microstructures comprising Co₃Ti precipitates with the L₁₂ structure embedded coherently within a Co matrix with the A1 structure. Whilst such alloys may be considered analogous to Ni-based superalloys, they exhibit only limited solubility for elemental additions and are prone to microstructural instabilities. In this study, a series of Co-Co₃Ti based alloys have been examined with varying alloying additions and the extent of their microstructural instabilities, as well as the resultant phases that formed, have been characterised. Consistent with previous reports, discontinuous precipitate coarsening was seen in binary Co-Co₃Ti alloys following prolonged exposure at high temperature. The addition of Cr and Mo to the alloy resulted in the formation a faulted Co-based martensite and Co₃Ti, in addition to a discontinuously formed lamellar aggregate of A3-Co and Co₃Ti. V additions were observed to change the intermetallic discontinuous transformation product to a hexagonal Cr-rich Co₃V phase with a six-layer stacking sequence. The addition of Ta changed the morphology of the L₁₂ precipitates, making them more cuboidal. As with the other alloys examined, thermal exposure gave rise to a discontinuous coarsening reaction. The implications of such microstructural stabilities on the prospect of Co-Co₃Ti based alloys are discussed. Funding is acknowledged through the Rolls-Royce / EPSRC Strategic Partnership under EP/M005607/1.

971 Phase transformations driven by the severe plastic deformation

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Severe plastic deformation (SPD) can lead to the bulk and grain boundary phase transformations in the materials, e.g. the formation or decomposition of a supersaturated solid solution, dissolution of phases, disordering of ordered phases, amorphization of crystalline

phases, synthesis of the low-temperature, high-temperature or high-pressure allotropic modifications, nanocrystallization in the amorphous matrix and pseudopartial grain boundary wetting. Even the SPD-treatment at ambient temperature T_{SPD} is frequently equivalent to the heat treatment at a certain elevated temperature T_{eff} . However, if the real annealing at T_{eff} leads to the grain growth, SPD leads to the strong grain refinement. The methods of T_{eff} determination after high-pressure torsion and the equifinality of T_{eff} are discussed.

972 Electron beam welding of TZM sheets

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Titanium-Zirconium-Molybdenum (TZM) alloy is in wide technical applications, like forging dies, X-ray targets for CT-tubes, or metal working tools. In the production route of some products, welding can play an important role. Refractory metals, amongst molybdenum, in general are difficult to weld, due to their high melting temperature and mainly due to their susceptibility for grain coarsening. Trials with TIG, electron beam welding (EBW), and laser beam welding showed some success, but with considerable embrittlement of the weld and heat affected zone. Electron beam welding (EBW) facilitates a very high energy density which enables welding with less total energy input compared to other welding techniques, thus accomplishing a smaller weld and heat affected zone. In this work results of welding studies on 2 mm thick TZM sheet metal butt welded without filler material are presented. With the aid of Design of Experiment (DoE), it is shown that with careful selection of the welding parameters it is possible to considerably reduce the size of the heat affected zone and the grain size of the weld. Furthermore, the influence of the welding parameters on the quality of the weld and the characterizing mechanical properties, ultimate tensile strength, hardness of weld- and heat affected zone is presented as well as the prevailing failure mechanism.

973 Systematic investigation of the temperature field in Atmospheric Plasma Processing (APP)

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The application of functional layers has increased constantly over the last decades [1]. Coating processes, like plasma spraying allow efficient processing of metal or oxide particles, and have been found their application in various sectors of industries already. Ultra fine cleaning, surface activation or surface modification with the plasma arc are also under investigation currently. In the scope of this work, the influence of four different main parameters – current, working distance, feed rate and gas flow – on the arc temperature field were investigated. Due to complex and different interactions of these parameters on the temperature field, the temperature distribution in steel and aluminium sheets were systematically carried out. Furthermore, the relationship between the measured surface temperatures and the wettability of the substrates is discussed. To generate the required data, two different experimental setups were used. First, the spatial heat distribution of the plasma arc was measured with a special arrangement of thermocouples. Second, the temperature fields were measured during the plasma surface treatment of DC01 and 6082 substrates. In addition to measurements with NiCr-Ni

thermocouples, an investigation with an IR-Camera was performed. After the plasma surface treatment, the wettability was determined by contact angle measurement. The obtained results and especially the measured temperature fields will be used in a next step to validate numerical simulations with SYSWELD and ANSYS CFX, which will be used for process optimization.
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974 Effect of initial microstructure on the static recrystallization behaviour

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Magnesium AZ31 alloy sheets were rolled at 100 °C at a high rolling speed of 1000 m/min. Six increasing reductions were employed in order to generate a series of microstructures ranging from: i) fully twinned/shear banded (reductions of 8%-30%), to ii) partially dynamically recrystallized and twinned (reductions of 49%-58%), to iii) fully dynamically recrystallized (reduction of 72%). The as-rolled specimens were annealed at 200 to 500 °C for increasing times. The microstructures were characterized by optical microscopy and electron backscattered diffraction (EBSD). Vickers hardness tests were carried out to track the progress of recrystallization during annealing. The recrystallization kinetics were analyzed in terms of the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model. The effects of the initial microstructure and microhardness on recrystallization behavior were studied.

975 Use of carbon nanocoil as a catalyst support in fuel cell

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Carbon nanocoils (CNCs) are helical-shaped carbon nanofibers (CNFs) with coil and fiber diameters of 400–1000 nm and 120–400 nm, respectively [1]. In this study, we used CNC as a catalyst support in direct methanol fuel cell (DMFC) and compared its catalytic activity with those of the catalysts supported on the other three types of carbon nanomaterials: Arc Black (AcB), Vulcan XC-72R (Vulcan), and VGCF-X. Pt and Ru were loaded onto the nanomaterials by a reduction method using sodium borohydride. Transmission electron microscopy and X-ray diffraction (XRD) revealed the PtRu catalyst particles to be 4–6 nm in diameters. The shifts in the Pt (111) XRD peaks of the catalysts on CNC and VGCF-X were larger than those on AcB and Vulcan, indicating a higher degree of alloying between Pt and Ru. Electrochemical studies of the catalysts during methanol oxidation were carried out using cyclic voltammetry. The catalyst particles supported on CNC and VGCF-X exhibited higher catalytic activity than those on AcB and Vulcan [2]. The membrane-electrode-assembly using the CNC-supported PtRu and commercial Pt catalysts was prepared and mounted in a DMFC cell. The polarization characteristics and electrochemical impedance spectra of the DMFC were measured using a fuel cell impedance meter [3]. [1] M. Yokota, et al, J. Nanosci. Nanotechnol. 10 (2010) 3910–3914. [2] Y. Suda, et al, Mater. Today Comm., 3 (2015) 96-103 [3] Y. Shimizu, et al, Electrochem., 83 (2015) 381-385

976 Effect of interfacial thermal resistance on effective thermal conductivity in aluminum matrix composites

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The interfacial thermal resistance between the matrix and the reinforcement is thought to have a considerable effect on the effective thermal conductivity of composites. To investigate the degree of the effect of the interfacial thermal resistance, we developed a simulation code which can calculate the effective thermal conductivity of composites with considering the interfacial thermal resistance. From calculation results, we defined the critical size of the reinforcement, $L_{cr} = \lambda/h$, where λ is harmonic mean of thermal conductivity of the reinforcement and the matrix and h is the coefficient of heat transfer between the reinforcement and the matrix. The critical size of the reinforcement is important to design composites. If the size of reinforcement is smaller than the critical size, it is predicted that the effective thermal conductivity will decrease by the interfacial thermal resistance. On the other hand, if the size of reinforcement is large enough, the effective thermal conductivity will not decrease. The device to measure the effective thermal conductivity of the composites was fabricated to verify the calculation results. In this study, comparison between the measured and the calculated effective thermal conductivity will be made, and the effect of the interfacial thermal resistance will be discussed.

977 InGaAs quantum wells and wires embedded in GaAs for high-efficiency solar cells

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Strain-balanced InGaAs/GaAsP quantum wells have been used for adjusting the absorption edge of a middle cell of a multi-junction cell while keeping the lattice-matching relationship among subcells. By thinning the layer thickness to a couple of nanometres and inserting GaAs interlayers between wells and barriers, it has been possible to extend the absorption edge to 1.2 eV and achieve an internal quantum efficiency close to 80% with InGaAs wells, which are the requirements for current-matched InGaP/GaAs/Ge 3-junction cells. When the InGaAs/GaAs/GaAsP superlattice was grown on a 6-degree-off substrate, surface migration of adatoms over multiple steps and resultant step bunching induced non-uniform incorporation of atoms between steps and terraces and in-plane undulation of layer thickness appeared, which is like "embedded nanowires". The cross-sectional TEM image and the X-ray diffraction reciprocal-space map confirmed such a wire-like structure. Even though the layer undulation makes it more challenging to stack a lot of layers by strain balancing, it was possible to grow 50-period InGaAs/GaAs/GaAsP superlattice on a 6-degree-off GaAs substrate. When the performance of the GaAs single-junction cells including the superlattice was compared between the cells grown on the exact and the 6-degree-off substrates, the "wire" structure grown on the 6-degree-off substrate exhibited superior efficiency of carrier collection from narrow-gap InGaAs layers, suggesting a potential merit of the "wire" structure for achieving higher efficiency when it is inserted in a middle cell of a multi-junction cell as a band-gap adjuster.

978 Hydrogen transport properties through Ni-Nb-Zr amorphous metallic ribbons and hydrogen effect on their mechanical properties

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Atomic hydrogen diffuses in a metallic lattice much faster than other small elements such as carbon, nitrogen and oxygen. The faster diffusion of hydrogen is the basis for the use of metallic membranes for hydrogen separation applications. Amorphous metallic membrane is one of the candidates for such applications. Some alloys with high zirconium content have been reported to exhibit as high hydrogen flux as that of palladium membranes, however, not much information are available regarding the diffusion characteristics and structural stabilities.

To evaluate the hydrogen diffusivity as well as other hydrogen transport properties, a time-lag technique was employed to evaluate the chemical diffusivity and permeability of hydrogen through amorphous metallic alloys. While hydrogen diffusivity for the amorphous structures was smaller than that for a palladium membrane, the amorphous membranes exhibited higher hydrogen solubility, resulting in comparable hydrogen permeability. To study the mechanical stability of the amorphous ribbons under hydrogen condition, a systematic study was made by performing nanoindentation tests with cube-corner and spherical indenter tips on Ni-Nb-Zr metallic glass ribbons having various Zr contents. Weight gain measurements after hydrogen charging and thermal desorption spectroscopy studies were utilized to identify how the total hydrogen is partitioned into mobile and immobile parts. These, in turn, indicate the significant role of hydrogen mobility in the amorphous structure on the mechanical properties. In high-Zr alloys containing immobile hydrogen, both hardness and shear yielding stress increase significantly; while in low-Zr alloys having only mobile hydrogen, decrease in both were noted.

979 Coatings of anodic titania nanotube arrays grown on titanium tubular electrodes

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Since reported in 2001, research into growth and application of anodic titania nanotube arrays has been focusing on planar titanium electrodes. Although patterned, curved, or cylindrical substrates were also employed in a number of applications, the study of nanotubes grown on a titanium tubular electrode is rather inadequate, despite their expected uses in thermal fluids. In this presentation, our recent studies on growth of titania nanotubes on tubular electrodes will be introduced. The initial results show that nanotubes are formed at both outer and inner surfaces of the electrode. The nanotube length at the outer surface decreases gradually from the side facing the cathode to that at the other side, while the length at the inner surface smears out this trend. This is due to the effect of the electric field emanating from the potential drop in the organic electrolyte. As such, uniform nanotubes are obtained by tailoring the electric field at the inner surface of titanium tubular electrodes with a length of 1 meter.

980 Investigation of annealing behavior of ultrafine-grained aluminum processed by different cooling rates after hot rolling

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A commercially pure aluminum AA1050 was processed by hot rolling followed by subsequent fast cooling (water quenching, WQ) or slow cooling (furnace cooling, FC). The materials treated via these two distinct methods were then cold rolled to acquire the ultrafine-grained (UFG) structures. The WQ UFG Al exhibited a higher yield stress and a total elongation, which are superior to those of the FC one. Microstructural examinations indicated that the WQ UFG Al has smaller grain dimensions, enhanced dislocation accumulation, a higher rate of strain hardening due to solid solution pinning and an increased proportion of high-angle grain boundaries. These microstructure configurations explain the better mechanical properties of the WQ sample. Additionally, the thermal stability of the WQ UFG Al was observed to enhance due to an increase in concentration of solid solution atoms, relative to the FC sample. X-ray pole figures of the annealed WQ samples indicated that annealing occurred in a way of grain coarsening and the rolling texture was retained. Electron backscattered diffraction and transmission electron microscopy were employed to observe the microtextures of the annealed samples. The annealing behaviour of these two samples will be discussed in detail.

981 Direct fabrication of hydroxyapatite by selective laser melting

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Hydroxyapatite (HA) was directly fabricated by selective laser melting without using binder, and the constituent and microstructure was investigated by Fourier transform infrared spectroscopy (FTIR) and Scanning electron microscope (SEM), respectively. The decomposition of HA powders often occurs prior to melting, which makes the powder size decrease largely when melting begins, so that it is difficult for sintering of the powders. The melt pool can be formed and solidified into a bulk part when the powders were compressed into a block in advance of selective laser melting. The melting of the compressed powder block can become easily by increasing beam size and decreasing the powder size. With increasing the energy density, majority of the HA in the solidified part decomposed into calcium phosphate, including α -tricalcium phosphate (α -TCP), β -tricalcium phosphate (β -TCP), tetracalcium phosphate (TTCP) and some amorphous phases.

982 Effect and mechanism of heat treatment temperature on microstructure and mechanical properties of 0Cr16Ni5Mo martensitic stainless steel

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The quenching treatment (QT) condition and tempering precipitation of low carbon martensitic stainless steel 0Cr16Ni5Mo are characterized by optical microscope, scanning electron microscope, transmission electron microscope and X-ray diffraction. The results show that the QT condition is identified as 950°C for 1h to make sure the carbides completely dissolve into martensitic matrix and prevent the grains coarsening too much. After quenching followed by tempering below 500°C, reversed austenite grows up along martensite lathes; Tempering above 550°C, massive lumps of retained austenite appear at grain and martensite boundaries, and $M_{23}C_6$ carbides precipitate on austenite. Lumps of retained austenite cannot improve impact energy but destroy tensile strength. Tempering at 600°C, the content of retained austenite 29% is the maximum. Tempering above 600°C, secondary quenching happens and grain sizes are refined, which leads to strength increase and toughness decrease.

983 Influence of β -phase on initial pitting process of AZ91D magnesium alloy

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AZ91D magnesium alloy for casting is significantly corroded in chloride environment. In addition, Al-rich- α phase, which is formed during cooling process around β phase, is one of the important factors on corrosion behavior in AZ91D. It is expected that pitting formation is dominant in the corrosion behavior in magnesium alloys. To clarify the influence of the Al-rich- α phase to pitting formation and corrosion behavior in AZ91D alloy, we have investigated the relationship between microstructure and pitting formation in this study. We have carried out polarization curve measurement, scanning electron microscope (SEM) observation and electron probe micro analyzer (EPMA) before and after the corrosion-tests.

EMPA mapping of as-cast samples indicates AZ91D alloy consist of α phase, Al-rich- α phase and β phase. Al-rich- α phase which is composed of 10 mass% Al formed around β phase. After the corrosion examination, holes were observed at Al-rich- α phase around the β phase, and it is thought that small pits grow up to be holes, because β phase is remained, while the Al-rich- α phase around the β phase preferentially dissolves with time. The result indicates that Al-rich- α phase around β phase enhance the formation of pitting and preferentially dissolve it in initial stage of corrosion in AZ91D alloy.

984 Hydroxyapatite coating and silver nanoparticles assemblies on additively manufactured Ti6Al4V scaffolds

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Custom orthopedic and dental implants may be fabricated by additive manufacturing, for example using electron beam melting technology. This study is focused at the modification of the surface of Ti6Al4V alloy fabricated via additive manufacturing technology (EBM[®]) by radio frequency (RF) magnetron sputter deposition of hydroxyapatite (HA) coating and electrophoretic deposition of silver nanoparticles (AgNPs). The addition of AgNPs is suggested to provide antibacterial effect. The negatively charged AgNPs were synthesized by wet chemical reduction of silver nitrate by glucose in water solution. Thin nanocrystalline HA film was deposited by RF magnetron sputtering while the surface topography of Ti6Al4V samples remained unchanged. Silver nanoparticles were deposited over the surface of Ti6Al4V alloy from polyvinylpyrrolidone stabilized AgNPs solution. A uniform distribution of the AgNPs with a diameter of the metallic core of 100 ± 20 nm and ζ -potential of -15 mV was observed across the surface of the scaffolds. The HA and AgNPs-coated Ti6Al4V samples were studied to determine the surface wettability, hysteresis, and surface free energy. Hysteresis and water contact angle measurements revealed the effect of the deposited HA and AgNPs layer, namely an increased water contact angle compared to the as manufactured scaffolds. A higher surface free energy is observed for AgNPs-coated Ti6Al4V surface (70.17 mN/m) as compared to the uncoated surface (49.07 mN/m).

985 Mechanical properties and magnetic susceptibility of Ti-X alloys fabricated by selective laser melting process for new biomaterial devices

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In this study, the Ti-X alloy was fabricated by selective laser melting process, and their microstructure, mechanical properties and magnetic susceptibility were investigated for new biomaterial devices. The dense builds were obtained in SLMed Ti-X alloys. This alloy dominantly consisted of the b phase (bcc), confirmed by XRD. However, it is confirmed the composition segregation of Ti and X in this alloy. The compression test result, SLMed Ti-X alloy shows good mechanical property than As-received Ti-6Al-4V alloy (JISH4650 TAB640H), the yield strength higher than Ti6Al4V 400MPa, and shows good ductility. Thus, SLMed Ti-X alloy are good candidate for new biomaterial devices. However, we need to make the further investigation that why this alloy shows excellent mechanical properties.

986 Creep deformation mechanism in Mg-Y and Mg-Y-Zn dilute solid solution alloys

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Creep behavior of hot-rolled Mg-0.2Y and Mg-0.3Y-0.02Zn (in mol%) alloys were investigated. The values of apparent activation energy for creep in both of Mg-0.2Y and Mg-0.3Y-0.02Zn alloys are around and/or more than 200 kJ/mol at the temperature range from 450 to 650 K. TEM observation of Mg-0.3Y-0.02Zn alloy has been revealed that the non-basal a-dislocation slip is activated during creep at the temperature range from 480 K to 650 K. Therefore, high temperature creep deformation mechanism is observed at this temperature region, which is observed at only very high temperatures ($>0.7T_m$) in many hcp metals and alloys. On the other hand, in crept Mg-0.3Y-0.02Zn alloy at 450 K, many c-dislocations are found on the basal and the non-basal planes. Most of a-dislocations are found only on the basal planes at this temperature and the strong interaction between non-basal c-dislocation and basal a-dislocations are observed during creep deformation. Forest-dislocation strengthening is expected during creep at this temperature and the deformation mechanism is considered to change from the cross-slip controlled dislocation creep to another one between 480 K and 450 K.

987 Effect of the melt flow induced by travelling magnetic field on microstructure formation of solidified peritectic Sn–Cd alloy

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The peritectic alloys, such as some types of steel, Ni-Al, Fe-Ni, Ti-Al, Cu-Sn, are commercially important. In contrast to other types of alloys, many unique structures (e.g. banded or island ones) can form when peritectic alloys are directionally solidified under various solidification conditions. In the course of the directional solidification experiments performed in traveling magnetic field (TMF) was observed that the melt flow has a significant effect on the solidified structure of Sn-Cd alloys. In the present work this effect was investigated experimentally for the case of Sn–1.6 wt.% Cd peritectic alloy. During the experiments a Bridgman-type gradient furnace was used equipped with an inductor, which generates a traveling magnetic field in order to induce a flow in the melt. The first 40 mm of the sample solidified without magnetic stirring, and then 60 mT magnetic field was switched on, which stirred all the remaining melt during solidification. This part of alloy was solidified with different growth velocities (v) of 0.01 and 0.02 mm/s. The microstructure was investigated by Light and Scanning Electron Microscope (Hitachi S-4800). The Cd concentration was measured along several lines as function of diameter of the samples by a BRUKER AXS type EDS without standard. The cell size of the primary tin phase was measured by an image analyser on the longitudinal polished sections along the entire length of the samples. As an effect of the melt flow induced by TMF the microstructure of directionally solidified Sn–1.6 wt.% Cd peritectic alloy changes.

988 New Fe-Al-O based ODS alloys; processing, microstructure and properties

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Oxide dispersion strengthened (ODS) ferritic steels represent a top creep-resistant structural material due to the attractive interaction of dislocations with oxides [1,2]. Moreover, the thermal stability and resistance to coarsening of oxides is excellent. The leading idea is to provide very cheap new ODS alloy with excellent creep and oxidation resistance up to very high temperatures of about 1200°C. The matrix of such new ODS alloys consists only of Fe10wt%Al coarse grained ferrite, having the solidus temperature of 1500°C, strengthened with 2-3 vol% of alumina particles of the size of 5-15 nm. Processing of the new ODS alloy comprises two steps. 1) mechanical alloying of the Fe and Al powders in a controlled oxygen atmosphere, and 2) consolidation of the powder, sealed in an evacuated steel tube, by hot rolling. During the hot rolling spontaneous precipitation of alumina particles occurs and coarse grains of about 0.02 mm in size grow by recrystallization. The new ODS alloy is afterwards annealed in air in order to stabilize the microstructure and ensure easy machinability of the material. The new ODS alloy can also be shaped by hot forming e.g. by forging. The microstructure is characterized and basic mechanical tests at different temperatures are performed for the produced new ODS alloy and the results are presented. [1] J. Rösler and E. Arzt, Acta Metall. 38, 671 (1990). [2] M. C. Brandes, L. Kovarik, M. K. Miller, G. S. Daehn, M. J. Mills, Acta Mater. 60, 1827, (2012).

989 The low-nickel cryogenic steel alloyed by nitrogen

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Phases diagram are calculated, temperature and concentration areas of existence of equilibrium phases are defined. Crystallization of Cr19Ni6Mn10Mo2N steel goes with precipitation of delta phase according to the scheme L-L+delta-L+delta+gamma-delta+gamma, critical concentration of nitrogen is equal 0,41%, and recommended 0,30%. The single-phase area gamma exists at temperatures of 1250 – 1100°C, at 1100°C AlN nitrides are appear, at 960 °C Cr₂N nitrides. Researches of structure and properties in the cast and deformed states are conducted. Low-nickel Cr19Ni6Mn10Mo2N steel has the high level of durability ($R_m = 960$ MPa, $R_{02} = 690$ MPa, $E = 33\%$, $KCU^{-163} > 1,5$ MJ/m²) in the hot-rolled and tempered from austenitic area state that provides its effective application at cryogenic temperatures, and also in the conditions of the Arctic. Increase of durability over level, characteristic for classical steel of the Cr18Ni10 type, is reached due to additional solid solution hardening.

990 Multiaxial strain path changes in 316L steel: Insitu neutron diffraction and multi-scale modelling

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A change in strain path may have a significant effect on the mechanical response of metals. Little work has been done to quantify and understand how the inter- and intragranular strains are affected during a change in strain path. Here we present multiaxial proportional and non-proportional deformation studies performed on 316L steel cruciform samples during neutron diffraction. The experiments have been carried out using in-situ neutron diffraction at the POLDI beamline in the Swiss neutron spallation source. The evolution of peak broadening during strain path changes sheds light on microstructural evolutions and the onset of in-plane texture formation. We find significant recovery of the internal strains during both unloading and elastic reloading along a direction perpendicular to the original loading direction. Such novel and unique insight into the materials behaviour during a strain path change cannot be captured by current crystal plasticity models. A multi-scale model, combining macroscale finite element approach and mesoscale elasto-viscoplastic fast Fourier transform technique is employed to test hardening laws at different length scales. Virtual diffraction peaks are generated from the mesoscale model and compared with experimental ones to test the validity of these hardening laws. Results reveal that during a 90° strain path change in 316L stainless steel, recovery of internal stresses plays an important role on the subsequent mechanical response and needs to be accounted for in existing hardening laws. The research is performed within the ERC advanced grant MULTIAX (339245)

991 Multiaxial proportional and non proportional deformation studies on ultrafine grained materials: insitu Xray diffraction

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Miniaturized uniaxial tensile and/or compression deformation set-ups that can be used in-situ during synchrotron radiation and/or in SEM are nowadays relatively common and known to provide valuable insights on deformation mechanisms and microstructures. Miniaturized multi-axial deformation devices allowing to change the strain path during deformation are however rare. Here we present multiaxial proportional and non-proportional deformation studies performed on ultrafine grained Al5Mg and Cu samples made by several plastic deformation. The Al5Mg samples have been provided by the laboratory of Prof. M. Wagner (TU Chemnitz), the Cu samples by the laboratory of Prof. R. Pippan (Erich Schmid Institute, Leoben). The experiments were carried out with a newly developed miniaturized biaxial tensile machine that can be used in a synchrotron beamline in transmission or reflection or in a SEM environment. The deformation rig allows operation of the two deformation axes independently and uses samples with a cruciform shape. The samples have been cut out from a bulk material using picosecond laser ablation. We show how the nature and magnitude of intergranular strain strongly depends on the applied stress state and demonstrate the presence of a micro yielding that depends on the strain path followed. The difference and similarities between both fine grained materials will be discussed. The research is performed within the ERC advanced grant

MULTIAX (339245)

992 Tension density as counter force to the Lorentz force density

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The accurate prediction of conduction property of materials is useful for the design of nanosize electronic devices. To include quantum effects, quantum mechanics is widely used, while electromagnetic fields are treated as classical external fields, and hence those are inconsistent with Maxwell equation, though the target is electric conduction, electromagnetic phenomena. In quantum electrodynamics (QED), electromagnetic fields are treated as quantum photon fields and consistent with Maxwell equation. Therefore, we consider these materials to be studied by QED. On another merit of QED, physical quantities can be treated as local quantities. For nanosize materials, ordinary averaged quantities are not suitable, since physical quantities of these materials are dominated by impurities or an interface. Hence we have proposed the analysis by Rigged QED. In this work, it is numerically confirmed that for equilibrium current the acceleration by the Lorentz force density is balanced with the tension density defined in Rigged QED at any point. In other words, the tension density is the counter force to the Lorentz force. This balance is explained, for example in the Drude model, by the assumption of the introduction of the phenomenological friction term, which is parameterized by the mean free time. Our tension density is introduced from the equation of motion of the kinetic momentum in QED and can be calculated. We take benzenedithiol (BDT) as an example for the numerical demonstration of the balance between the tension density and the Lorentz force density in a nonequilibrium steady state, where nonzero electric current exist.

993 The effect of Ni on the surface oxide layer during simulated brazing of aluminum alloys

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Over the past few decades, the desire for a more lightweight, energy efficient automobile has become apparent. Some of the lightest structural metals available to the automotive industry presently are aluminum alloys offering high thermal conductivity and excellent corrosion resistance. A major application of aluminum in automotive component manufacturing is in heat exchangers used in vehicle thermal management for optimal vehicle performance. These heat exchangers are manufactured via joining of Al brazing sheets that consist of a lower melting 4XXX clad aluminum alloy, or filler metal, and a 3XXX core alloy for component durability. A natural impediment to joining these brazing sheets lies in the surface oxide which inhibits the spreading of the molten aluminum filler metal. Previous studies have shown that the intermetallic Ni-Al reaction that occurs upon heating can disrupt this tenacious oxide layer, but the mechanism by which this occurs is unclear. In this study, Al braze sheet surfaces were chemically modified to create different surface oxide layers, and a thin Ni layer was sputtered onto the sheet surface. Through the use of Differential Scanning Calorimetry (DSC), Optical and SEM imaging, the metallurgical reactions that occur between the Ni layer and clad surface

during a simulated brazing process were visualized and quantified. However, the method of oxide disruption needed further investigation using X-ray Photoelectron Spectroscopy (XPS) which will be discussed. (Oral Presentation Preferred)

994 In-situ study of the recrystallization behavior of an age hardening AlMgScZr alloy

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Static recrystallization in metals is classically studied as alternating series of heat treatments and microstructural investigations often combined with a modeling approach, in general using a JMAK-type of equation. For alloys with constant growth rates this approach requires only a limited number of experiments and is thus reasonably used to study for example the fraction of recrystallization as a function of time. To investigate more detailed aspects of recrystallization such as the temporal and local initiation or sequence, however, the observation of the microstructure during annealing is more useful. Such in-situ approaches are particularly suitable when considering alloys that show non-constant growth rates, for instance alloys that exhibit recrystallization and second phase precipitation in a similar temperature regime. In such a case where Zener drag occurs, analyzing the recrystallization behavior in a classical way requires a huge amount of experiments especially when several annealing temperatures and deformation grades are investigated. In this work we present the static recrystallization behavior of an age hardening AlMg4Sc0.4Zr0.12 alloy in the temperature regime where both recrystallization and precipitation of the strengthening $\text{Al}_3(\text{Sc,Zr})$ phase occur. We investigate the recrystallized area fraction and grain size as a function of the local strain and temperature using in-situ confocal laser scanning microscopy (CLSM) and EBSD. It was found that CLSM is a powerful tool to visualize the temporal sequence of recrystallization in particular the stop of grain growth due to Zener pinning. The results show excellent correlation to EBSD analyses.

995 High pressure synthesis of boron nitride polymorphic phases and their applications

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Hexagonal BN (hBN) and cubic BN (cBN) are known as the representative crystal structures of BN. The former is chemically and thermally stable, and has been widely used as an electrical insulator and heat-resistant materials. The latter, which is a high-density phase, is an ultra-hard material second only to diamond. On the other hand, wurtzite BN (wBN) has been known as a metastable polymorphic form of BN but thus were hard to be studied as a crystalline form because solvent growth of wBN is not possible. Martensite phase transformation from well crystalline hBN to wBN were realized for studying properties of wBN crystals. In this paper, an achievement in high quality hBN, cBN crystals growth by using flux process and synthesis of wBN crystals under high pressure and their characterization will be introduced.

996 Anisotropic plasticity and crystallographic fatigue crack growth in lath martensite structures of carbon steel

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Lath martensite is the most important constituent of high-strength steels. Hence, the brittleness of martensite is a matter of concern, and it may result in cracking during the forming process of dual-phase steels, fractures in the martensite-austenite constituents formed in weld steels, and hydrogen embrittlement related to deformation-induced martensitic transformations in metastable austenitic steels. Strengthening is achieved through a combination of several mechanisms such as mutual interaction of dislocations, solid solution of carbon atoms, and the grain refinement effect. The presence of such varied effects complicates the understanding of the strengthening mechanism. In addition to that, lath martensite has a hierarchical structure comprising prior austenite grains, packets, blocks, and laths. We have not yet exactly understood which boundary affects the strengthening and cracking. The micro-constituents are on the order of several nanometres to several tens of micrometres in size. It is therefore difficult to evaluate the mechanical characteristics of each substructure using conventional mechanical testing methods. We have recently developed micro-mechanical testing of materials used for MEMS applications and thus analysed the mechanical characteristics on the scale of a few tens of micrometres. We employed micro-tensile testing and micro-fatigue testing in this study to evaluate the effects of each substructure and each boundary on the deformation and fatigue crack growth behaviours of the lath martensite. The plasticity of the lath martensite structures strongly depended on the habit plane orientation. Micro-fatigue testing of the single packet structures revealed that cracks preferentially grew along the block boundaries.

997 3-D dynamic explicit FE-analysis of Charpy impact test

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Dynamic explicit FE-analysis of Charpy impact test has been carried out, in order to investigate inertial effect on stress/strain fields ahead of V-notch in Charpy specimen. In this study, deformation behavior of the Charpy specimen and constraint effect on stress field in plastic zone near V-notch were numerically simulated by the 3-dimensional FE-analysis considering contact of the specimen with rigid striker and rigid anvils. The strain rate effect on flow stress and temperature rise during impact loading were included in the dynamic analysis. This analysis shows that the impact load exhibits oscillation, and the contact stiffness between the specimen and the rigid striker affects on the oscillation of impact load. The analysis is validated by comparison with the experimental result by using the instrumented Charpy impact testing machine, which measured impact load and load-point displacement. The oscillation on load-time curve was recorded. The magnitude and period of inertia peak load obtained by the FE-analysis are almost consistent with the experimental result. It is found that the inertial oscillation of stress near V-notch are smaller than that of impact load. This result indicates that the inertial effect of impact loading on stress field is negligible for Charpy impact test.

998 Solidification pathway for the formation of Bcc/T₁/T₂ three-phase microstructure in Mo-Nb-Si-B quaternary system

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In the present study, in order to define the reaction pathway for the formation of the solidification microstructure consisting of (Mo,Nb)-bcc, (Mo,Nb)₅Si₃-T₁ and (M,Nb)₅SiB₂-T₂ in a Mo-Nb-Si-B quaternary system, the three-phase solidification microstructures have been examined in the cast Mo-Nb-Si-B alloys. Microstructures of the arc-melted cast alloys with various compositions of Mo-32.6Nb-(14-27)Si-(2-11)B (at.%) were observed by SEM. The chemical compositions of constituent phases in the studied alloys were analyzed by EPMA. The microstructure observation determines the path of the liquidus valleys on the bcc/T₁/T₂ three-phase region in the Mo-Nb-Si-B quaternary system. The composition analyses demonstrate a chemical composition of the finally solidified region is approximately Mo-32Nb-19Si-5B (at.%), which is almost constant independent of the compositions of the studied alloys. These results indicate the solidification reaction is a three-phase eutectic. In this talk, the three-phase eutectic microstructure of the directionally solidified alloy will be presented as well.

999 Stress relaxation characteristics of oxygen-free copper and Cu-Ni-Si alloy sheets subjected to continuous cyclic bending

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Stress relaxation resistance is often required in application of electrical parts for copper and its alloys. The stress relaxation behavior should be influenced by stored strain through manufacturing process. In the present study, oxygen-free copper and Cu-Ni-Si sheets were prepared by continuous cyclic bending (CCB), which was proposed as a useful straining technique to produce the higher strain on the surface and the lower strain in the center layer of metal sheets. The CCBent sheet was analyzed by scanning electron microscope/ electron back scatter diffraction (SEM/ EBSD) technique to investigate relationship between the stored strain and Taylor factor (TF) in the CCBent sheet before and after stress relaxation test. As a result, the stored strain is high for the high TF region, whereas a significant increase of the stored strain with the decrease of the Taylor factor appears in the vicinity of the minimum TF value of 2 in the CCBent copper sheet. The stress relaxation performance was improved by the strain accumulated through CCB. Kernel Average Misorientation (KAM) tends to increase during the stress relaxation test in the front side of specimens of the Back-top stress relaxation test or in the range of a small Taylor factor. This fact suggested that formation of high TF texture would enable improvement of stress relaxation resistance for materials.

1000 Microstructure observations of graphite in gray cast iron and ductile cast iron using TEM

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Cast iron is an iron alloy mainly composed of carbon and silicon, the amount of carbon is more than 2.1 mass%. Cast irons, gray cast iron and ductile cast iron, have been used as industrial parts and automobile parts widely because they have a good wear resistance and an excellent machinability. Several spheroidization theories have been proposed, but, it is not established clearly yet. In this study, the microstructure of spheroidal graphite and flake graphite were investigated to reveal and understand the graphite formation mechanisms using FCD450 and FC250 alloys. In scanning electron microscopy (SEM) observation, graphite shape were observed in both alloys after deep etching and the shapes of spheroidal graphite and flake graphite were revealed clearly. Spheroidal graphite in FCD450 is well dispersed, however flake graphite in FC250 is a complex network. Transmission electron microscopy (TEM) samples were prepared using focused ion beam (FIB). In TEM observation, internal microstructure of spheroidal graphite was similar to annual rings in trees observed. Selected area electron diffraction (SAED) from the center of spheroidal graphite showed ring-shaped diffraction and indicated aligned orientation of the C-axis from the center to the outer part.

1001 Fabrication of ductile bulk nanocrystalline Ni-W and Fe-Ni alloys by electrodeposition

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Grain size refinement had been believed to be the promising way to strengthen materials while maintaining the tensile ductility and fracture toughness. However, many studies on nanocrystalline materials have reported that the tensile ductility of nanocrystalline materials is quite low and the obtained data are scattered widely. We consider this might be the result of problems in the sample fabrication. The reason of the scattering is the high notch sensitivity brought about the insufficient thickness of the specimens and the inhomogeneity of the nanostructure. One more important factor for the tensile ductility is reducing internal stress. It is necessary to increase current efficiency for reducing internal stress. Moreover, the control of crystal growth mode is very important to obtain large tensile ductility. In the present study, bulk nanocrystalline Ni-W and Fe-Ni alloys with a millimeter thick and minimized internal stress, concentration gradient and fluctuation were fabricated by electrodeposition. These nanocrystalline alloys exhibit large tensile elongation of more than 10%.

1002 Friction stir processing (FSP) of selective laser melting (SLM) produced Al-CNT composites

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The superior elastic modulus, strength and wear resistance of particulate-reinforced metal composites (MMCs) have drawn much attention in various industries ranging from defence, aerospace and automobile industries. The addition of small amount of reinforcement particles is able to provide significant improvement in strength. The remarkable mechanical and geometrical properties of carbon nanotubes (CNTs) have attracted much interest for MMCs fabrication. Aluminium reinforced with multi-wall carbon nanotubes were produced using Selective Laser Melting (SLM). Very fine grains were successfully achieved in both methods with high Vickers hardness values. However, cavities were present in SLM AlSi₁₀Mg-CNT parts resulting in higher stress concentration brittle fracture appearance. The high laser absorption and higher thermal conductivity of CNTs resulted in the formation of the cavities in the melted parts. FSP was performed on parts to improve the dispersion of the reinforcement particles. Microstructural evolution and mechanical properties were analysed.

1003 Controlling microstructures of Co-based L1₂/fcc two-phase superalloys having oxidation resistance

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Discovery of Co-Al-W-based L1₂/fcc two-phase microstructure has demonstrated a new class of high-temperature structural materials called as "Co-based two-phase superalloys". Extensive effort has been accumulated on the studies of phase equilibria of the ternary systems and of effect of additional elements on microstructure, the gamma-prime solvus temperature, oxidation resistance and so on. The effect of several alloying elements on physical properties of Co-Al-W-based two phase alloys has been investigated. The alloying elements modify the compositions of constituent gamma and gamma-prime phases, lattice constants of the phases, gamma-prime solvus temperature and oxidation resistance. In the present study, we have focused to design an alloy having cuboidal microstructure and oxidation resistance, which is achieved by the controlling lattice constants of the constituent phases. The effect of alloying elements on the lattice constants has systematically been investigated and an empirical equation to control the lattice misfit has been derived.

1004 Ultra-fine microstructures immediately beneath denomination cracks in fully pearlitic steels

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Wire-drawn fully pearlitic steel was twisted at room temperature. As a result, a delamination crack propagated along the longitudinal direction of the wire. The fracture surface of the

delamination crack was compared with the fracture surface obtained by an impact test at 120K, demonstrating that the delamination crack is neither a simple cleavage nor ductile shear one. Backscattered electron and transmission electron microscopy images indicated that the cementite lamellae immediately beneath the delamination crack had vanished. In addition to that, a fine-grained structure was observed. This indicated that the delamination fracture was not a brittle one but a shear one associated with local severe plastic deformation.

1005 The effect of thermal cycling on microstructure of Er₂O₃ coating layer prepared by MOCVD process

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Er₂O₃ is a high potential candidate material for tritium permeation barrier and electrical insulator coating for advanced breeding blanket systems with liquid metal or molten-salt types. Recently, Hishinuma et al. reported to form homogeneous Er₂O₃ coating layer on the inner surface of metal pipe using Metal Organic Chemical Vapor Deposition (MOCVD) process. In this study, the influence of thermal history on microstructure of Er₂O₃ coating layer on stainless steel 316 (SUS 316) substrate by MOCVD process was investigated using SEM, TEM and XRD. The ring and net shape selected-area electron diffraction (SAED) patterns of Er₂O₃ coating were obtained each SUS substrates, revealed that homogeneous Er₂O₃ coating had been formed on SUS substrate diffraction patterns.

Close inspection of SEM images of the surface on the Er₂O₃ coating before and after thermal cycling up to 700°C in Ar atmosphere, it is confirmed that the Er₂O₃ grains were refined by thermal history. The column-like Er₂O₃ grains were promoted to change to granular structure by thermal history. From the cross-sectional plane of TEM observations, the formation of interlayer between Er₂O₃ coating and SUS substrate was also confirmed.

1006 Design of strong and stable "high entropy alloys" (HEA) by multi-objective optimisation using thermodynamics and physical models

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The so-called "high entropy alloys" (HEA), sometimes rather called "highly concentrated alloys", have become a subject of huge interest in the past years, due to the unique combinations of properties they can display. Among others, they can be extremely resistant to corrosion thanks both to the use of efficient scale-forming elements as well as to their single-phase microstructures (hence with precipitate-free grain boundaries), and at the same time they can exhibit high mechanical properties, with yield stresses sometimes well in excess of 1000 MPa, as well as reasonable ductilities. The development of these alloys, typically containing between 5% and 35% of at least five different elements, has mainly relied on trial-and-error experiments, and more recently on modelling, with discrete predictions or sometimes using a systematic exploration of given alloy systems. Such design approaches have been, however, mainly limited

to the calculation, verification or optimisation of a single alloy characteristic, in most cases with the main aim of ensuring the formation of a single phase. We propose a significant breakthrough in “HEA” design, by using a multi-objective genetic algorithm optimisation of composition relying on: (i) both computational thermodynamics (Thermo-Calc) and the combination of most of the physical criteria available in the literature to guide the formation of a single phase, (ii) the maximisation of yield stress using a recently developed model to predict solid solution hardening in HEA, (iii) the minimisation of density, and (iv) the minimisation of the cost of alloying elements. Designed alloys should outperform existing HEAs.

1007 Effect of short-range ordering of solute atoms on elastic properties of Mg-Zn-Y alloy single crystals with long-period stacking ordered structures

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The elastic properties of Mg–Zn–Y alloy single crystals with an 18R- or 10H-type long-period stacking ordered (LPSO) structure were studied. Two directionally solidified (DS) Mg–Zn–Y alloy polycrystals, mainly consisting of 18R- or 10H-type LPSO structure, were prepared using the Bridgman technique. For the DS polycrystals, a complete set of elastic constants was measured during cooling from 300 to ~7 K. By analyzing the elastic stiffness of DS polycrystals on the basis of an inverse Voigt–Reuss–Hill approximation [1], the elastic stiffness components of the single-crystalline LPSO phases from 300 to ~7 K were determined. The elastic properties of the 18R- and 10H-LPSO phases were also evaluated by first-principles calculations based on density functional theory. Comparison of the measured elastic properties at ~7 K with the first-principles calculations revealed that the elastic properties of the LPSO phase were virtually dominated by the formation energy of short-range ordered solute atom clusters. Interestingly, the effect of the formation energy was significant in the elastic moduli related to the atomic bonding between the stacking layers. [1] M. Tane, H. Kimizuka, K. Hagihara, S. Suzuki, T. Mayama, T. Sekino, Y. Nagai, *Acta Mater.*, 96 (2015), 170–188.

1008 Nanoscale AB-type carbonated hydroxyapatite fabricated based on sea shells

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Hydroxyapatite (HA) is a well known biomaterial for bone grafting. Unfortunately, the osseointegration performance of normal HA is limit due to the lack of carbonate ions in the structure. Recently, carbonate-substituted hydroxyapatite (CHA) was reported to be a better bone substitution than HA because it has almost same chemical composition and structure as natural bone minerals. In this work, we purpose a method to directly fabricate nano CHA through a simple hydrothermal exchange from sea shells. The morphology and the amount of carbonate-substitution of the CHA fabricated can be well controlled by adjusting the PH values during the reaction: with the increasing of PH value, the morphology of the final product was transferred from long sheet-like to petal-like and the amount of CO_3^{2-} substitution was increased

gradually. CKK-8 assay was used to evaluate the bioactivity of the CHA. It was found that both of the sheet-like and petal-like CHA possessed significantly improved bioactivity compared to the control group, and the sheet-like CHA had the best bioactivity.

1009 Comparison of microstructure and mechanical behavior of the ferritic stainless steels ASTM 430 stabilized with niobium and ASTM 439 stabilized with niobium and titanium

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The use of Ferritic Stainless Steels have become indispensable due its lower cost and the possibility to replace austenitic stainless steels in many applications. In this study, cold rolled sheets of two stabilized ferritic stainless steels with 85 % thickness reduction were annealed by applying a heating rate of 24 °C/s and a soaking time of 24 s. The niobium stabilized ferritic stainless steel type ASTM 430 (430Nb) was annealed at 880 °C while the niobium and titanium bi-stabilized steel ASTM 439 was annealed at 925 °C. The annealed samples were tensile tested and due to the smaller grain size, steel 430Nb, showed a higher yield stress and a higher total elongation. Concerning drawability the steel ASTM 439 presented a better performance with higher average R-value, lower planar anisotropy coefficient and a greater value for Limit Drawing Ratio (LDR). These results are explained in terms of the differences in size and volume fraction of precipitates between the two steels.

1010 Combination of strength and ductility in nanotwinned austenitic 304 stainless steels

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Recently, a novel strategy was proposed for strengthening austenitic steels by introducing nanotwinned austenite grains which contain multiple twins with twin boundaries spaced in the nanometer regime. In the present work, nanotwinned austenitic 304 stainless steels were prepared by dynamic plastic deformation, which exhibit a high strength with a limited ductility. After thermal annealing, an enhanced strength-ductility combination was observed in the nanotwinned samples in comparison with the martensitic/austenitic 304 stainless steel prepared by plastic deformation and subsequent annealing. We investigated the work hardening behavior of nanotwinned austenitic grains. In the early stage of deformation, the work hardening rate dropped linearly like common Kocks-stage behavior due to the interactions of dislocation and twin boundaries. Subsequent deformation induced stacking faults and martensitic transformation in nanotwinned austenitic grains before necking, which makes the work hardening rate increase. Both stacking faults and the nano-sized α' -martensites disperse in nanotwinned austenitic grains, which are considered as strong obstacles to impede the motion of dislocations. The occurrence of stacking faults and strain-induced martensitic transformation

constrains dynamic recovery of dislocations and deformation localization, thereby contributing significantly to the work hardening and ductility.

1011 Deposition and characterization of boron-carbon-nitrogen (BCN) thin films for wear-resistant applications

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B-C-N ternary system with its superhard phases has been of great interest during the last ten years. Materials in this system are characterized by short bond lengths and expected to combine some specific properties of diamond, cubic boron nitride (c-BN), hexagonal boron nitride (h-BN) and boron carbide (B_4C), such as high hardness, low friction coefficients, good wear resistance and high thermal and chemical stability. Therefore, they are considered promising candidates as hard and protective coatings for cutting tools and other wear-resistance applications. In this study, 300-350 nm thick BCN films with different compositions were grown by reactive DC magnetron sputtering of a B_4C target by varying the N_2/Ar proportion in the sputtering gas from 0 to 50%. The increase in the nitrogen concentration had drastic effects on the films microstructure. Hardness, elastic modulus and wear rates of BCN films were significantly decreased with the increase of nitrogen in the coating composition. The results demonstrated that thin films in the ternary triangle B-C-N with tailored properties can easily be deposited by DC magnetron sputtering with varying N_2 flow rates.

1012 Influence of the beta / alpha+beta transformation on the stresses and strains evolutions during quenching of Ti17 and Ti6Al4V alloys from the beta phase field

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The evolution of the internal stresses and strains during quenching of Ti17 and Ti6Al4V alloys from the beta phase field has been simulated numerically by taking into account the coupled thermal, mechanical and microstructural evolution phenomena. Emphasis was put on the influence of the beta/alpha+beta phase transformation on the internal stresses evolutions, which is rarely discussed in the case of titanium alloys as compared to steel. The flow stress was calculated by using an isotropic visco-elasto-plastic law depending on temperature and microstructure. The parameters were determined experimentally for Ti17 alloy as a function of temperature and the microstructural state: either only beta or alpha+beta. In the latter case, the amount of the alpha phase as well as its morphology were accounted for. As for the prediction of phase transformation kinetics, global JMAK model previously developed was used. For the Ti6Al4V alloy, experimental data from literature were used. The effect of the small volume change due to the transformation was examined, as well as the possible occurrence of phase transformation plasticity. The coupled calculation of thermal, microstructural and mechanical evolutions was set up in the finite element code Zebulon. Cylindrical geometries were considered with a diameter sufficiently large to obtain significant thermal and microstructural

gradients. The calculation results show that the $\beta \rightarrow \alpha + \beta$ phase transformation has a significant effect on the level of the residual stresses and strains. The differences between β -metastable Ti17 and $\alpha + \beta$ Ti6Al4V alloys are analyzed and discussed.

1013 Design and characterization of conductive biopolymer nanocomposite electrodes for medical applications

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Mechanical stiffness, skin irritations, allergic reactions, or corrosions are drawbacks of the widely used metal based electrodes applied for medical purpose nowadays and present limiting factors for a reliable skin-surface interfacing and comfort in use [1].

To overcome these problems we have designed and tested flexible electrodes based on biopolymer nanocomposite materials. While numerous studies of conductive polymers have been conducted especially for applications in the field of organic photovoltaics and flexible organic electronics, the promising field of conductive biopolymer nanocomposites and hybrid-biopolymers is emerging. In this study, we have designed and characterized electrodes of a flexible and conductive nanocomposite material using a biocompatible and biodegradable polymeric matrix of polyhydroxyalkanoate (PHA) containing conductive nanowires. The biopolymer nanocomposites and their electrical conductivities were investigated by optical microscopy, atomic force microscopy (AFM), and electrical four point probing. We present results obtained for different PHA-polymers and various nanowire load concentrations. Finally, our developed biopolymer nanocomposite electrodes have successfully been tested for TENS and ECG applications. The results are discussed in comparison with conventional electrodes.

[1] Hanns Plenck Jr, The Role of Materials Biocompatibility for Functional Electrical Stimulation Applications, Artificial Organs, 35(3), 237-241 (2011).

1014 Thermal joining of highly conductive bonds by means of reactive multilayered Al-Ni nanofolds

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Motivated by the demand for electrified long-range vehicles, the performance of traction batteries has to be improved. To achieve a high power density, the battery cells must be interconnected with a low electrical resistance in the joints. The joining process has to fulfill additional requirements, as the battery cells may only be exposed to very low mechanical and thermal impacts. Reactive nanofolds are an innovative technology to meet the mentioned requirements. These folds are multilayered systems consisting of several hundred alternating monolayers of aluminum and nickel, each with a thickness in the range of nanometers. Their unique ability is to react with temperatures up to 1500 °C for a duration of a few milliseconds upon external ignition. This thermal reaction energy serves as a heat source in the joining process to melt the materials in the interface. The joining partners solidify and form an adhesive bond when clamped properly. However, the advantageous characteristics are opposed to

complex reaction mechanisms and an unknown interaction of the process parameters. For an industrial application of the joining technology, the requirements to activate the exothermic reaction must be known. Therefore, the process window and the mechanism of ignition have to be determined. For this purpose an experimental test rig was developed to generate and monitor short circuit currents to ignite the nanofolds. The amperage as well as the layer composition of the nanofolds were varied within a parameter study. Two independent process windows for a stable ignition were identified for all analyzed nanofolds.

1015 Relationship between microstructure and mechanical properties in Q&P-steels

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Third-Generation advanced high strength steels are being developed with the goal of reducing the body-in-white weight while simultaneously increasing passenger safety. This requires not only the expected increase in strength and elongation, but also improved local formability. Optimizing elongation and formability were often contradictory goals in dual-phase steel developments. Recent results have shown that so-called "quench and partitioning" (Q&P) concepts can satisfy both requirements. Many Q&P-concepts have been studied at ThyssenKrupp Steel Europe. Thorough investigation of the microstructure has revealed relationships between features such as the amount, morphology and chemical stability of the retained austenite and the obtained mechanical properties. An evaluation of the lattice strain by means of electron-back-scattering-diffraction has also yielded a correlation to the obtained formability. The aim of this work is to present the interconnection between these microstructural features and propose hypotheses for the explanation of how these features influence the macroscopically observed properties.

1016 Progress in the understanding of the microstructure evolution of direct laser fabricated TiAl

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With the introduction of TiAl in aircraft jet engines, there is an increasing demand for the evaluation of novel processing routes for gamma titanium aluminides such as additive manufacturing (AM). A Ti-47Al-2Cr-2Nb powder material has been used as feedstock for laser fabrication of 3D samples by means of "Selective Laser Melting" (SLM) and "Direct Metal Deposition" (DMD). A number of processing parameters including laser power, laser scan rate, powder feed rate, have been varied to evaluate their effects on the material soundness. Optimised conditions can significantly reduce the crack sensitivity for this relatively low ductility material. In particular, crack-free experimental conditions have been identified by using additional heating strategies, thus limiting built-up residual stresses during fast cooling. The different samples have been examined using optical and scanning electron microscopy in the as-build condition. The non-equilibrium cooling conditions generate ultra-fine and metastable structures exhibiting high microhardness values. A range of post-heat treatments have been performed to relieve the residual stresses and to tailor more uniform microstructures.

Conventional heat treatments in the alpha+gamma two-phase domain or in the alpha single phase domain have been successfully used to fully restore homogeneous microstructures either duplex or fully lamellar. A comparison is made with microstructures of both laser treated materials and of conventionally processed materials.

1017 Mechanical tuning of collagen fibrils through osmotic stress

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Reduction of the fibril diameter is caused because of dehydration, i.e. removing of ions and unbound water from the intrafibrillar space. As a result the axial intermolecular distance decreases and now collagen molecules are more densely packed. The increase in density explains why the mechanical properties are increased in the transverse direction. Our results show that the osmotic stress generated in our model system influences the elasticity of collagen fibrils to a very large extent in the transverse direction (42-fold change) and to a significant amount in the longitudinal direction. In vivo, osmotic stress may be preferentially increased or decreased by the amount of proteoglycans secreted from cells. This could be an important mechanism for a much quicker change in tensile stiffness as can be alternatively achieved via enzymatic crosslinking, which may take months and years. The extent of the change possible strongly depends on physiological concentrations of proteoglycans as well as the osmotic pressure these can generate per molecule, and remains to be elucidated.

1018 Control of the surface of quantum dots and semiconductor oxides for photovoltaics

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Nanostructured metal oxide semiconductors (MOS), such as TiO₂ and ZnO, have been regarded as an attractive material for the quantum dots sensitized solar cells (QDSCs), owing to their large specific surface area for loading a large amount of quantum dots (QDs) and strong scattering effect for capturing a sufficient fraction of photons. However, the large surface area of such nanostructures also provide easy pathways for charge recombination, and surface defects and connections between adjacent nanoparticles may retard effective charge injection and charge transport, leading to a loss of power conversion efficiency. Introduction of the surface modification for MOS or QDs has been thought an effective approach to improve the performance of QDSC. In this paper, the recent advances in the control of nanostructures and interfaces in QDSCs and prospects for the further development with higher power conversion efficiency (PCE) have been discussed.

1019 Microstructure and properties of an Al-12.7Si-0.7Mg alloy extrusion after an end-quenching test

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The cooling temperature field, microstructure and properties of an Al-12.7Si-0.7Mg alloy extrusion after an end-quenching experiment at 540 °C were investigated by means of an end-quenching test, hardness test, electrical conductivity determination and TEM observations. The results showed that the cooling rate of the alloy extrusion decreases sharply as the distance from the end-quenching spray point increased, when the distance from the end-quenching spray point is within 70 mm. Additionally, the cooling rate of the alloy extrusion decreased slowly along the length of the alloy extrusion when the distance from the end-quenching spray point exceeded 90 mm. The cooling rate of the alloy extrusion at distances of 20 mm and 32.5 mm from the end-quenching spray point exceeded 34 °C/s and 24 °C/s, respectively. Both hardness and electrical conductivity of the alloy extrusion within 32.5 mm from the end-quenching spray point were comparable to those of the alloy extrusion of off-line quenching at over 520 °C. There was no obvious precipitate in the alloy extrusion matrix 20 mm from the end-quenching spray point, and there were few fine and heterogeneous short rod-like precipitates in the alloy extrusion matrix 32.5 mm from the end-quenching spray point. However, there were many precipitates with lengths of approximately 10 nm in the alloy extrusion matrix 45 mm from the end-quenching spray point, and the lengths of the precipitates increased to approximately 20 nm when the distance from the end-quenching spray point exceeded 57.7 mm. The critical cooling rate of the 4-mm thick Al-12.7Si-0.7Mg alloy extrusion should exceed 24 °C/s.

1020 Microstructure evolution and its effect on creep strength of single crystal Ni-based superalloys with various orientations

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By means of creep properties measurement and microstructure observations, the effect of microstructure evolution on creep strength of the single crystal Ni-based superalloys with different orientations is investigated. Results show that, during creep at 1040 °C/137 MPa, the γ' phase in [001]-oriented alloy is transformed into plate-like N-type rafted structure, the one in [011]-oriented alloy is transformed into the stripe-like rafted structure along [001] direction, the one in [111]-oriented alloy is transformed into the plate-like rafted structure along the (010) plane. It is thought that the configuration evolution of γ' phase is attributed to the lattice expanding and contracting on the different planes of the cubical γ' phase. The γ' phase with different configurations results in the different creep resistance of alloys, the sequence of creep resistance of alloys with different orientations are determined to be $\sigma_{i[001]} > \sigma_{i[111]} > \sigma_{i[011]}$, and the effective creep activation energies of the alloys are calculated to be $Q_{e[001]} = 281.32$, $Q_{e[011]} = 146.87$ and $Q_{e[111]} = 182.61$ kJ/mol. But after pre-compressed treated along the [100] direction of [011] oriented alloy, the γ' phase in the [011] oriented alloy is transformed into the stripe-like rafted structure along the [100] direction, which make the creep life of the alloy

enhancing from 1 h to 174 h at 850°C/400MPa, and from 74 h to 165 h at 1040°C/137MPa. This is attributed to the pre-compressive treatment eliminating the gable channels to form the “labyrinth” structure, which may increase the resistance of dislocation motion.

1021 Tensile deformation mechanisms of Cu-Al alloys with high strength and good ductility

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Fully recrystallized Cu-4.4at.%Al alloy and Cu-10.8at.%Al alloy with grain sizes ranging from 0.5 micrometer to 80 micrometer were fabricated by cold rolling and annealing. Tensile tests show that both the tensile strength and uniform elongation are ameliorated with decreasing the stacking fault energy (SFE). The strain hardening behavior is thus employed to explain this phenomenon. The strain-hardening curves of the Cu-4.4at.%Al alloy shifted slightly with increasing the grain size, but the strain-hardening curves of the Cu-10.8at.%Al alloy are very sensitive to the grain size. TEM examinations of both alloys tensioned to different strains show that the Cu-4.4at.%Al alloy is favored by dislocation slip; in contrast, dislocation slip, stacking faults (SFs) and deformation twins (DTs) are widely observed in the Cu-10.8at.%Al alloy, and their roles are transformed at different strain levels in the specimens with different grain sizes. Finally, mechanisms of achieving high strength and ductility in low-SFE materials were analyzed based on the strain-hardening behavior and deformation patterns.

1022 Effect of SPD processing technique on grain refinement and properties of an austenitic stainless steel

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The formation of nanocrystalline structures and mechanical properties were studied in a nitrogen-bearing 304-type stainless steel subjected to severe plastic deformation (SPD). The steel samples were processed at ambient temperature using three different methods, i.e., multidirectional forging, unidirectional bar rolling and high pressure torsion. All these techniques produced extensive grain refinement. The microstructures consisting of austenite/ferrite crystallites with transverse dimensions of 50 and 30 nm evolved in the rolled and forged samples, respectively. The austenite fractions comprised approximately 0.4. In contrast, the microstructure consisted mainly of austenite with an average grain size of about 23 nm evolved after high pressure torsion. All samples of the stainless steel subjected to severe plastic deformation demonstrated significant strengthening. The ultimate tensile strengths of 2065 MPa and 1950 MPa, were obtained after rolling and high pressure torsion, respectively. The ultimate tensile strength of samples subjected to multidirectional forging was 1540 MPa. Effect of processing technique on the formation of nanocrystalline structure and mechanical properties is discussed.

1023 On the contribution of deformation temperature and strain to the work-hardening behavior of the twinning induce plasticity (TWIP) steel

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The austenitic TWIP steel with composition of 0.6C-18Mn-1.5Al (wt.%) showed a strength level of 1 GPa and uniform elongation of 65% at room temperature due to the formation of mechanical twins during deformation as a result of Stacking Fault Energy (SFE) of 25 mJ/m². The mechanical properties and the microstructure of the steel were studied at five different temperatures: ambient, 100, 200, 300 and 400°C. The microstructure was also analysed as a function of strain. The detailed analyses of the volume fraction of twins, the volume fraction of twinned grains, mean free path, SFE were performed after all temperatures and deformation conditions. The relationship between the mechanical properties and the formation of complex microstructure with dislocation dissociation, stacking fault formation, mechanical twinning and dynamic strain aging was found. The result also revealed that the interaction of the gliding dislocations with the stacking fault interfaces was the main contributor to the strain hardening augmentation. The mechanical twinning phenomenon was quantified and found to be an important microstructural feature that hinders the recovery at the latter phase of the deformation.

1024 High strength titanium alloys with harmonic structure for enhanced properties: Microstructure and mechanical properties

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The harmonic structure has a network structure of continuously connected "shell" with ultrafine grains and a dispersive structure of coarse-grained "core". This makes them special and different compared to heterogeneous "nano-micro" bimodal microstructures usually produced via PM route or via heat treatment following large plastic deformation. Actually, in comparison to heterogeneous bimodal microstructures, controlling the harmonic structure characteristics such as shell and core grain sizes, shell area fraction and shell network size is expected to induce outstanding mechanical properties. Indeed, current investigations of the mechanical properties under tensile tests revealed that materials with harmonic structure design exhibit both high tensile strength without loss of ductility, which is usually found in conventional metallic materials with homogeneous ultrafine-grained microstructures. However, due to more and more demanding and complex performances for structural materials, optimized microstructures such as the harmonic structures need to be validated by studying their behavior and stability under various loading conditions and environments. In the present work, mechanical properties under simple shear test (monotonic and cyclic), cold rolling, high strain rate loadings and in situ X-ray diffraction tensile tests were conducted on pure Ti, Ti64, Ti15333 titanium alloys with harmonic structure. The underlying deformation mechanisms were analyzed by EBSD, conventional and in situ TEM experiments. The results are discussed and compared with

conventional titanium alloys.

1025 Effect of tempering on microstructure and creep properties of a P911-type steel

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Microstructure and creep properties of a P911 steel normalized at 1050°C and then subjected to one-step tempering at 750°C for 3 h and two-step tempering at 300°C for 3 h + 750°C for 3 h were examined. The tempered martensite lath structure with a lath thickness of 340 nm evolved after both tempering regimes. However, relatively high dislocation density of $5 \cdot 10^{14} \text{ m}^{-2}$ remained after two-step tempering, while one-step tempering resulted in a dislocation density of $3 \cdot 10^{14} \text{ m}^{-2}$. The precipitation of M_{23}C_6 carbides with a mean size of 120 nm on high- and low-angle boundaries and V-rich MX carbonitrides having a “wing” shape with an average length of about 40 nm took place in the both cases. Although, the additional number of Nb-rich $\text{M}(\text{C},\text{N})$ carbonitrides precipitated on dislocation during low temperature tempering with a mean size of 10 nm was found after two-step tempering. The creep tests were carried out under constant load condition at 650°C using two stress levels: 100 and 118 MPa. Analysis of creep rate versus time curves showed that the use of two-step tempering decreases the minimum creep rate by retarding the onset of acceleration creep. The effect of microstructure and a dispersion of Nb-rich $\text{M}(\text{C},\text{N})$ carbonitrides on the creep properties is discussed. The study was financially supported by the Russian Science Foundation, Project no. 14-29-00173.

1026 TEM study of dislocations and stacking faults in low-angle grain boundaries of alumina

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Alumina ($\alpha\text{-Al}_2\text{O}_3$) is one of widely used structural ceramics. The microstructures of alumina have been studied for a long time. Dislocations in alumina are known to be typically dissociated into a few partial dislocations with a stacking fault in between. However, their atomic structures are not fully understood. In this study, we investigated the atomic structures of $b = 1/3\langle 11\text{-}20 \rangle$, $\langle -1100 \rangle$, and $1/3\langle -1101 \rangle$ dislocations in low-angle grain boundaries of alumina. Alumina bicrystals with a low-angle tilt grain boundary were fabricated by diffusion bonding at 1500°C in air. From the bicrystals, TEM samples having a grain boundary were prepared using mechanical grinding and Ar ion milling. The TEM samples were observed by transmission electron microscopy (TEM). Our results showed that the dislocations in the boundaries are dissociated into partial dislocations with a stacking fault. We found stacking faults on the $\{11\text{-}20\}$, $\{1\text{-}100\}$ and (0001) plane, associating with the dissociation reactions of the $1/3\langle 11\text{-}20 \rangle$, $\langle -1100 \rangle$ and $1/3\langle -1101 \rangle$ dislocations, respectively. The stacking faults were further investigated by theoretical calculations on the basis of first-principles methods. From the experimental results and the theoretical results, the atomic structures of the stacking faults were finally determined. In the presentation, we will discuss the dissociation reactions of the

dislocations in detail.

1027 Application of diffraction-amalgamated grain-boundary tracking to deforming aluminium polycrystals

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A novel experimental method has been developed by amalgamating a pencil beam X-Ray diffraction (XRD) technique with the recently developed grain boundary tracking (GBT) technique. XRD and GBT are both non-destructive in-situ analysis techniques for characterizing bulk materials, which can be carried close to the point of fracture. DAGT provides information about individual grain orientations and 1-micron-level grain morphologies in 3-dimensions together with high-density local strain mapping. An Al-3 mass % Cu model alloy was used to investigate its deformation behavior under tension. The morphology of the grains was determined by the X-ray microtomography imaging and the liquid metal wetting technique, after which GBT provided an accurate description of the position and morphology of all the grains in a region of interests. Diffraction spots in the XRD experiments were related to grains, making it possible to describe crystallographic orientation of all the grains. It has been revealed that deformation is localized at both microscopic and meso-scopic levels. Inhomogeneous deformation was observed in each individual grain. In addition, a group of a few grains coordinately interacts and specific grain boundaries thereby exhibit intense strain localization. Hydrostatic tension was also observed at quadruple junction points and its mechanism was analyzed.

1028 Prediction of crack initiation and growth from white-etching areas in bearing steels

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The presence of non-metallic inclusions on fatigue strength is a factor of paramount importance in the performance of bearing steels. Bearings can fail after a number of cycles due to rolling contact fatigue (RCF), even if the contact stress levels applied are below the yield strength of the material. Material inhomogeneities act as stress raisers at the subsurface of bearing raceways, from where cracks can nucleate and eventually propagate to the surface. The hard white-etching butterfly wings stem from fine precipitate dissolution and the formation of nanocellular structures; they can originate at voids, non-metallic inclusions, residual carbides, or pre-existing microcracks already formed at early stages of the bearing life.

An extensive analysis has been made on the nature and evolution of butterflies under different rolling contact pressures. Around 1000 butterflies has been measured and characterised. Several mechanisms and factors have been proposed to explain the formation and evolution of butterflies. In this work, a Hertzian contact analysis has been combined with phase kinetics model to describe the butterflies kinetics. The computation of the residual stresses after unloading, maximum shear stresses and stress intensity factors on the different modes of loading of cracks are compared with the experimental data obtained. The factors affecting crack

nucleation and growth, and the corresponding microstructural evolution in damaged areas are described, providing a tool for material design in order to increase component life.

1029 Athermal ω -phase transformation and mechanical properties in binary Zr-Nb biomedical alloy

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Magnetic resonance imaging (MRI) is used as an important diagnostic tool for orthopedic and brain surgery because various cross-sectional views are obtained in the body. The artifacts occur on the MRI images around metal implants which are also caused by the heat generation and displacement of the metal implants in high magnetic field. Zr-Nb alloys have recently received a considerable attention to decrease artifacts in MRI because magnetic susceptibility of these alloys are lower than Co-Cr alloys and Ti alloys for biomedical applications. The ω -phase transformation strongly affects the magnetic susceptibility and mechanical properties in the Zr-Nb alloys but this has not been clarified so far. In this paper, therefore, it is to clarify the phase transformation behavior in Zr-xNb alloys ($10 \leq x \leq 18$) by electrical resistivity measurements and transmission electron microscopy (TEM) observations. The athermal ω -phase was observed to precipitate in the alloys with $x = 10$ and 12 which exhibit the positive temperature coefficient dependence in the resistivity curves. In contrast, the alloys with $x = 14$ and 18 show an anomalous negative temperature coefficient in the resistivity curves. In addition, the selected area diffraction pattern from the alloy with $x = 14$ corresponds to not only ω -phase but also diffuse satellites at the temperature where the negative temperature coefficient dependence in the resistivity curves. This diffuse satellites appear due to the transverse wave from lattice modulation. Finally, the effect of these transformation behavior was discussed on the basis of the deformation mode in this paper.

1030 Experimental study and thermokinetic modelling of carbides precipitation sequences in 2,25Cr-1Mo bainitic steel

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In this work, carbides precipitation sequences have been studied for different conditions of time and temperature ranging respectively between 0 and 24 hours and 650 to 725°C. The carbides have been thoroughly characterized by means of TEM study on carbon replicas and electro-etching extraction procedure coupled with X-ray diffraction allowing a complete description of these phases: crystallographic structure, size, morphology and chemical composition. Nucleation sites and nucleation mechanisms have also been determined: M_3C precipitate upon decomposition of austenite, then M_7C_3 carbides nucleate at the interface between cementite and matrix. M_2C precipitate homogeneously in the matrix, while $M_{23}C_6$ are located at dislocations within the bainitic laths. These observations were successfully reproduced by a thermokinetic approach using the Matcalc software.

1031 Design of high strength aluminium alloys by application of rapid solidification and hot extrusion technology

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Mechanical properties of alloys are determined by the combination of various strengthening mechanisms. Additions of different alloying elements give a possibility to design and use of particular mechanism to achieve various engineering goals such as high strength, ductility or corrosion resistance. In practice, commercial materials are designed and optimized with respect to the existing industrial technologies, however by application of non-standard processing techniques such as Rapid Solidification (RS) improvement of materials performance can be expected. It was found, that combination of rapid solidification and hot extrusion technique, promotes significant structure refinement which in turns results in significant increase of strength and plasticity of commercially available alloys. As an example, a typical corrosion resistant aluminum AA5083 alloy with the grain size below 1 μm can be prepared by combination of melt spinning technique and conventional hot extrusion. Such processing give rise 50% increase of the yield strength without any lose of plasticity. It is expected that much more can be achieved if the chemical composition of the alloy will be tailored directly for the application of RS technology. In the present work, a non-conventional approach to aluminum alloys design based on rapid solidification and hot extrusion technology will be presented. The opportunities and challenges will be discussed in terms of production of high strength alloys. As an example, Al-Mg system based materials were designed and produced, which exhibit yield strength of 500 MPa, maximum strength of 600MPa and plasticity of 12% in as extruded condition.

1032 Strain rate dependent failure of material interfaces at nano-microscale via nanoimpact experiments

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The overall strength of the material depends on the properties of constitutive materials and on the interfaces where constitutive materials interact with each other. The focus of the presented work is on investigating strain rate dependent constitutive properties of a set of metallic and glass-epoxy interfaces ubiquitous in nature using a combined experiment-theoretic approach that delves on measurement of interface dynamic thermomechanical properties. Defects in laminated materials mostly occurs/starts at the interfaces that leads to its overall failure. Individual properties of constitutive materials are well investigated in the literature but there are not enough studies to quantify the interface strength between two different materials. In the present work we have performed experiments on the glass-epoxy interfaces and metallic (superalloy-steel, Ti-steel etc.) at different strain rates using nano-impact experiments. The strain rate in the experiments are up to 300 s^{-1} . The experiments are conducted on samples with different interface thickness from 10 to 200 μm . The effect of interface layer thickness and strain rate on fracture and toughness resistance is presented along with failure mechanisms.

Analyses point out the role of mechanical loading, and interface hierarchy in unique thermomechanical properties of examined interfaces. More importantly, interface topology and roughness are shown to play an important role in hierarchy dependent properties of biological materials. Based on analyses new interface constitutive laws as well as rate dependent cohesive separation laws are formulated and presented. Such laws are then used to analyze interface crack propagation problem as a function of applied strain rate.

1033 Ni-Fe alloy nanoparticles for hydrogen and syngas production by steam reforming of biomass tar

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The conversion of biomass to hydrogen and synthesis gas (CO+hydrogen) has attracted much attention from the viewpoint of biomass utilization of the power generation and production of liquid fuels and chemicals. A conventional method is a non-catalytic gasification with air at very high temperature (> 1073 K) in order to suppress the tar formation. In order to avoid very high reaction temperature. We have attempted the catalytic steam reforming of tar derived from the pyrolysis of biomass at lower temperature such as 773-873 K [1]. Here, the catalyst development is essential. Our group has reported alpha-alumina supported Ni-Fe [2], Ni-Co [3], Co-Fe [4, 5] alloy catalysts for the tar steam reforming. Our group also developed the catalysts having Ni-Fe and Co-Fe alloy nanoparticles (Ni-Fe/Mg/Al [6-9], Co-Fe/Ni/Mg/Al [10, 11]) prepared using hydrotalcite-like precursors by calcination+reduction. Ni-Fe alloy nanoparticles with uniform composition like Fe/Ni=0.2 were synthesized and showed very high catalytic activity in the steam reforming to produce hydrogen and CO at low temperature [6-9]. Moreover, the addition of trace Pd to Ni/Mg/Al enhanced the catalytic performance in the oxidative steam reforming [12], where Pd atoms were alloyed with Ni metal particle at the surface. References: [1] Bioresource Technol., 178 (2015) 53, [2] Appl. Catal. A, 392 (2011) 248, [3] Fuel, 112 (2013) 654, [4] Appl. Catal. B, 121-122 (2012) 95, [5] Appl. Catal. B, 140-141 (2013) 652, [6] Appl. Catal. B, 102 (2011) 528, [7] ChemSusChem, 5 (2012) 2312, [8] ChemSusChem, 7 (2014) 510, [9] Appl. Catal. A, 506 (2015) 151, [10] Appl. Catal. B, 150-151 (2014) 82, [11] Appl. Catal. B, 160-161 (2014) 701, [12] Appl. Catal. B, 179 (2015) 412.

1034 Producing amorphous/crystalline composites by powder metallurgy

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CuZr-based alloys have unique physical and mechanical properties, e.g. high strength, which associates with low ductility. It is known that this properties can be enhanced by adding crystalline phase to the amorphous alloys creating amorphous/crystalline composite. The present work intends to produce amorphous particle reinforced crystalline matrix composite by powder metallurgy. As the first step the amorphous powders were synthesized by ball-milling of crystalline powders. $(\text{Cu}_{49}\text{Zr}_{45}\text{Al}_6)_{80}\text{Ni}_{10}\text{Ti}_{10}$ (at%) and $(\text{Cu}_{49}\text{Zr}_{44}\text{Al}_7)_{80}\text{Ni}_{10}\text{Ti}_{10}$ alloys were ball-milled for 12 hours in order to reach the fully amorphous structure. The impact energy of

the balls during milling causes the change of the structure in the powders.

Furthermore, high purity crystalline copper powder was mixed with the amorphous powders in concentration range from 0 to 50 wt%. The powder mixtures were hot pressed at 390 °C for 4 hours under argon atmosphere. The optimal annealing temperature was determined beforehand in order to prevent crystallization of amorphous powders. In our experiments porosity and homogeneity of the particles were investigated by light microscopy and image analyser. The connection between the particles were analysed by scanning electron microscopy.

1035 The effect of Sc addition on microstructure in Mg-Gd alloys

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Traditionally, Mg-Gd alloys have been strengthened by dispersed precipitates. Several reports are available about Sc addition to Mg alloys for improving a creep resistance. In this research, aging behavior of Mg-Gd, Mg-Sc and Mg-Gd-Sc alloys including the same amount of solute elements were investigated to understand the effect of Sc on microstructures and mechanical properties during aging. Hardness measurement revealed that Sc addition delayed to form precipitate. Close inspection of TEM micrographs, beta double-prime formed at an initial stage of aging and beta prime was observed at a peak-aged stage in Mg-Gd and Mg-Gd-Sc alloys. In Mg-Sc alloy, there is no evidence of precipitate formation during aging at 473K.

1036 Development of the advanced TBC for high efficiency gas turbine

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Turbine inlet temperature has been increasing for the demand of higher thermal efficiency of combined cycle gas turbines. Currently, MHI has been actively participated in the Japanese national project which targets 62% combined cycle thermal efficiency through the development of 1,700 °C class gas turbine. Parts of the component technologies developed in the national project are applicable to 1,600 °C class gas turbine called M501J, which has the highest turbine inlet temperature in the world. In particular, thermal barrier coatings (TBCs) are one of the most essential technologies to achieve the target. During the development of the advanced TBCs for new challenges, the pilot power plant called T-point in MHI's factory had been necessary. The advanced TBC coated on the actual hot parts can be verified in the plant and feedback could be obtained after operation. In this paper, the TBC development and verification utilizing the MHI's actual power plant are discussed.

1037 Analysis of excellent mechanical properties balance of 0.1%C-2%Si-5%Mn ultrafine fresh martensite and ferrite austenite steels

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It is very difficult to emerge a steel which has an excellent total balance of high strength, high ductility with both uniform elongation and local elongation, and high toughness. That is because these properties are in the trade-off relation. Tensile strength (TS) increases, total elongation (TE) decreases. Therefore, $TS \times TE = 20000 \text{ MPa}\%$ is said to be limit of the balance. Development of the steel with $TS \times TE > 30000 \text{ MPa}\%$ is a common target for researchers in the world. 0.1%C-2%Si-5% Mn martensitic steels have a high tensile strength of more than 1400MPa and a high total elongation of 17% and ferrite+austenite steels have a high tensile strength of more than 1200MPa and a high total elongation of 30 %. They are considered to be very attractive in industrial application. However, the mechanism of the excellent tensile strength and ductility balance is not clarified. To investigate the mechanism, transformation behavior and work hardening behavior were examined. Tensile strength, uniform elongation and total elongation were simultaneously improved with increasing in Mn content. Work hardening is an important role to enhance both strength and ductility. Dislocation density once decreased with increasing in strain slightly. Then in the case of 1.5%Mn, dislocation density continued to be decreased, on the other hand, in the case of 5%Mn, dislocation density increased with strain. Mn helps to enhance dislocation density with tensile strain even though dislocation density is very high, resulting in higher work hardening rate. Mechanism is analyzed with synchrotron radiation. In addition, effect of martensite grain size effect on mechanical properties is discussed.

1038 Zinc oxide sputter lubricative coatings

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Reduction for energy saving has been required of friction loss of the moving parts, bearings, in a turbine system and thus save fuel and extend the lifetime of the moving parts. Many low-friction coatings have been developed so far. Friction coefficients of solid lubricants usually however increase due to reaction with oxygen in air, in oil, in the harsh condition of high-temperature and high-humidity. Metal oxide coating is a strong candidate to solve this problem, because of the stable crystal structure in the harsh condition. Friction coefficient of metal oxides known so far is usually too large to be used as lubricative coatings. We have therefore developed the reduction method of friction force of metal oxide coatings with crystal preferred orientation by using a combinatorial sputter coating system. We have succeeded in depositing ZnO coatings with controlled crystal preferred orientations on stainless steel plates by r.f. sputtering. ZnO coatings with bi-crystal orientations of (002) and (103) have the best low-frictional properties in atmosphere, UHV, and lubricative oil. The potential of ZnO coating was shown to be used as low-friction coating in oil and vacuum. We have also developed an efficient technology to optimize the crystal preferred orientations for the lowest friction of the coating and observed a piezoelectric effect of ZnO coatings.

1039 The limits of grain fragmentation in severe plastic deformation

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Ultra fine grain size materials are good candidates for light weighting because of their high strength thanks to the Hall-Petch relation. One promising way to produce them is severe plastic deformation (SPD) which is a top-bottom technique where grain fragmentation is induced by the extreme large plastic strains. The main mechanism of grain refinement is the grouping of geometrically necessary dislocations (GNDs) into walls of increasing disorientation. It is shown in this lecture that their density is first increasing up to about an equivalent von Mises strain of about 4 than begins to decrease and levels off in a steady state. At the same time, multiscale polycrystal simulations show that the strain heterogeneities are reduced in the polycrystal when the grain size is decreasing. In nano-polycrystalline materials, it is approaching the Taylor homogeneous polycrystal deformation mode. Recent detailed microstructure studies show that the disorientation distributions between neighboring grains can be related to the grain fragmentation process. The difference between the so-called correlated (between neighbors) and the non-correlated (randomly assigned) disorientation distributions have been shown to correlate with the GND density evolution of the polycrystal. These new elements of SPD research help us in the understanding of the grain fragmentation process and especially the limiting stage during which the smallest possible grain size can be obtained.

1040 Multilayer thin films: How residual stresses influence the fracture properties

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Over the past few years the miniaturization of devices has become more and more important, with an increase in complexity at the same time. Therefore, small scale testing methods are indispensable to determine the materials properties locally. Up to now not much attention has been paid to the fracture properties of thin films, where residual stresses have to be taken into account, as they influence performance and fracture behaviour tremendously. We concentrate on recent developments regarding the local determination of residual stresses and how they influence fracture properties of thin films. The materials investigated are Cu-W-Cu and W-Cu-W trilayer systems, with an individual layer thickness of 500 nm, on a Si substrate. Cantilevers are fabricated using a cross section polisher and focused ion beam (FIB) milling. The residual stresses are determined by means of an improved ion beam layer removal (ILR) method, where the stress is calculated from the deflection of a cantilever. This deflection changes due to stress redistributions, when the film is gradually removed using FIB milling. Subsequently, fracture experiments are performed in-situ in an SEM to get a detailed view on the ongoing fracture processes. For a comprehensive analysis, an accompanying finite element based modelling approach is introduced to determine crack-driving forces in the presence of interfaces and residual stresses. We want to emphasize the importance of residual stresses when addressing the fracture behaviour of thin films.

General possibilities, challenges and benefits of our approaches, including an outlook on the

determination of the interface toughness, will be discussed.

1041 Effect of a number transition metals on the cohesion properties of Cr-base alloys

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We used the results of ab initio calculations [1] to investigate the influence of a number of metals (W,Ta,Nb,Mo,V,Hf,Ti,Zr,Ni) on the cohesive properties of Cr-base alloys. A value of the partial cohesive energy is a parameter that allows us to determine the effect of an alloying element on the cohesive strength of the bulk. A parameter equal to the change in the work of GB separation with alloying was used to estimate the effect of alloying elements on the GB cohesive strength. The data calculated demonstrate that the most effective strengthening element for the bulk and GB in Cr-base alloys is W. Ta, Nb, Hf, Zr are the additions that have a significant influence on GB as the surface-active elements and besides Nb, Hf and Zr lead to weakening of interatomic bonding in GB. We assume that such a behavior of Nb, Hf and Zr can be used to activate a mechanism of GB sliding and promote plastic deformation in Cr-base alloys. That is important because Cr and its alloys are prone to embrittlement at low or intermediate temperatures. We further verify our assumption by experimental investigation of W and (Nb+Hf+Zr) influence on the mechanical properties and microstructure of a Cr-base alloys. We observed an increasing strength properties due to W addition and increasing elongation as a result of (Nb,Hf,Zr) alloying. [1] V.N. Butrim et al. Adv. Mater. Res. 1119 (2015) 569-574

1042 Evaluation of coating thickness on the cutting sharpness and durability of Zr-based metallic glass thin film coated surgical blades

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The cutting sharpness and durability of commercial blades without and with 200-500 nm thicknesses of Zr-Cu-Al-Ag-Si and Zr-Cu-Ni-Al-Si metallic glass thin film (MGTF) coating were systematically characterized and evaluated. The amorphous states of all MGTF coatings were examined by grazing incident X-ray diffraction analysis. The observation of scanning electron microscopy reveals that MGTF coated blades own much smoother surface than the commercial one which indicates smaller friction force during cutting process. Results of sharpness test in the beginning reveal that all MGTF-coated blades possess smaller value of blade sharpness index (BSI) than the commercial one. The cutting durability of the MGTF-coated and commercial blades was evaluated by the variation of BSI values by 25 cm cutting test. The commercial blade was blunted (BSI=0.49) after 20 cm cutting path. Conversely, the 300 nm-thick MGTF-coated blades present the smallest BSI value after 20 cm cutting path in comparison with other thicknesses MGTF-coated blades and commercial blade. As a result, the

cutting durability of surgical blade can be significant improved by the MGTF-coating, can keep the BSI value less than 0.45 after 20 cm cutting test.

1043 Reverse transformation behavior induced by shot-peening for SUS410S martensitic stainless steel

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When metallic materials are shot-peened on their surface, deformation-induced layer with nanocrystalline structure is formed due to huge shear strain. In previous study, it has been reported that reverse transformation from martensite to austenite occurs by shot-peening of SUS304 austenitic stainless steel and Fe-Ni alloys which contain strain-induced martensite. However, it is not clear that the above reverse transformation by shot-peening is a phenomenon peculiar to strain-induced martensite. In this study, SUS410S martensitic stainless steel is selected to study the phase transformation behavior of thermally-induced martensite by the shot-peening. It is found that volume fraction of austenite is increased by the shot-peening. This means that thermally-induced martensite also transforms into austenite by the shot-peening. Moreover, volume fraction of austenite at peened-surface is found to be increased as distance between nozzle and specimen decreases, since the larger shear strain can be induced. In addition, thickness of the deformation-induced layer has larger by decreasing the distance. It can be concluded that the reverse transformation of thermally-induced martensite in SUS410S martensitic stainless steel occurs by large shear strain during the shot-peening.

1044 Alloy anodization towards the structural and compositional design of nanostructured oxide layers

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The formation of nanostructured materials has attracted much attention due to their excellent properties compared to bulk ones. It is well known that anodization is often carried out in various acidic electrolytes in order to form highly ordered nanoporous oxide layers on aluminum. However anodization of the other metals than aluminum in acidic electrolyte leads to the formation of compact flat oxide layer or porous layers with random size distribution and arrangement. The key to form oxide layers consisting of ordered pore or tube arrays on the other metals is the addition of fluoride species to electrolyte, which induces the partial dissolution of electrochemically formed oxide layers. As a result the initiation and growth of nanoporous and nanotubular oxide layers are facilitated. So far it has been demonstrated that the morphology of anodic porous and tubular oxide layers can be tuned by tailoring anodization conditions such as anodization voltage, electrolyte composition and temperature and optimized anodization leads to the formation of extremely high aspect ratio porous and tubular oxide layers. Furthermore anodic porous and tubular oxide layers have been applied in many fields, focused on utilizing their high surface area. Recently the compositional modification of anodic porous and tubular oxide layers, i.e., decoration of the anodic layers with secondary functional material or doping secondary element into the layers has been studied to improve properties of the

oxides. This presentation reports alloy anodization as one of the approaches to achieve structural and compositional design for nanoporous and nanotubular oxide layers.

1045 Stabilization of retained austenite by carbon and nitrogen in Q&P processed martensitic stainless steel

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Quenching and partitioning (Q&P) process is effective for dispersing a significant amount of retained austenite in low carbon martensitic stainless steel, and it was revealed that the mechanical property of the Q&P processed alloy could be markedly improved owing to TRIP effect. Since this kind of alloys hardly undergo austenite to ferrite diffusional transformation during the process due to the large amount of Cr, carbon (nitrogen) partitioning proceeds under a nearly ideal condition (near CCE). As another characteristics of Q&P process of stainless steels, it should be noted that not only carbon but also nitrogen can be utilized as an austenite stabilizer, which is not available in the low alloy TRIP steels. By replacing carbon with nitrogen, some merits would be expected particularly in terms of corrosion resistance, and also, various changes might be caused in morphology of microstructure, stability of austenite, thermodynamic equilibrium condition, kinetics of interstitial diffusion, and so on. In this study, Fe-12Cr-0.1C and Fe-12Cr-0.1N alloys were prepared to investigate the difference between carbon and nitrogen in optimized condition for obtaining the maximum amount of retained austenite, and then the austenite stabilizing effect was compared in consideration of the difference in Ms temperature between both alloys.

1046 Nanoindentation study on incipient plasticity in the vicinity of grain boundaries in nickel and sulfur-doped nickel

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Grain boundaries significantly influence the mechanical properties of polycrystalline materials. Recent studies using a nanoindentation technique show that grain boundaries can act not only barriers to glide lattice dislocations but also as effective sources of lattice dislocations. However, little information is available for the influence of impurity segregation to grain boundaries on the local plastic deformation in the vicinity of grain boundaries. The motivation for this study is therefore to reveal incipient plastic deformation near the impurity-segregated grain boundaries. Nanoindentation tests were performed at well-characterized grain boundaries in pure and sulfur-doped polycrystalline nickel. Pop-in events associated with the grain boundaries were observed in the load-penetration curves. We found that the critical stress for dislocation generation at grain boundaries in pure nickel depended on the grain boundary character; the special boundaries possessed a higher critical stress than the general boundaries. Sulfur segregation to grain boundaries in nickel caused a decrease in the critical stress for dislocation generation at grain boundaries. The decrease of the crystal stress was more pronounced at the special boundaries than the general boundaries, making the critical stress less depend on the grain boundary character. On the other hand, the grain boundary hardening was

more significant at the general boundaries than at the special boundaries. Of particular interest is a finding that the higher the degree of grain boundary hardening, the lower the critical stress for dislocation generation at grain boundaries.

1047 Hybrid quantum/classical simulations for dopant segregation and optical response of nanomaterials

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We present combined approaches of quantum and classical atomistic simulations for simulating effects of nanoscale modification on materials. Two types of methodologies have been developed; a hybrid density-functional/classical molecular dynamics method and a classical finite-difference time-domain method coupled with the time-dependent Schrodinger-equation. The former method has been applied to atomistic and electronic analyses of structure of dislocations (edge and screw) in α - Al_2O_3 and effects of dopant segregation to the dislocations. Results show a stable inter partial-dislocation distance and its excellent agreement with recent HRTEM analysis [1]. An Er segregation into a screw dislocation in alumina takes place with unconventional character in determining stable dopant positions. The latter approach has been developed for simulating effects of defects/dopants in nanomaterials on its optical responses. We have designed a graphene array on a SiO_2 substrate for controlling electromagnetic reflection with biased gates [2]. In practice, the graphene ribbons may contain defects and/or contaminants which degrade wave-front control of the device. The present method incorporates such effects by dealing explicitly with the electronic structure via quantum simulation. We demonstrate usefulness of the methodology by applying it to effect of a defect on the optical response of graphene ribbons. This work was supported in part by the Elements Strategy Initiative for Structural Materials (ESISM) via the MEXT by Japan. [1] K. Tsuruta, E. Tochigi, Y. Kezuka, K. Takata, N. Shibata, A. Nakamura, and Y. Ikumura, *Acta Mater.* 65, 706 (2014). [2] T. Yatooshi, A. Ishikawa, and K. Tsuruta, *Appl. Phys. Lett.* 107, 053105 (2015).

1048 Effect of chemical state of silver added to calcium phosphates on dissolution behavior, antibacterial activity, and cytotoxicity

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Excellent bone compatibility has allowed the widespread application of calcium phosphates (CaPs) such as hydroxyapatite (HAp) and tricalcium phosphate (TCP) as materials for bone filling, bone grafting, coating, and so similar applications. Since infection due to the bacteria attached on the surface of implant material is concerning, addition of silver (Ag) to CaPs has been studied. Dissolved Ag ions possess antibacterial activity. In our previous study, Ag-incorporated beta-TCP (TCP-iAg) and HAp with metallic Ag (HAp-mAg) were prepared by precipitation and sintering. In this study, the dissolution behavior, antibacterial activity, and cytotoxicity of Ag-containing CaPs were evaluated. Immersion test was performed for the sintered compacts by using Tris-HCl solution (pH 7.4). The specimens were incubated in

Escherichia coli suspension, and the number of viable bacteria after specific durations was counted to evaluate the antibacterial activity. The cytotoxicity of sintered compacts was assessed based on ISO 10993-5 guidelines by using V79 cells. Amount of Ag ions in the Tris-HCl solution from TCP-iAg was higher than that from HAp-mAg. We noted a decrease in the viable bacterial number for sintered compacts, including TCP-iAg; on the other hand, there was no change in the viable bacterial number for HAp-mAg and sintered compacts without Ag. The Ag ions released on the dissolution of β -TCP are thought to promote antibacterial activity. No cytotoxicity against V79 cells was observed in case of Ag-containing β -TCP and/or HAp sintered compacts.

1049 In-situ measurement of resistivity in pure titanium during elastic/plastic deformations

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Understanding of microstructures and deformation behaviours in titanium is very important for employing the materials appropriately in biomedical/healthcare applications because available elements are extremely limited. The quantitative evaluation of microstructures and lattice defects can be performed by several techniques. It is well known that resistivity is sensitive to microstructural change and introduction/annihilation of lattice defects. However, the dimensions of the specimen must be accurately measured in order to obtain the resistivity from four-point DC electrical resistance measurements. This means that the resistivity cannot be measured in irregularly shaped specimens, for example, under tensile deformation. However, resistivity can be estimated using the empirical relationship. The purpose of this study was to determine Matthiessen's empirical relationship and to estimate changes in resistivity during tensile deformation of pure Ti to understand the behaviours of lattice defects under deformation. A characteristic texture was developed in the obtained plate; two accumulations of (0001) pole appeared symmetrically on the plate. The angle between the poles was about 90-100°. Resistance measurement was performed during tensile deformation for the specimens prepared from the plate. Basically resistance increased with the tensile deformation in both elastic and plastic regions. However, in elastic deformation, strong anisotropy of resistance could be confirmed to appear depending on the tensile direction to the texture; the resistance decreased with deformation in the tensile direction perpendicular to the two (0001) poles. Introduction of vacancies could be detected in elastic/plastic deformation. Some of vacancies were swept out by induced dislocations just after the yielding.

1050 Micro-mechanical characterisation of hydrogen embrittlement related to twin boundary in type 304 stainless steel

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Micro-mechanical testing was employed to analyse hydrogen embrittlement related to twin boundary in a type 304 metastable austenitic stainless steel, with a particular focus on deformation-induced martensitic transformation. We performed micro-tensile tests and micro-

compact-tension fatigue tests that allow for testing small single crystals and twinned bi-crystals with defined crystallographic orientations. The crystallographic orientations were determined using electron backscatter diffraction (EBSD) analysis. Micro-tensile specimens with a gauge section of $20\mu\text{m}\times 20\mu\text{m}\times 50\mu\text{m}$ were fabricated using focused ion beam. Single-crystalline (SC) specimens and twinned bi-crystalline (TW) specimens were prepared with their loading directions (LDs) parallel to $[111]$ direction. For the (TW) specimens, the twin boundary was set perpendicular to the LD. A set of specimens were cathodically charged with hydrogen. Micro-tensile testing was performed at room temperature in air and at a displacement rate of $0.1\text{ }\mu\text{m/s}$. In the SC and TW specimens, hydrogen increased the flow stress but decreased the elongation-to-failure. The fracture surfaces of the hydrogen-charged SC and TW specimens exhibited a quasi-cleavage and a planar facet, respectively. The planar facet corresponds to the twin boundary. Also, liner steps were observed in three directions with a mutual angle of 120° in the hydrogen-charged TW specimen. EBSD observations after fracture revealed that variants having the habit plane parallel to the primary slip plane were formed in each crystal. We will discuss the mechanism for the formation of the planar facet, combined with the result of the fatigue crack growth test of the hydrogen-charged TW specimen.

1051 Rotary bending fatigue behavior of selective-laser-melted Type 630 stainless

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Rotary bending fatigue tests were conducted using selective-laser-melted (SLM) Type 630 stainless steel and conventionally-melted Type 630. Based on the results, the fatigue behavior and the fracture mechanism were discussed. Acicular martensite was uniformly distributed in the microstructure of the conventionally-melted Type 630, while martensite-poor, namely ferrite-rich areas were recognized in the SLM specimen. Because of the additive lamination of melted powders, relatively high temperature was kept during SLM process, resulting in the prevention of full-martensite structure. The hardness of SLM specimen was lower than that of conventionally-melted one. In addition, some pores with the size of $70\text{-}100\mu\text{m}$ were distributed in the SLM specimen. It indicates that the pores were formed during additive lamination of melted powders. The fatigue strength of SLM specimen was much lower than that of conventionally-melted one. It is attributed to the lower hardness of SLM specimen with ferrite-rich structure. Furthermore, the fatigue crack initiation from defects had strongly detrimental effects on the fatigue strengths in the SLM specimen. In order to improve the fatigue strength, heat treatment ($1050\text{ }^\circ\text{C}$ for 4 hour, followed by water quenching) was examined for the SLM specimen. As a result, the fatigue properties of the SLM-quenched specimens was slightly improved, but was still lower than that of the conventionally-melted one.

1052 Multiscale modeling of deformation and fracture in polymers

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Polymer materials are drawing much attention among both science communities and industrial circles due to their various applications including structural materials for vehicles and airplanes, tires, battery separators, and so forth. Thus, there is growing importance of understanding deformation and fracture mechanisms in polymers to design materials with improved reliability. In this study we work on building proper modeling methods and perform simulations to tackle the problems of deformation and fracture in polymers. First, we introduce a finite-element method (FEM) modeling of fracture in rubbers. Here we focus on dynamic crack propagation in highly viscoelastic materials. To perform FEM simulations of fracture in rubbers, it is essential to adopt proper constitutive laws of viscoelasticity and fracture criteria. We will demonstrate FEM simulation results using constitutive laws based on experiments and discuss the validity of the computational approach. One of the major challenges in computational simulation of polymers is the difficulty in constructing appropriate constitutive laws in continuum modeling. As an effort to overcome this problem, we introduce an approach of 'bottom-up' multiscale simulation. We start from all-atom molecular dynamics (MD) to coarse-grained MD, trying to establish the constitutive law to describe deformation behavior and fracture criteria. We devised a sophisticated modeling scheme of constructing energy functions used in the coarse graining model. We will show results of polycarbonate as an example of our approach. We gratefully acknowledge financial support by ImPACT Program of Council for Science, Technology and Innovation (Cabinet Office, Government of Japan).

1053 Effects of cross-rolling on deformation texture evolution in unalloyed titanium

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Unalloyed titanium (CPTi) was rolled with 20% reduction in each pass at 293 K using a cross-roll mill. Multi-pass flat-rolling was carried out without any lubricants up to the true strain of 1.0, where two kinds of rolling directions such as tandem (uni-direction for all passes) and reverse (opposite direction in every passes) were adopted. The upper and lower rolls were crossing each other, and their rolling axes were at an angle of 0, 5 or 10 degree with parallel position. Strain of samples was increased proportionally as higher passes regardless of the rolling conditions. The transverse direction (TD) split deformation texture in titanium was generally developed under the cross angle of 0 degree. In the present strips of tandem, a main orientation was identified as (-12-18)[10-10]. In the case of tandem with the cross angle of 5 degree, a fiber texture was developed along (-12-18). That is the reason why a rotation in the rolling direction (RD) was overlapped. In the case of reverse with the cross angle of 5 degree, the main orientation was separated into [10-10] and [2-311] which were corresponded to TD and RD splits, respectively.

1054 Heat treatment of biomedical Ni-Ti alloys – towards a one-step procedure for optimizing biocompatibility, pseudo elasticity and dimensional accuracy

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Therapy of cardiovascular diseases increasingly relies on implants made of pseudoelastic and biocompatible Ni-Ti alloys. The material's unique properties profile allows for construction of self-deploying minimally invasive devices that are positioned via a catheter, thus minimizing health hazard. In recent years, numerous attempts were undertaken to further optimize specific properties of NiTi bulk and surface towards biomedical applications utilizing sophisticated heat treatment procedures. However, research initiatives often focus on a single property, e.g. surface chemistry, and neglect essential further aspects, e.g. a shift of phase transformation temperatures and characteristics that may get altered simultaneously. In the present work, the effect of heat treatments is investigated considering bulk and surface properties of Ni-Ti. Simultaneously occurring surface oxidation and changes of phase transformation temperatures were determined in a quantitative manner. It is demonstrated that subtle changes of the heat treatment regime can be utilized for optimizing the surface of NiTi regarding its biocompatibility, whereas the phase transformation temperatures are shifted off limits, simultaneously, rendering the final implant useless for medical application. The potential for optimizing the material's properties profile towards biomedical applications using a single heat treatment procedure will be discussed considering surface chemistry, thickness of the oxide layer, shift of transformation temperatures and dimensional accuracy of shape-setting.

1055 Multi-stimuli responsive polymer gels via initiated chemical vapor deposition

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Polymer hydrogels are chemically modified to obtain a smart material that respond to more than one stimulus. In details, responsiveness to light irradiation and exposure to aqueous environment is combined to tune the mechanical properties and the degree of swelling within thin hydrogel films. Synthesizing hydrogels or modifying their surface with solution-based methods is often problematic due to swelling of the solvent in the hydrogel. This issue is completely circumvented by the use of a novel and solvent-free technique, named initiated Chemical Vapor Deposition (iCVD). The starting hydrogel film is a cross-linked polymer of 2-hydroxyethyl methacrylate (HEMA). The surface of the hydrogel is chemically modified by iCVD to introduce perfluorophenyl groups. The addition of the pendent perfluorophenyl functionality is confirmed by Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS). The modified hydrogel swells in water from 7 to 12 % depending on the percentage of cross-linker and in humidity in the range 2 to 30 %. By a post deposition reaction the perfluorophenyl groups are reacted with 4-Aminoazobenzene. Through photoisomerization of the azobenzene, the polarization within the hydrogel can be modified and therefor the affinity to water increases. A clear correlation between irradiation and swelling properties of the hydrogel is demonstrated. The materials are all bio-compatible and therefore suitable for a great variety of applications such as sensors, in pharmaceuticals or for cell cultures.

1056 Quantitative evaluation of dislocation nucleation in magnesium via atomistic simulations

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Recently developed Mg-Zn-Y alloy contains a phase with the LPSO structure. In phases with the LPSO structure, kink bands are observed and the overwhelming strength of this alloy is attributed to the existence of kink bands. Kink band formation was observed not only in the LPSO structure but also in other layered materials. Mechanism of kink band formation has been proposed by Hess et al. through the observation of compressive deformation of zinc single crystals. In their mechanism, nucleation of basal dislocation in the crystal acts as the elementary process of kink band formation. However, at the present moment, we do not possess sufficient information to judge the validity of this mechanism. Quantitative evaluation of dislocation nucleation gives us important information for this judgement. Accordingly, we have developed the method for quantitative evaluation of dislocation nucleation in the crystal via atomistic simulations. We applied this method to magnesium single crystals in order to reveal the dependence of the activation free energy of dislocation nucleation on the applied stress and the temperature. One of our findings through this work is that the compressive stress in [11-20] direction facilitates the basal dislocation nucleation, which may affect kink band formation in the LPSO structure. In this presentation, we will also show the results obtained by applying the method to more complex model, that is, Mg-Zn-Y ternary alloy consisting of LPSO structure.

1057 Atomic-scale modeling of point defects, phase stability, and the formation mechanism of Z phases CrMN (M=V, Nb, Ta)

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The challenge of raising the steam inlet temperature of fossil fired power plants calls for creep-resistant steels with a Cr content higher than 9% in order to achieve sufficient corrosion and oxidation resistance. However, it has been found that in 11-12% Cr ferritic-martensitic creep resistant steels strengthened by fine (V,Nb)N particles, precipitation of thermodynamically stable Z-phase particles, CrMN (M=V,Nb,Ta), in long-term service is unavoidable and detrimental. Usually, Z-phase particles are coarse and brittle and grow at the expense of the desired fine (V,Nb)N particles. A promising solution to this problem is provided by the idea to exploit the Z-phase as a thermodynamically stable strengthening agent. Hence the challenge is to control the precipitation of the Z-phase such that fine and long-term stable particles are formed. We present atomistic simulations, using density function theory, which reveal the essential mechanisms underlying the formation of Z-phases. The picture that evolves consists of the diffusion of Cr atoms into MN particles and their subsequent clustering in a layered arrangement which finally yields the transformation of the nitride particles to Z-phase particles. We study the thermodynamic stability of Z-phase, related structures and predecessors as well as the basic phase formation mechanisms.

1058 Investigation of microstructure evolution and phase transformations in ultra-fine grained metastable beta Ti alloys

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The main aim of this study to analyze the effect of the severe plastic deformation (SPD) on the microstructure and mechanical properties of metastable beta Ti alloys. Experiments were performed on two different alloys: Ti-15Mo and Ti-6.8Mo-4.5Fe-1.5Al which were subjected to severe plastic deformation (SPD) by high pressure torsion (HPT) and equal channel angular pressing (ECAP). The effect of ultra-fine grained (UFG) structure on phase transformation was studied. The UFG microstructure was observed by scanning and transmission electron microscopy (SEM and TEM). The increase of hardness with increasing equivalent strain was determined by microhardness mapping. Structure of lattice defects was further studied by advanced techniques of positron annihilation spectroscopy (PAS).

1059 Influence of heat treatments on the behavior of maraging steels in phenomena of hydrogen embrittlement

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Hydrogen Embrittlement (HE) in 18 Ni (250) maraging steel with different extent of aging was studied. Heat treatments were carried out at a temperature of 480 °C in order to obtain under-, peak- and over-aged specimens. Hydrogen effective diffusivity (D_{eff}) was determined via a solid-state sensor and hydrogen solubility (C_H) was measured via thermal conductivity detector. Hydrogen diffusivity in the decreasing order was observed to be under-, peak- and over-aged conditions, while solubility has an opposite trend. As the time of aging enhance, precipitates gradually grow and increase, while retained austenite forms. In over-aging condition, the numerous incoherent precipitates and retained austenite are strong and irreversible traps for hydrogen, leading to a smaller diffusivity and greater solubility. Tensile tests were performed under a slow strain rate to evaluate the mechanical strength loss in specimens after their electrochemical hydrogen charging in an alkaline solution; moreover, SEM analyses were carried out to characterize fracture morphology. The tensile tests showed that all aged specimens were susceptible to HE, to different degrees. Peak-aged specimen was the most susceptible to HE, with a fragility index ($F\%$) calculated regard to σ_{UT} of 40,6%; over-aged specimen was the less susceptible ($F\%$ of 3,2), in spite of the larger quantity of hydrogen absorbed under the same hydrogen charging condition. In over-aged specimen irreversible hydrogen traps, like retained austenite, impeded hydrogen transport toward the strained regions, thus, the resistance to HE was improved. Here, retained austenite presence in over-aged steel is proved via TEM analyses.

1060 Anisotropic characteristics and constitutive modelling of Ti6Al4V sheets deformed at elevated temperature and strain rate

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Tensile tests were performed at elevated temperature and strain rate in order to investigate the plastic flow behaviour, anisotropic characteristics and microstructural evolution of Ti6Al4V sheets under testing conditions similar to the ones experienced during hot stamping operations, but far different from the ones typical of the titanium superplastic regime to which several literature records are devoted. It is shown that the Ti6Al4V anisotropic characteristics under the investigated forming conditions are influenced by the variation of the material texture as a function of the testing temperature as well as the flow stress behaviour by the deformation temperature, strain rate and rolling direction. On the basis of the flow stress data, two different constitutive models, namely the Johnson-Cook and the Arrhenius ones, were proposed to predict the behaviour of Ti6Al4V sheets deformed at elevated temperature and strain rate. The comparison with the experimental results suggests that the Arrhenius model guarantees a better accuracy in predicting the flow stress behaviour of Ti6Al4V than the Johnson-Cook one.

1061 Neutron and synchrotron studies on self healing of creep damage in Fe-based alloys

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When exposed to moderate stress levels at elevated temperatures for long times steel components can exhibit creep fracture, which arises from the formation, growth and coalescence of (initially) nanoscale pores at the grain boundaries. Self-healing of such pores is a promising new approach to enhance the component lifetime. We demonstrate that self healing is achieved by precipitation of substitutionally dissolved Au and Mo atoms in the iron lattice on the creep cavity surface slowing down or even stopping further growth. The creep behaviour and microstructure evolution is studied for ferritic Fe-Au and Fe-Mo model alloys for different applied stress levels at a constant temperature of 550 °C. We found that an efficient self healing of creep damage can be achieved in the Fe-Au alloy by selective precipitation of solute Au at the creep cavity surface [1]. The self healing of creep damage by site-selective Au precipitation results in an improved creep life time. A similar site-selective precipitation at creep defects was also observed for solute Mo in the Fe-Mo alloy. The mechanism for the improved creep lifetime is clarified further by microstructural studies of the Fe-Au and Fe-Mo alloys after creep failure using Small-Angle Neutron Scattering (SANS) [2] and synchrotron nano X-Ray Tomography, complemented with Electron Microscopy (SEM, TEM, EBSD, EBSD) and Atom Probe Tomography (APT). For lower stress levels pore filling fractions of up to 80% have been observed. [1] S. Zhang et al., Adv. Eng. Mater. 17 (2015) 598. [2] S. Zhang, et al., Acta Mater. 61 (2013) 7009.

1062 Early instability phenomena of IN792 DS superalloy

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The microstructural stability of the directionally solidified Ni base IN792 superalloy has been investigated by Mechanical Spectroscopy (MS), i.e. internal friction (IF) and dynamic modulus measurements. Repeated IF test runs from room temperature to 1173 K have been carried out on the same samples and a Q^{-1} maximum has been always observed above 700 K. Its position does not depend on the resonance frequency. After each run the values of modulus and Q^{-1} at room temperature change indicating that a progressive irreversible transformation occurs. Damping phenomena have been attributed to the rearrangement of dislocation structures in disordered matrix which modifies dislocation density and average distance of pinning points. The results are supported by X-ray diffraction (XRD) and transmission electron microscopy (TEM) observations.

1063 Reactive nanosystems: Billion atom reactive and quantum molecular dynamics simulations

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Billion atom molecular dynamics simulations are used to investigate critical issues in the area of structural and dynamical correlations, and reactive processes in nanostructured materials under extreme conditions. Cavitation bubbles readily occur in fluids subjected to rapid changes in pressure. We use billion-atom reactive molecular dynamics simulations on a 163,840-processor BlueGene/P supercomputer to investigate chemical and mechanical damages caused by shock-induced collapse of nanobubbles in water near silica surface. Collapse of an empty nanobubble generates high-speed nanojet, resulting in the formation of a pit on the surface. The gas-filled bubbles undergo partial collapse and consequently the damage on the silica surface is mitigated. Quantum molecular dynamics (QMD) simulations are performed on 786,432-processor Blue Gene/Q to study on-demand production of hydrogen gas from water using Al nanoclusters. QMD simulations reveal rapid hydrogen production from water by an Al nanocluster. We find a low activation-barrier mechanism, in which a pair of Lewis acid and base sites on the Al_n surface preferentially catalyzes hydrogen production. I will also discuss on-demand production of hydrogen gas from water using and LiAl alloy particles. Research reported in this lecture was carried in collaboration with Rajiv Kalia, Aiichiro Nakano and Ken-ichi Nomura from the University of Southern California, and Fuyuki Shimojo and Kohei Shimamura from Kumamoto University, Japan.

1064 Effect of heat treatments on TiH₂ surface composition and hydrogen release

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Titanium hydride (TiH₂) is an attractive material for many technological applications due to its excellent capacity of hydrogen storage, and was shown to be a suitable blowing agent for aluminium alloys foams. In spite of numerous research works, its decomposition with consequent hydrogen release is still not understood completely. The present work was carried out to investigate the effect of heat treatments performed in various atmospheres on phase transformations and surface structure of TiH₂ resulting from hydrogen desorption.

Untreated TiH₂ has been characterized by Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and X-Ray Photoelectron Spectroscopy (XPS) with an aim to describe the shape, size and size distribution of particles, the phases and the chemical state of particle surface. XRD at increasing temperatures permitted to identify the sequence of phase transformations when TiH₂ is heated both in Ar and air. Hydrogen release has been quantitatively measured by Temperature Programmed Desorption technique (TPD) to find a correlation with the specific structure. The same examinations were made on TiH₂ previously submitted to oxidative treatments at different temperatures and for increasing treatment time, both in static and gas flow atmospheres. The thickness of the oxide layer on TiH₂ surface, the nature of oxides in this layer and their spatial distribution has been investigated through XPS depth profiles. Experimental results provide a clear picture of bulk and surface characteristics of TiH₂ in relation to hydrogen desorption after the examined heat treatments. On this basis the theoretical models proposed in literature are critically discussed.

1065 Texture gradient through thickness of a cross roll-bonded aluminum composite

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A common feature in rolled aluminum sheets is the texture gradient because of shearing occurring at the sheet surface in contact with rolls. The common shear texture component in aluminum is the rotated-Cube one {001}<110>[1]. The present work aims to study the gradient evolution after cumulated cross-rolling (90° rotation from the rolling direction) on an AA5754/AA6061 composite at room temperature. The composite's microstructure observed from EBSD measurements after the first roll-bonding shows that the AA6061 layer displays a deeper shearing area (embodied by the rotated-Cube component). Typical rolling components (S {123}<634>, Dillamore {4 4 11}<11 11 8> and Brass {011}<211>) are present in the thickness of both aluminum alloys. After the stacking and the cross-rolling, the four layers (Al6061/Al5754/Al6061/Al5754) are greatly heterogeneous: each of them has its own texture. In addition, because of the 90° rotation, the Brass component turned into the ND rotated-Brass {011}<755> one [2]. At the surface, components are the same as after the first rolling. In the thickness, the rolling components remain whereas near the last interface in AA6061, a large area with the major Dillamore component stands. This component evolution corroborates VPSC simulation [3]: the stacking prior to the cross-rolling redistributes the rotated-Cube

component in the thickness and then the cross-rolling turns it into the Dillamore component.

[1] L. Su, et al, J. Alloys Compd. 594 (2014) 12–22. [2] S.-H. Hong et al, J. Eng. Mater. Technol. 124 (2002) 13–22. [3] H. Azzeddine et al, Mater. Charact. 97 (2014) 140–149.

1066 All-solid-state argyrodite-based lithium batteries

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Several sulfide glasses, glass-ceramics or ceramics have been developed as promising electrolytes for all-solid-state Li-ion batteries. Ionic conductivity values ranging, at 298 K, from 10^{-4} to 10^{-2} S/cm have been reported with electrochemical stability windows up to 10 V (vs Li/Li⁺). These solid electrolytes have been demonstrated suitable to fabricate all-solid-state batteries with different electrode materials (LiCoO₂, Li₄Ti₅O₁₂). The so-called Li-Argyrodite Li₆PS₅X (X= Cl, Br, I) family presents ionic conductivity values up to $7 \cdot 10^{-3}$ S/cm at 298K. The Br-containing Li-Argyrodite has been used to fabricate all-solid-state batteries in between carbon coated Li₄Ti₅O₁₂ (c-LTO) and Li-Al alloy as positive and negative electrode materials, respectively. We developed a straightforward route for the synthesis of the three phases Li₆PS₅X (X= Cl, Br, I), by mechanical milling. The study of the lithium/electrolyte interface showed a good stability of the Li₆PS₅Cl phase towards lithium plating and stripping, the Br- and I-containing phases being less stable. Nevertheless, at high current density, losses of electric contact have been observed and appear to be related to dendrites formation. LiCoO₂ and Li₄Ti₅O₁₂ half-cells using Li₆PS₅Cl as the solid electrolyte exhibit a capacity of 125 mAh/g for both materials at 0.064 mA/cm² and ambient temperature. All-solid-state full-cells operating at room temperature were assembled and the optimization of several assembly parameters lead to batteries delivering a capacity of 100 mAh/g of LiCoO₂ for extensive cycling at 10 C rate, at 150°C .

1067 High porosity titanium coatings by cold spraying for photocatalytic water splitting

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Recently, hydrogen production via photocatalytic water splitting has attracted remarkable attention and has been extensively studied because of its great potential for low-cost and clean hydrogen production. The photocatalytic material should absorb the photons efficiently, it should facilitate the separation between holes and electrons and it should be electrically conductive to transport the generated charges and avoid the recombination of holes and electrons. Well known photocatalytic semiconductors are TiO₂ or BiVO₄. A high electrical conductivity and a large specific surface can be achieved by porous metal coatings. The photocatalytic properties can be assured by semiconductor oxides layers as TiO₂. In the present case, the performance in water splitting is studied for cold gas sprayed porous titanium coatings covered with thin films of TiO₂. Cold gas spraying is mainly used to produce dense metallic coatings from solid particles aiming for similar properties as the bulk material. In the present, work cold spraying of porous titanium coatings and respective characterization is in focus for optimizing photon efficiency. Metallographic characterization was performed by quantitative

image analysis using optical microscopy porosity data and SEM analysis for evaluating the coating/substrate interface integrity. For processing photocatalytically active semiconductor layers, porous titanium coatings were thermally oxidized for the duration of 4 hours to obtain pure TiO₂- Anatase, which is photocatalytically active under UV-light irradiation and can be used for water splitting. The photoactivity of the different coatings is discussed under viewpoints of needed thickness and optimum porosity for the use in water splitting.

1068 High heating rates and their influences on austenite formation

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Fast industrial processes are of wide interest in industry. Short processing times, which lead to comparable microstructures and mechanical properties as for longer processes, can result in a desired higher productivity. Induction hardening is one example for a fast heat treatment, which can be up to 10 times faster than a conventional process. The inductive process uses a fast austenitizing with heating rates up to 100 K/s and short holding times at austenitizing temperature, which are the main differences to conventional heat treatments. This leads to accelerated kinetics of the ferrite to austenite phase transformation and thus increased A_{c1} and A_{c3} temperatures. Furthermore, during fast heating carbon volume diffusion is the transformation-dominating step over the more sluggish diffusion of substitutional atoms. The aim of this work is to analyze the consequences of different heating rates on the austenite formation of a 50CrMo4-steel with different prior microstructures by kinetic transformation simulation. DICTRA is used for the simulation of ferrite to austenite transformation at different heating rates, employing the local equilibrium approach at a moving interface. Simulated phase fractions and transformation kinetics are compared with dilatometric experiments. Validated simulation results are used as input data in subsequent precipitation simulations of further 50CrMo4-steel processing steps with MatCalc. Consequences of heating-rate dependent carbon segregation on the as-quenched state are discussed.

1069 Decomposition of beta-quenched microstructures during linear heating of titanium alloys

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The phase transformation kinetics of the alpha+beta Ti-6Al-6V-2Sn (Ti-662) and metastable beta Ti-5Al-5Mo-5V-3Cr-1Zr (Ti-55531) alloys quenched from the beta field is studied as a function of heating rate using in situ high-energy synchrotron X-ray diffraction. Quantitative phase analysis based on the Rietveld method provides the continuous evolution of phase volume fractions and lattice parameters, revealing variations in the phase transformation sequence. Moreover, the investigations are complemented with metallographic analysis to evaluate the phase transformation mechanisms occurring at different heating stages. The initial microstructure for the Ti-662 alloy consists basically in a matrix of equiaxed beta grains fully transformed into orthorhombic alpha" martensite, while the Ti-55531 alloy presents an initial

configuration formed by equiaxed metastable beta grains with dispersed omega athermal particles. During the first stage of slow heating spinodal decomposition of alpha" martensite and beta matrix takes place for the Ti-662 and Ti-55531 alloys, respectively. For the latter, this effect occurs together with diffusion-driven formation of the metastable phases omega and isothermal alpha". Furthermore, the results show that the stable alpha phase can form via three different paths: slow heating rates provide homogeneous distribution of fine alpha plates formed through the evolution of (a) the orthorhombic alpha" martensite and (b) omega phase, while (c) fast heating rates may suppress this last mechanism and promote the formation of alpha from beta grain boundaries.

1070 Can heat treatment improve duplex electroless nickel coatings?

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Electroless nickel coatings are used in several industries because of their good properties: nickel-phosphorous is particularly used for its good corrosion resistance while nickel boron has better mechanical properties. Duplex coatings combining two layers of electroless nickel appear thus as a way to combine the advantages of both types of coatings. This was studied on as-deposited coatings and improvement was observed for some properties but not all. In this talk, the mechanical and tribological behaviour of duplex coatings after heat treatment in slightly reducing atmosphere (95% Ar- 5% H₂) at 400°C for one hour will be discussed. Those properties have been investigated by microscopy, hardness testing, roughness measurements, scratch tests and wear testing (abrasion and pin on disc).

1071 Microstructural and mechanical comparison of Ti and Ti-alloys after severe plastic deformation

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Titanium and its alloys are heavily researched as implant materials, due to their preferential bio-inertness and bio-compatibility. There is the need to increase the strength of some materials for the application as a load bearing implant. One way to do that, is severe plastic deformation, for example by high pressure torsion (HPT). The change in mechanical behavior was investigated for Ti, Ti45Nb (beta-Ti alloy) and Ti13Nb13Zr (alpha+beta-Ti alloy). The fracture mechanical investigation revealed a strong direction dependence of the fracture toughness of the HPT-deformed materials. Additionally, the investigated materials showed a very good strength to fracture toughness ratio. Microstructural investigations showed for the deformed Ti45Nb no phase transformation. This is in contrast to the observed formation of the omega-Ti phase in Ti and Ti13Nb13Zr. Thus, the deformed Ti45Nb shows great promise as an implant material because of its increased strength and sufficient fracture toughness without a significant change in Young's modulus.

1072 Atomic structure and dynamics of bulk metallic glasses

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We have used coherent electron nanodiffraction to investigate the nanometer-scale structure and dynamics of bulk metallic glass alloys. Spatial fluctuations in diffraction in the form of fluctuation electron microscopy show that both Zr-Cu-Al and Pd-Si metallic glasses contain two structure types with different approximate local rotational symmetry. Regions with four- and six-fold approximate symmetry are we call “crystal-like”; regions with odd-order or limited rotational symmetry are non-crystal-like. In the Zr-Cu-Al system, higher glass forming ability is correlated to reduced thermal stability of crystal-like regions. Temporal fluctuations in diffraction in the form of electron correlation microscopy measure the structural relaxation time τ in the supercooled liquid state of glass-forming alloys with nanometer spatial resolution. Initial measurements on $\text{Pd}_{40}\text{Ni}_{40}\text{P}_{20}$ result in $\tau(T)$ is approximate agreement with bulk results from calorimetry and dynamic mechanical analysis. Measurements on $\text{Pt}_{57.5}\text{Cu}_{14.7}\text{Ni}_{5.3}\text{P}_{22.5}$ nanowires with a high-speed direct electron camera at 2 ms time resolution demonstrate the capability to measure relaxation times from nanoscale objects.

1073 Atomistic prediction of relaxation state tuning of metallic glass by pressurized thermal loading process

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Metallic glasses have excellent mechanical properties such as high strength, large elastic elongation. Since the mechanical properties of metallic glasses depend on relaxation state, tuning technique of relaxation state is one of keys to promote practical usage of metallic glasses through further improvement of their properties. In this atomistic study, we show the tuning of relaxation state by thermal and pressure loading process. Thermal loading below T_g generally induces aging. In contrast, we show that the thermal loading above T_g and subsequent fast cooling induces rejuvenation, which is the configurational excitation of glass state. We also show the effect of pressure during thermal loading process on the thermal rejuvenation. Pressure is found to be one of key factors affecting relaxation state of glass system during thermal loading process. We make clear the atomistic structural aspect in rejuvenation of metallic glasses. Moreover, it is demonstrated that the rejuvenation induced by thermal and pressure loading process changes plastic deformation behavior of metallic glasses in uniaxial loading tests and also in nanoindentation tests.

1074 Influence of ageing treatment on precipitation evolution and mechanical properties of 0Cr13Ni8Mo2Al high-strength stainless steel

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The precipitation evolution and mechanical properties of 0Cr13Ni8Mo2Al high-strength stainless steel were investigated in the temperature range from 510 to 620 °C mainly using physicochemical phase analysis and uniaxial tensile and instrumented impact testing, respectively. It is found that different types of nano-scale precipitates appear along with raising ageing temperature. At the low temperature below 565 °C, massive Mo₂C precipitates with needle or rod shape less than 10 nm diameter is separated out inside the grains and at their boundaries, resulting in relatively high strength and low impact absorbing energy. Aged from 565 to 595 °C, irregular M₂₃C₆ precipitates with dimension from 300 to 800 nm, torispherical Ni₃Al precipitates with size from 10 to 20 nm, and spherical NiAl phase with diameter from 30 to 60 nm are dissolved out in the martensitic matrix. Moreover, face-centered cubic Ni₃Al is transferred to body-centered cubic NiAl when temperature is raised in this range. Aged at 595 °C, only M₂₃C₆ and NiAl phase exist in the matrix. During the ageing process the strength of 0Cr13Ni8Mo2Al stainless steels depends on the precipitation and dislocation strengthening via hardening mechanism analysis.

1075 Shock-induced reaction characteristics of an Al/Ni composite processed via accumulative roll-bonding

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Al/Ni composite has been widely recognized as a member of multifunctional energetic structural materials (MESMs), which could release energy due to exothermic chemical reactions initiated under shock loading conditions. In this study, an Al/Ni composite was produced via accumulative roll-bonding (ARB) process up to four passes. Scanning electron microscopy was employed to study the microstructure evaluation during ARB process. Quasi static compression tests and split hopkinson pressure bar tests were conducted to determine the mechanical properties and deformation behavior under various strain rates. The shock-induced reaction behavior of the material was investigated using a quasi-sealed test chamber. The effect of ARB processing on mechanical behavior and shock-induced reaction behavior of the composite was discussed.

1076 Pore-structure adjustment and mechanical property of porous TiAl alloy prepared using titanium hydride and aluminium compact

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Porous TiAl alloy shows excellent properties like high stiffness, oxidation resistance and low density. These features make the open-cell porous TiAl a good candidate for a kind of lightweight materials at high temperatures. In this presentation, porous TiAl alloy have been fabricated successfully by the sintering reaction method using titanium powder mixed with different proportions of titanium hydride and pure aluminium powder compacts. The effects of the titanium hydride ratios on the phase compositions, pore characteristics, expansion and mechanical properties were investigated. The results show that using titanium hydride to replace pure titanium powder has no significant influence on the phase evolution during the sintering process. And the pore structure of porous TiAl alloy, such as porosity and pore size distribution so on, can be adjusted through regulating ratio of titanium hydride to titanium. Furthermore, the oxygen content of the porous TiAl alloy also is reduced, resulting in improved compression strength.

1077 Deformation twinning in HCP metals: Nucleation, growth, and interactions

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Hexagonal close packed (HCP) materials such as Mg, Zr, Ti, and Be are used in automotive, nuclear, aeronautic and defense technologies. The basic HCP plasticity mechanisms are slip and twinning. Dislocation slip is easiest along the $\langle 11-20 \rangle$ compact directions, either on the (0001) basal plane or on the $\{10-10\}$ prismatic planes, depending on the material. These dislocations, however, cannot accommodate deformation along the c-axis. Twinning thus takes place and results the formation of a sheared domain. Twin shear localizes in a defined domain of the grain. Twin nucleation must precede twin propagation and growth. Twin nucleation is driven by local stress states and local atomistic configurations at grain boundaries. While twin propagation is driven by long range stress states across grains through the motion of twin boundaries either by gliding of twinning dislocations on the twin plane along the twinning direction or migration of twin boundaries normal to the twin plane via nucleation and gliding of twinning dislocations on the twin plane. Corresponding to crystallography of hcp structure, twin variants on different twin planes nucleate, propagate, and grow during deformation. These twin variants interact and react each other, forming twin-twin junctions that influence twin propagation and growth during loading, as well as de-twinning and nucleation of secondary twins during unloading. For a comprehensive understanding of twin nucleation, propagation and twin-twin interactions, to characterize structures of twin boundaries and twin-twin junctions at atomic level and their influence on mechanical properties at micro/meso-scales is essential and correspondingly highlighted in this lecture.

1078 Strain rate sensitivity on tensile deformation behavior of GH4199 superalloy under dynamic loading

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The strain rate sensitivity on tensile deformation behaviors of aging treated GH4199 alloy was investigated in the present study. It is found that the yielding strength and tensile strength increase with the increasing of strain rate ($10^{-3}\text{s}^{-1}\sim 10^{-1}\text{s}^{-1}$). Since the deformation time is shorten at higher strain rate, and the dislocations movements are blocked. Thus the flow stress increases at higher strain rate, resulting in higher strength. While the elongation decreases first and then increases with the increasing of strain rate, and it has a minimum value at the strain rate of 10^2s^{-1} . When the strain rate is low ($10^{-3}\text{s}^{-1}\sim 10^{-1}\text{s}^{-1}$), the number of slip system increases with the increasing of strain rate, and the dislocations density increases, so the dislocation movements become harder and have no-enough time to slip with shorter deformation time. That is why the elongation decreases as the strain rate increases to 10^2s^{-1} . But when the strain rate is higher than 10^2s^{-1} , the dislocations slip at the initial stage of plastic deformation, then deforming twinning occurs if the shear stress is higher than the critical shear stress. The deforming twinning could relieve the stress concentration, leading to the increase of ductility of the alloy. While with the increasing of aging time, $\gamma\epsilon$ phase become larger and the strengthening effect increases. But if the $\gamma\epsilon$ phase size reaches a critical value, multi-slipping of the dislocation disappear and the dislocation have to bypass the larger $\gamma\epsilon$ phase, so the strength decreases.

1079 In situ investigation on the deformation-induced phase transformation of metastable austenite in Fe-13%Cr-4%Ni martensitic stainless steel

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Deformation-induced phase transformation (DIPT) of metastable austenite in Fe-13%Cr-4%Ni martensitic stainless steel has been investigated using in situ synchrotron high-energy X-ray diffraction. It is found that the DIPT of metastable austenite occurs during the macroscopic elastic stage and is accompanied by the yielding of metastable austenite at microscopic level. The DIPT rate is accelerated by yielding the martensite matrix, which increases the available nucleation sites for DIPT by enhancing the plastic deformation in austenite. Additionally, transmission electron microscopy observation and crystal plasticity finite element method simulations have been carried out to clarify the relationship between DIPT and the micromechanical behavior of the constituent phases.

1080 Crystal orientation control and magnetostrictive performance of RFe₂-based alloys by high magnetic fields

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Owing to their excellent magnetostriction performance at room temperature, giant magnetostrictive materials based on RFe₂ (R= rare earth) compounds are widely used in the field of advanced technologies. TbFe₂ and Tb_{0.3}Dy_{0.7}Fe_{1.9} are two typical compounds of such materials. The magnetic phase of these materials is RFe₂ which shows a huge magnetostrictive anisotropy and exhibits easy-magnetization direction along $\langle 111 \rangle$. Therefore, to obtain a better magnetostrictive property, extensive efforts have been made to achieve uniform texture along or near the $\langle 111 \rangle$ direction. In this work, the influences of uniform and gradient high magnetic fields on the crystal orientation behavior of RFe₂ phase in Tb_{0.3}Dy_{0.7}Fe_{1.9} alloys and their magnetostrictive property during solidification process were investigated. The dependence of the crystal orientation and magnetic properties of the alloys on the magnetic flux density and the product of the magnetic flux density and its gradient was examined. It was found that the application of the uniform high magnetic field can strongly alter the crystal orientation behavior of RFe₂ phase. The change in crystal orientation of RFe₂ phase is strongly dependent on the magnetic flux density. The application of gradient high magnetic field can produce a gradient magnetostriction distribution in the alloys. The degree of the magnetostriction gradient is strongly dependent on the product of the magnetic flux density and its gradient.

1081 Paracrystalline materials and high-entropy alloys

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There has been a long history for the studies of paracrystalline materials [1,2]. By definition, Paracrystals are the condensed phases in which each lattice site can be indexed by pairs (in 2D) or triplets (in 3D) of integers but the shape and size of the unit cell may be widely varied [2]. Traditionally, scientists accepted the opinion that there should be no possibility for paracrystalline grains as large as crystalline ones in condensed phases because of the more serious lattice deformation as the grain size increasing of the former. However, the discovery of high-entropy alloys (HEA) [3] broke the above conjecture and demonstrated the first example of real 3D paracrystalline bulk [4,5]. HEA formation benefits from the high configurational entropy in multi-principal-element alloy which leads to the relative lower Gibbs free energy to the other potential precipitate phases as the melt solidifies. This results in the unique paracrystalline lattice structure through freezing one of the high-entropy configurations when HEA alloy solidified. In this presentation, we will explain why the bulk of paracrystalline structure can be grown up and present discussions on the close correlation between the unique properties and their paracrystalline configurations in HEA phases.

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1082 The effect of initial micro-structures on deformation behaviors of commercial pure titanium

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In this paper, the effect of initial micro-structures on deformation behaviors of commercial pure titanium was elaborated by investigating the evolution of dislocation boundary and its adiabatic shear sensitivity. At the low or medium strain rates, the main plastic deformation mechanism of as-annealed commercial pure titanium was dislocation slipping. Meanwhile, geometrically necessary boundaries (GNBs) with different directions generated and crossed with each other. However, new dislocation boundaries formed in as-cold rolled plates, which were parallel to the initial ones induced by cold rolling. When the strain rate was up to 1000 /s, the initial dislocation boundary played an adverse role in the adiabatic shear sensitivity of commercial pure titanium. The adiabatic shear band was the high-speed deformation characteristic micro-structure in commercial pure titanium. In addition, dynamic recrystallized grains generated in the center of the adiabatic shear band, whose formation was consistent with the sub-grain rotation mechanism.

1083 In situ study on interface evolution of Al/Cu bimetal by synchrotron X-ray radiography

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Synchrotron X-ray radiography was used to in situ study the diffusion behavior around the interface of Al/Cu bimetal. The dendritic growth around the interface is mainly controlled by the constitutional undercooling induced by the diffusion of Cu in Al-rich side. Four transition zones of solute profile around the interface were identified to be I (α -Al), II (Al+Al₂Cu), III (Al₂Cu) and IV (AlCu, Al₃Cu₄ and Al₂Cu₃), respectively. The concentration variations of Al and Cu around the interface were quantitatively analyzed through the extraction of gray level from sequenced X-ray images. The diffusion coefficients of Cu in Al were calculated from the known concentration variations by an inverse method based on Fick's second law. The diffusion coefficients of Cu in Al were found to follow linear Arrhenius equation dependencies with the pre-exponential factor of $2.83 \times 10^{-5} \text{ m}^2 \text{ s}^{-1}$ and the activation energy of 96.0 kJ mol⁻¹ in a temperature range of 893K to 970K.

1084 Grain boundary plane orientations in recrystallized high purity aluminum and iron

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High purity (99.99 wt.%) aluminum and iron samples were cold rolled with 85% thickness reduction followed by a recrystallization annealing at 330°C and 630°C, respectively. Then the

grain boundary plane orientations were studied by means of five parameter analysis (FPA) which is based on electron backscatter diffraction (EBSD) and stereological method. The results indicate the grain boundary plane orientation in recrystallized microstructure and its evolution with grain growth is quite different between aluminum and iron. In aluminum, low angle grain boundaries (mis-orientation angles ranging from 2° to 15°) constitutes the main part of the entire grain boundaries in the recrystallized microstructure and grain boundary planes orient mostly on $\{2\ 2\ 3\}$ at the stage of primary recrystallization and then it evolves sequentially to $\{1\ 1\ 1\}$ and $\{0\ 0\ 1\}$ with grain growth. Discussions point out the recrystallization of high purity aluminum after cold rolling could be a so-called continuous recrystallization which involves sub-grain coalescence and sub-grain growing, and during grain growth, the crystallographic texture evolves from $\{0\ 1\ 1\}\langle 1\ 1\ 2\rangle$ (Brass) to $\{0\ 1\ 1\}\langle 0\ 0\ 1\rangle$ (Goss) and $\{0\ 2\ 3\}\langle 0\ 0\ 1\rangle$ co-existed, and low angle grain boundary planes usually orient on the exact position of low energy tilt boundaries due to the effects of crystallographic texture and crystallographic plane energy. This is the reason for the regular evolution of grain boundary plane orientations as observed. However, grain boundary planes in recrystallized iron orient mostly on $\{0\ 0\ 1\}$ and it doesn't change during grain growth. This result is attributed to the typical discontinuous recrystallization occurred in iron during annealing after cold rolling.

1085 A practical anodic and cathodic curve intersection model to understand multiple corrosion potentials of Fe-based glassy alloys in OH⁻ contained solutions

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Two anodic peaks (P1 and P2) present in the polarization curve of Fe₇₈Si₉B₁₃ glassy ribbon in NaCl + NaOH solution when scanning rate v or NaOH concentration c_{NaOH} is high enough. The former is ohmic resistance controlled, which is associated with the formation of SiO₂, and the latter is adsorption controlled, accompanying the damage of the SiO₂ film. Three corrosion potentials (E_{c1} , E_{c2} , and E_{c3}) are observed in the polarization curve for Fe-based glassy ribbons in OH⁻ contained solutions when v or c_{NaOH} reaches a specific value.

Multiple corrosion potentials' occurrence is explained by a practical anodic and cathodic curve intersection model. In this model, we choose the measured anodic curve as the apparent anodic curve, from it subtract the measured anodic curves with various v or c_{NaOH} and linearly fit the difference as the imaginary cathodic line. By moving cathodic line evenly or rotating it and imposing it with the apparent anodic curve, the number of intersections can be obtained and the number of corrosion potentials can be predicted. The practical model shows that the distinct P1 is the necessary condition for the occurrence of three corrosion potentials. The formation of P1 or P2 is closely associated with the composition and microstructure of Fe-based alloys.

1086 Microstructural evolution of AZ31 under the application of high density electric current pulses

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Mg alloys are mainly applied in airplane, automobile, and electronic industries ascribed to their superior specific mechanical properties. However, due to their hexagonal close-packed (HCP) structure, Mg alloys exhibit poor ductility at room temperature and obvious strain rate sensitivity. Therefore, many kinds of methods have been used to enhance the materials' mechanical properties by improving its microstructures. In the present work, electric current pulse treatment, as an instantaneous high-energy input method is applied for improving the microstructures of the AZ31B Mg alloy by controlling the function times. Results show that when the current passing the samples with just one time, the misorientation angles between grains have a great deflection and the grain sizes have a little change. For samples treated with current passing two times, the tensile twin begins to form and the low angle grain boundaries become lower. In addition, some refined grains form from the triple grain boundaries. With further more times current passing, the fraction of tensile twin increases and the high angle grain boundaries become more and more. Especially, the refined grains distribute more homogeneously. As a result, with the function times increasing, the content of high angle grain boundaries and tensile twin is enhanced, which is beneficial to improving the materials' mechanical properties.

1087 Refined and Uniform Microstructure with Superior Mechanical Properties in Medium Plate Microalloyed Steel with Reduction in Mn-content during Ultrafast Cooling

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We describe here the relationship between electron microscopy and mechanical property studies in industrially processed titanium bearing microalloyed steel plates that involved processing using the recently developed ultrafast cooling (UFC) approach. Given that the segregation of manganese is generally responsible for microstructural banding in low-alloy steels, which can deteriorate the tensile property in the direction of thickness, the manganese-content was reduced by ~0.6-0.8% with the objective to obtain uniform microstructure across the thickness of the steel plate. Besides, non-uniform distribution of accelerated cooling along the thickness direction also leads to inhomogeneous microstructure across the plate thickness. In order to obtain near-uniform microstructure and similar mechanical properties from the surface to the center of plate, fast and effective cooling process is necessary. In this regard, refined and uniform microstructure that was free of microstructural banding was obtained via UFC process across the plate thickness, with strict control and faster cooling rate on the run-out table. Furthermore, grain refinement and random precipitation in the ferrite matrix contributed ~100 MPa toward yield strength. The study underscores the potential of processing medium and heavy plates of titanium bearing microalloyed steels plates with uniform and refined microstructure across the thickness via thermo-mechanical controlled processing

(TMCP) involving UFC.

1088 Linear friction welding of IN718 to Ti6Al4V

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Linear friction welding, an emerging automated technology, has potential for solid-state joining of dissimilar bi-metallic combinations that enable tailoring of the mechanical performance, whilst limiting the assembly weight for increased fuel efficiency. However, bimetallic assemblies are quite difficult to manufacture, especially when the material combinations can lead to the formation of intermetallic (brittle) phases at the interface, such as the case with assembly of Ti base alloys with Ni base superalloys. The intermetallic phase, once formed, lowers the performance of the as-manufactured properties and, in-service, its growth can lead to unreliable performance. In this project, it was demonstrated that linear friction welding can be applied to join Ti6Al4V (workhorse Ti alloy) to IN718 (workhorse Ni-base superalloy) with minimized interaction at the interface. Of particular merit is that no intermediate layer (between the Ti alloy and Ni-base superalloy) was needed for bonding. Characterization of the bimetallic assemblies included examination of the flash and interface regions, microstructure and hardness.

1089 Changes in the electrical resistivity of amorphous carbon nitride films for potential applications to pressure sensors

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Amorphous carbon nitride ($a\text{-CN}_x$) films are known as useful coating materials. In addition, hydrogen free $a\text{-CN}_x$ films show interesting electrical and optical properties such as low dielectric constant, UV luminescence and photoconductivity. Recently, we reported novel properties of hydrogen free $a\text{-CN}_x$ films, that is, the electrical resistivity changes according to the pressure of measurement atmospheres. This phenomenon suggests that the $a\text{-CN}_x$ films have potential applications to pressure sensors. In this paper, we present the resistivity changes of the $a\text{-CN}_x$ films after exposure to several kinds of gases. The hydrogen free $a\text{-CN}_x$ films were prepared by reactive radio frequency magnetron sputtering using a graphite target and pure nitrogen gas. Nitrogen pressure during deposition was varied from 16 to 107 Pa, which produces columnar film structures with different diameters. The substrate temperature during deposition was fixed at 873 K. The electrical resistivity was measured at 307 K in vacuum and under gas atmospheres such as nitrogen, where the pressure was varied from about 2 to 150 Pa. The resistivity of the $a\text{-CN}_x$ films dropped quickly after gas introduction and it approached almost the constant value when the gas pressure reached the pre-fixed value. After the constant value, the resistivity returned to the original one when the chamber was evacuated. This change in the resistivity according to the change in the measurement pressure was well reproduced. The difference in the resistivity between in vacuum and nitrogen atmosphere was found to increase with increasing the gas pressure during the measurement.

1090 Development of hetero-nano structure in a 316LN austenitic stainless steel during cold-rolling

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The development of the hetero-nano structure in a 316LN stainless steel have been investigated during cold-rolling from 50 to 92% reduction in thickness. Optical microscope observations of the specimen cold-rolled to 50% reduction revealed that the formation of deformation twins in the elongated grains. From complementary transmission electron microscopy observations from the transverse direction (TD) of the rolled specimens, the deformation twins are confirmed as $\{111\}$ - $\langle 211 \rangle$ type. The $\{111\}$ twinning planes inclined at about 25° from rolling direction (RD). Contrary to this, the some portion of grains were filled with the high density of stacking faults. After cold-rolling to 80% reduction, almost all grains were filled with the twins. The twinning planes inclined at about 10° from RD. Shear bands also developed in numerous grains. These bands were inclined at about $20\sim 45^\circ$ from the RD and divided the grains into the rhombic domains of deformation twins. Further rolling to 92% reduction resulted in the development of the hetero-nano structure consisting of elongated ultra-fine grains embedded with the rhombic domains of deformation twins. The twinning plane of the twin domains are nearly parallel to the RD. The average twin thickness is $20\sim 70$ nm. The elongated direction of the ultra-fine grains were nearly parallel to the RD, and the average boundary-spacing along the normal direction (ND) is about 100 nm. Ultra-fine deformation twins with a few nano-meter thickness were frequently observed within the elongated grains. The twinning planes are inclined about 45° from RD or ND direction.

1091 Microstructure of AZCa912 continuous casting bar after hot compression

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Magnesium and magnesium alloys have attracted much attention as the ultra-light structural material because of its excellent light weight and mechanical properties. However, magnesium generally has high reaction ability. Therefore, a problem of magnesium is rapid oxidation of melting metal in the air. On the other hand, noncombustible magnesium alloy including Ca is low reaction ability in the air. Thence, the alloy is compatible with continuous casting method. In our previous study, an AZCa912 continuous casting bar was formed with about $16.9\ \mu\text{m}$ dendrite arm spacing and its hot compression properties were measured. AZCa912 alloy is noncombustible magnesium alloy. Cracks on the side surfaces of the samples were formed by compression at $250\sim 300^\circ\text{C}$, $0.01\sim 1/\text{s}$ and 350°C , $1/\text{s}$. However, inner cracks were not observed and $\sim 5\ \mu\text{m}$ fine-grains were formed. Some samples had an unchanged casting structure after hot pressing, and the percent of the structure remaining unchanged decreased with low-temperature compression. In this study, AZCa912 continuous casting bars with dendrite arm spacing of $15\sim 25\ \mu\text{m}$ were formed and its hot compression properties were measured. AZCa912 bars with 50 mm diameter were formed by a direct chill continuous casting method. Grain sizes in the bars were $120\sim 250\ \mu\text{m}$. As compared with AZCa911 or AZ80 continuous casting bar, many side cracks were easily formed. However, inner cracks were not obtained and fine-grains were

formed. Some samples had unchanged casting structure after hot pressing and amount of the unchanged structure was different with initial microstructure.

1092 Electron beam welding of high strength quenched and tempered steel

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The paper presents the results of metallographic and mechanical properties of electron beam welded joints of high strength quenched and tempered steel grade S690QL. The microstructure of base metal is composed of bainite and martensite mixture at hardness about 290 HV0,5. The microstructure of the weld heat affected zone near the fusion line mainly consists of martensite with a hardness of 420 HV0,5. It should be noted, however, that the microstructure of the steel varies with the distance from the fusion line. The observed changes in the microstructure of the weld are consistent with the transformation of austenite CCT diagram for the tested steel. The mechanical properties of welded joint confirm the standard's requirements. The strength of welded joint is over 850MPa.

1093 Study of growth kinetics of deformation twins in AZ31 magnesium alloy

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A new model for deformation twinning is developed based on dislocation dynamics and the classical pole dislocation mechanism. Twinning nucleation and growth is characterized experimentally by EBSD technique. Deformation twins nucleate at the grain boundary and grow very fast across the grain. We assume that the aspect ratio q ($q = e/l$, where e is the thickness of the twin oblate spheroid and l is the diameter) representing the shape of the twins remains constant in the early stages until thickening of the twins sets in. Our twinning model predicts the thickness of deformation twins in AZ31 magnesium alloy as a function of strain and grain size in agreement with new experimental results. The model is implemented in the thermokinetic simulation software MatCalc and combined with other constitutive microstructural evolution models such as, for instance, the precipitation kinetics framework. This allows for predictions of the deformation twin volume fraction in Mg alloys under various thermo-mechanical treatments.

1094 High toughness and self-lubricative carbon nanotubes-ceramic composites

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Carbon nanotubes (CNT) have some characteristics such as lightness and a very high length/diameter ratio as well as physical and mechanical properties that make them interesting

for ceramic-matrix composites applications, notably when high toughness and self-lubrication are required. Critical issues are the CNT dispersion in the powder and densification of the composite while avoiding or at least limiting CNT damage. The obtained properties greatly depend on the type of CNT used and on their interaction with the matrix. The presence of CNT also induces changes in the sintering kinetics and mechanisms, which can greatly modify the matrix microstructure, which may in turn have a greater effect on the properties than the actual presence of CNT. Key parameters related to the CNT include the number of walls, length and covalent or non-covalent functionalization. The influence of some parameters on the SENB toughness and on the resistance to friction and wear of CNT-oxide composites will be discussed. Results on CNT-MgO [1], CNT-Al₂O₃ [2] and CNT-ZrO₂ [3] composites will be presented. The composite powders are prepared either by the in situ growth of the CNTs within the powder or by mixing routes. Densification is achieved by spark plasma sintering.

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1095 Numerical simulation of the temperature field evolution, phase transformation and mechanical deformation during flash butt welding of railway rails

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Rail manufacturers still apply empirical methods when optimizing results for Flash Butt Welding (FBW) railway rails. Generally, simulation can be a very efficient tool to support welding process optimization. For this purpose a new finite element (FE) model of the FBW process of rails – as commonly applied in rail manufacturing industry – is developed. It is implemented in a strong coupled thermo-electrokinetical and metallurgical calculation routine, using the software SYSWELD. Phase transformations are taken into consideration by a metallurgical model and an implemented welding CCT diagram. Material expulsion and deformation during upsetting are incorporated by a sequentially weak coupled mechanical FE-calculation. The focus of this work is to introduce a physically based model of the heat source specifically inherent to the FBW process. Varying contact conditions at the welding surfaces throughout the single process steps influence the electric transition resistance between the welded rails. It represents the main heat source to the FBW process, and hence, predominantly determines the temperature field evolution. Therefore, this model incorporates adapted electric transition resistance functions as main boundary conditions at the welding gap. Based on multiple measurements the simulation is optimized. As a result a more accurate illustration of the temperature field evolution when compared to literature sources is obtained. Based on the metallurgical calculation also a more accurate prediction of the microstructure evolution can be derived. Hence, this model can help to optimize the FBW process with respect to the required properties of the rail joint.

1096 Effect of different bainite morphologies on the formability of advanced high strength steels

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The further improvement of advanced high strength steel grades for the automotive industry is driven by the demand for high strength as well as for good formability. Steels with mainly bainitic matrix show good performance concerning these requirements. However, when forming these type of materials, it comes to microstructural changes which can strongly influence the performance in service. Therefore, indicators for microstructural damage have to be identified clearly. Within this study, the influence of different bainite morphologies on the formability was evaluated by three-point-bending tests and so-called hole expansion tests. The microstructural characteristics of the initial condition as well as the tested samples were investigated by light optical microscopy and scanning electron microscopy. Extensive electron backscatter diffraction investigations were conducted in order to show the texture development caused by deformation as well as to analyse the local strains in and adjacent to different microstructural constituents. The results allow a conclusion about the deformation mechanisms and damage initiators.

1097 Phase-field modeling of metal oxidation at elevated temperatures

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The oxidation kinetics of metals/alloys at elevated temperatures is essential to the corrosion-life prediction of the structural materials in advanced energy systems. The growth of oxide film on a metal surface typically involves coupling of chemical reaction and diffusion of charged point defects. A phase-field model is developed to address the essential physics during metal oxidation over a continuous range of time and film thickness. The model is able to reproduce the classic Deal-Grove model with respect to the linear-parabolic transition. In particular, the model reproduces Wagner's theory in the thick film limit and predicts a deviation from the Wagner-parabolic growth when the oxide film thickness is on the order of Debye length. The essential characteristics of electric field developed in an oxide film during high temperature metal oxidation will also be discussed.

1098 Applications of electric discharge assisted mechanical milling to the synthesis of high temperature and high hardness materials

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Electric discharge assisted mechanical milling (EDAMM) involves application of high frequency pulsed electrical discharges resulting in a localised dusty plasma environment during

mechanical milling. The electrical impulses are generated with voltages in the kV range and currents in the mA range and milling is typically performed in a hemispherical rod mill. The technique promotes high reactivity between the starting powder(s) and the surrounding ionized gas and has been shown to significantly enhance solid-solid and solid-gas reactivity resulting in both rapid reactions and, in some cases, new reaction routes. EDAMM is particularly suitable for exploration of routes for the rapid synthesis of high temperature and high hardness phases. Here we describe application of the technique to the synthesis of refractory metal carbides, nitrides, carbonitrides, and borides, where EDAMM processing parameters can be altered to control final product chemistry. We also present some preliminary results on application of EDAMM for the exploration new refractory metal high enthalpy alloys.

1099 Effects of microalloy additions and thermomechanical processing on austenite grain size control in medium carbon steel bar rolling

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Thermomechanical processing (TMP) of microalloyed steels has been widely implemented for austenite grain size control. Three AISI 1045 steels: a base steel, one modified with V, and one modified with V and Nb were examined to optimize microstructural conditioning prior to induction hardening. The effects of V and Nb, both in solution and present as precipitates, were considered for their influence on austenite evolution and transformation during bar rolling processes. Simulated bar rolling histories were evaluated using fixed-end hot torsion tests with a Gleeble® 3500. The effects of chemical composition and thermomechanical treatment on final microstructures were examined through analysis of industrially rolled bars as well as interrupted laboratory simulations of steel bar rolling processes in order to provide additional insights into the morphological evolution of austenite during bar rolling of microalloyed steels. Analysis of final grain size, constituent fraction, and precipitate distribution are complemented with analysis of austenite recrystallization during rolling, prior austenite grain sizes, inclination angles of individual austenite grains within hot torsion specimens, and parent austenite aspect ratios. The potential for utilizing TMP, in conjunction with microalloy additions, to enhance bar steel microstructures and subsequent performance is assessed by evaluating the induction hardening response of each steel systematically processed with different preconditioning treatments, and is also presented.

1100 Diffusion based modelling of isothermal solidification during brazing of aluminium alloys

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The furnace brazing of aluminum automotive heat exchangers utilizes Al brazing sheets that consist of a lower melting, Si containing 4XXX clad aluminum alloy or filler metal, and a low Si containing 3XXX core alloy. At the brazing temperature, diffusion of Si from the clad filler

into the core alloy results in isothermal solidification (IS) and a rapid reduction in the liquid fraction available to form a joint. Therefore the extent and rate of IS impacts the size of the joint fillet region and overall brazeability. In this work a diffusion based model was developed to predict the rate of IS in composite braze sheets. The model is able to take into account the influence of sheet thickness on IS and, using diffusivity values previously published by other researchers, correctly predicts the rate of IS experimentally measured in Al braze sheets using differential scanning calorimetry.

1101 Dissimilar electron beam welds of nickel base alloy A625 with a 9% Cr-steel for high temperature applications

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Welding of thick walled components with an electron beam has great potential due to the minimal heat input, high reproducibility and cost-efficiency. In the present work electron beam welding was used to join 50 mm thick plates of cast Ni-base alloy A625 to ferritic/martensitic 9% Cr steel (CB2) plates without using a filler metal. The joints were creep exposed at 625°C with stress levels ranging between 150 - 100 MPa. Microstructure analysis of the joints and the heat affected zone was carried out using hardness mapping, metallography and scanning electron microscopy with EBSD to determine the location and mechanism of the creep rupture. Creep fracture is located in the fine grain heat affected zone of the 9% Cr steel. The performance and related microstructure properties of the electron beam welded specimen are more than competitive to conventional metal-arc-welding procedures.

1102 The energy balance of GMAW processes and it's quantification

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The GMAW process has been applied for many decades to join metals, especially different steel grades and various aluminium alloys. As demands such as light weight construction, the joining of dissimilar materials and the increase of the performance of the welding process get more and more relevant, there is a need to develop modern welding processes that are able to provide an optimised amount of energy at the fusion zone. Besides the process-specific quantity of electric energy that is transformed to thermal energy, it is important to understand how much of this energy is transferred to the wire electrode by Joule heating and the amount of energy that is generated at the anode spot. In addition it is necessary to know the portion of the energy that is directly transferred to the work piece by the electric arc.

The optimal energy split that is needed, depends on the instantaneous boundary conditions such as the root gap, the surface condition of the base material, the weld geometry, the linear or angular alignment of the work pieces, etc. To provide a basis on which modern GMAW processes can be developed, the energy balance of different GMAW processes is quantified by the use of calorimetry, by providing analytical solutions and by creating an FEM Model.

The energy balance of the analysed GMAW processes are compared and development potentialities are carried out referring to the specifics of modern metallic construction materials.

1103 A Microbattery Made from Monocrystalline Silicon

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Silicon is known as a negative electrode material for Li-based batteries guaranteeing an outstanding high storage capacity. In many cases nanostructured Si in various forms is used to buffer the extreme changes in volume upon lithiation. Therefore, monocrystalline Si, as it is omnipresent in semiconductor industry, is anticipated to be excluded from any usage in modern energy storage systems. We will show, however, that structured monocrystalline wafer grade Si is excellently suited to be used in Si-based microbatteries that can directly implemented on the chip. Besides electrochemical characterization we used time-domain and high-resolution Li(7) and Li(6) nuclear magnetic resonance (NMR) spectroscopy to study local structures and the underlying lithium-ion dynamics, which crucially determine overall performance of the device [1]. The rechargeable microbattery developed takes advantage of a remarkable cycling stability and excellent charge capacities. It allows for unexpectedly high peak currents considerably widen the field of applications including mobile devices requiring an on-board power supply such as wireless sensors. [1] A. Dunst, M. Sternad, V. Epp, and M. Wilkening, J. Phys. Chem. C 119 (2015) 12183

1104 Influence of NbC-precipitation on hot ductility in microalloyed steel

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One of the main challenges during continuous casting of microalloyed steel is to avoid the formation of transverse surface cracks on the steel slabs. These cracks occur due to severe mechanical and thermal stresses in the slab during the straightening operation. The reason for this phenomenon is a ductility loss of austenite in a typical temperature range of 700°C - 1100°C. One of the main mechanisms reducing the ductility is the precipitation of carbides and nitrides. In this work, we correlate the ductility and precipitation state after two model cooling strategies in a microalloyed steel grade. Continuously cooled samples show a minimum of ductility at temperatures around 750°C. With increasing temperatures, deformability recovers again to reach full ductility again at 950°C. In contrast, samples treated with a fast intermediate cooling and reheating show constant low ductility in this entire temperature range. A transmission electron microscopy (TEM) investigation shows nanometer-sized NbC precipitates in the low ductility states. In contrast, in the samples with high ductility, large NbC precipitates with sizes of over 100 nm and significantly lower number density are observed. The experimental results show a good accordance with corresponding precipitation kinetics simulations carried out with the MatCalc software package.

1105 Multiscale micromechanical modelling for advanced high strength steels including both the TRIP and TWIP effect

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Advanced high strength steels, such as high manganese steels, show an extraordinary combination of strength and ductility. While this provides tremendous potential for many applications, these materials are not yet widely used in industry due to a number reasons. As simulation is nowadays an integral part of component layout e.g. in automotive industry, without suitable material models new materials will not be introduced into the production process. We present a crystal plasticity formulation that includes both twinning and transformation as additional deformation mechanisms besides the conventional dislocation slip. The twinning model is a crystal plasticity adaption of an analytical model by Steinmetz et al. (2013) while the TRIP model is developed to model the γ (fcc) \rightarrow ϵ (hcp) \rightarrow α' (bcc) transformation in austenitic steels. This physics-based strain rate- and temperature sensitive model reflects the formation of dislocation structures, twinning and martensitic phase transformations using physically-based model parameters and ab initio derived thermodynamic quantities. Experimental data for a Fe-22Mn-0.6C steel, which exhibits both the TWIP effect and $\gamma \rightarrow \epsilon$ transformation as a function of stacking fault energy and loading conditions, is used to validate the model. The combined model allows to systematically study under which conditions either one or both mechanisms together can be triggered during deformation. D. R. Steinmetz, T. Jäpel, B. Wietbrock, P. Eisenlohr, I. Gutierrez-Urrutia, A. Saeed-Akbari, T. Hickel, F. Roters, D. Raabe (2013): Revealing the strain-hardening behavior of twinning-induced plasticity steels: Theory, simulations, experiments, *Acta Materialia* 61: 494 – 510

1106 Effects of magnetic field intensity on carbon diffusion coefficient in pure iron in the paramagnetic ferrite region

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In the present study, effects of magnetic field intensity on carbon diffusion coefficient in pure iron in the direction parallel to the magnetic field in paramagnetic ferrite region above the Curie temperature were investigated. Specimens of high purity iron (99.99%) were buried in an air-proof melting pot filled up with cementation agent, and respectively subjected to isothermal annealing at 1123K for 60 min. A magnetic field with different intensity (0T, 0.1T, 0.7 T, 1 T, 6 T, 12 T) was applied during the whole heating, isothermal holding and cooling processes. The carbon penetration profiles from the iron surface to interior were measured by Field Emission Electron Probe Microanalyzer. The carbon diffusivities in iron under the magnetic field were measured from the carbon penetration profiles, the carbon diffusion coefficient in pure iron carburized with different magnetic field intensity was calculated according to the Fick's second law. It was found that the carbon diffusion coefficient increased gradually with the enhancement of magnetic field intensity. The above results indicated that magnetic field annealing obviously promoted the carbon diffusion in pure iron in the direction parallel to the magnetic field in

paramagnetic ferrite region above the Curie temperature, and this effect increased with further enhancement of the magnetic field intensity. The structure defects made by the magnetic field may cause the increasing of carbon diffusion flux.

1107 Microstructure evolution of AA 6061-T6 weld joints in ultrasonic vibration enhanced friction stir welding

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Ultrasonic vibration enhanced friction stir welding is a new variant of friction stir welding developed recently to improve the welding process and joint properties. In this study, the effect and role of ultrasonic vibration on Al 6061-T6 alloy during its friction stir welding was investigated by comparing local microstructure evolution and microtexture formation in its weld nugget with those of conventional friction stir welded Al 6061-T6 alloy. Results showed that ultrasonic enhanced joints have higher deformation characteristics in the nugget zone due to ultrasonic softening effect prior to welding. Electron back scattered diffraction studies showed that ultrasonic softening complements recrystallization process and results in finer grains along weld center axis while its influence is weakened at the weld nugget extremities and thus may contribute only towards grain orientation. Ultrasonic enhancement did not induce significant change to the local microtexture; rather the feature observed is a small angular shift in the shear plane which tilted the texture by an equivalent angle.

1108 Deformation mechanisms of a Fe-20Mn-3Al-3Si steel with different deformation processes

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The Fe-20Mn-3Al-3Si TRIP/TWIP steels have drawn significant attention due to their high strength and better plasticity. The microstructures and deformation mechanisms of a Fe-20Mn-3Al-3Si steel subjected to different deformation processes were studied using transmission electron microscopy. It is found that this steel primarily exhibits transformation-induced plasticity (TRIP) during cold-rolled or tensile deformation at room temperature. When the rate of strain is increasing, the steel may undergo a transition from TRIP to twinning-induced plasticity (TWIP) at room temperature. Gradient nanotwinned layers with high hardness and good thermal stability can be produced in this steel by means of a high strain-rate surface mechanical grinding treatment. The deformation process affects dislocation slipping, deformation twinning and martensite transformation. A deformation twin is usually very thin but paralleled deformation twins like to assemble twinned bundles. The deformation twins contain high density dislocations. The dislocation configuration and the interaction of dislocations with twin boundaries have influences on the recovery and recrystallization of the deformation twins.

1109 Metallic glass composite with good tensile ductility, high strength and large elastic strain limit

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Bulk metallic glasses exhibit high strength and large elastic strain limit but have no tensile ductility. However, bulk metallic glass composites reinforced by in-situ dendrites possess significantly improved toughness but at the expense of high strength and large elastic strain limit. Here, we report a bulk metallic glass composite with strong strain-hardening capability and large elastic strain limit. It was found that, by plastic predeformation, the bulk metallic glass composite can exhibit both a large elastic strain limit and high strength under tension. These unique elastic mechanical properties are attributed to the reversible B2-B19' phase transformation and the plastic-predeformation-induced complicated stress state in the metallic glass matrix and the second phase. These findings are significant for the design and application of bulk metallic glass composites with excellent mechanical properties.

1110 Mechanical properties of ARB processed Mg alloys with heterogeneous laminate structures

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Mg alloys have various advantages. However, the low formability due to the poor ductility of Mg alloys limits their engineering applications. In this study, Mg-3%Gd alloy and Mg AZ31 alloy were chosen to explore processing approaches for improving its strength and ductility. The alloy was processed by accumulative roll-bonding (ARB) at a high temperature to 2 cycles followed by annealing at various temperatures. The microstructures after annealing were characterized by the EBSD and TEM, and the mechanical properties were measured by a tensile test. Synchrotron X-Ray was also applied to analyse the lattice strain during in-situ tensile test. It was found that good combinations of strength and ductility can be obtained by proper annealing of the ARB processed alloys. The alloys have strengths as high as the ultra-fine sized alloys and elongations as large as the full annealed coarse grain counterparts. The good mechanical properties were related to the heterogeneous laminate structures composing of fine-sized recrystallized structure and ultra-fine-sized deformed structure.

1111 Multiscale modeling of solidification

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A major scientific challenge in developing a solidification model lies in the requirement to bridge different length scales of physical phenomena, and to deal with the multiphase transport phenomena. No one single model is able to span the entire range of length scales from the process level of industrial interest down to the atomic scale due to the limitation of the current

computational resources. Therefore, a trend for the process-modelling of solidification is to incorporate the interfacial phenomena occurring at the lower scale into the process scale of solidification with a so-called volume average approach. A casting of industrial interest can be discretized into large number of volume elements. The macroscopic transport phenomena (mass, momentum, enthalpy, species, etc.) are directly solved. All the microscopic phenomena occurring at the lower scale, e.g. the mass transfer and solute partitioning at the liquid-solid interface, will be volume-averaged in each volume element. Solidification thermodynamics, kinetics and multiphase hydrodynamics will be coupled. This presentation is to give an overview of the state-of-the-art of this field, and some modeling examples are demonstrated.

1112 Modification effects of Sb on Al7SiMg alloy measured with cooling curve analysis

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The modification mechanism of Sb on eutectic Si phase of Al-Si alloys is not understood very well. In this paper the modification effects of Sb on Al7SiMg alloy were investigated by using computer aided cooling curve thermal analysis coupled with microstructure observation. The results show that the addition of Sb with 0.1%~0.25% amount has refining effect on eutectic Si phase, and this modification effect can be recorded by the cooling curves. The addition of Sb decreases the growth temperature of the eutectic solidification by 5.0~6.9 degree, and this indicates that there is an undercooling effect with Sb modification. Moreover, faster cooling rate could obtain more superior morphology of eutectic Si, which could be changed from short rod to nearly granular particles. The morphology of eutectic Si is nearly unchanged with holding time extending from 0.5h to 2.5h, which means Sb is a long-term modifier for Al-Si alloys.

1113 Numerical simulation of pore evolution of 7050 aluminum alloy during hot compression process

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Evolution behavior of pores in 7050 aluminum alloy during hot compression process has been investigated by finite element (FE) numerical simulation. The representative volume element (RVE) model containing one isolated pore is built, in which the gas in pore is treated as ideal gas. Effects of initial pore inner pressure and deformation temperature on pore evolution have been investigated. The simulation results indicate that stress concentration exists around the pore in the compressing process. At the simple compression condition, the inner pressure of the pore increases but the volume decreases as the bulk metals deforms. However, the volume reaches a plateau after the yield point of bulk metal. The plateau volume depends on the initial inner pressure of the pore and the flow stress of the bulk metal. Since the inner pressure of the pore balances with the flow stress of bulk metal at the interface, the temperature affects the evolution behavior of the pore through its influence on the flow stress of the bulk metal primarily.

1114 On the correlation between local strain heterogeneity and plasticity of metallic glasses

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Due to its long range disordering atomic structure, metallic glasses (MGs) have different deformation mechanism from crystalline materials. They deform by localized shearing instead of dislocation, which increase the difficulty of building structure-properties relationship in MGs, and a clear structure-properties relationship was still lack in MGs. This talk attempts to search the structural origin of plasticity of MGs from the localized and statistical information. It was found global plastic deformation connected with the fluctuation and evolution of local strain fields. The current work may help for understanding structure-property relationship in metallic glasses.

1115 Processing and thermoelectric properties of new Si-/Se-/ Sn-based intermetallics

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Global warming is the driving force for developing new functional thermoelectric generators based on new materials which contain at least one of the elements Si, Se, Sb or Sn. We describe four processing methods and their characterization by SEM and thermoelectric properties. The earth-abundant Mg_2Si requires the method of cyclic hydrogen loading, which has two advantages: It suppresses the oxide formation, and promotes the driving force for formation of the intermetallic phase. With subsequent SPS-sintering bulk material can be produced, which shows large electric conductivity and Seebeck coefficient. While most intermetallic alloys can economically be produced by arc-melting, Selen has a low melting point and its intermetallic phases can be processed by hot pressing or capsule annealing, when residual Oxygen is removed. Equally axial channeling pressing (EACP) is increasing in popularity as it is one of the appropriate processing methods and has the advantage of applying shear energy for self-driven formation reaction. Arc melting is the conventional method for producing most metallic alloys, including Sn- and BaCuSi-alloys and show large Seebeck coefficient, but deviations from stoichiometry influence the sensitive thermoelectric properties. For up-scaling recently new innovative processing methods have been reported, such as ball-milling, the jelly-roll method, or powder-in-tube technologies, which require subsequent diffusion annealing. This innovative field of metallurgy will gain interest in near future because there are many new possibilities for new functional metals.

1116 Defects and charging processes in Li-ion battery cathodes studied by in-operando magnetometry and positron annihilation

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Research in the field of modern battery materials demands characterization techniques which may yield insight into the atomistic processes of battery charging and discharging. Using LiCoO_2 as a model system, the potentials of SQUID magnetometry and positron annihilation spectroscopy for studying battery material will be outlined. We could recently implement an in-situ electrochemical cell in a SQUID-magnetometer [1] which, for the first time, allowed in-operando continuous monitoring of the magnetic susceptibility of LiCoO_2 during consecutive charging and discharging cycles. The distinct variation of the magnetic behaviour with Li content turns out as powerful diagnostic tool, since the magnetic material parameters are highly sensitive to phase composition, structural disorder, defects, and the oxidation state of the transition metals ions. Most recently, these studies could also be applied for more complex cathode materials, such as $\text{LiCo}_{1/3}\text{Ni}_{1/3}\text{Mn}_{1/3}\text{O}_2$. Positron annihilation ideally complements SQUID magnetometry, since it enables the specific detection of vacancy defects which are formed upon battery charging. Studies on commercial polycrystalline Li_xCoO_2 revealed vacancy-type defects on the Li-sublattice the size of which increases with Li-extraction [2]. Indication for Li-reordering at the reversibility limit of extraction is found which nicely correlates with characteristic changes of the magnetic parameters in this concentration regime. Currently these studies are being extended on Li_xCoO_2 thin-films of solid state batteries making use of the slow-positron beam of the Heinz Mayer-Leibnitz neutron source. [1] St. Topolevec et al, Rev. Sci. Instr. 86 (2015) 063903 [2] P. Parz et al., Appl. Phys. Letters 102 (2013) 151901

1117 Design and fabrication low-cost Al/C composites with high thermal conductivity

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In the microelectronics industry, the ever-increasing power density and continuing miniaturization of semiconductor and optoelectronic devices lead to mounting thermal management challenges. In order to realize high performance, long life and high reliability, it is imperative to develop very efficient electronic packaging materials with high thermal conductivity (TC) and low coefficient of thermal expansion (CTE). Towards this goal, different composite systems have been investigated. It is generally believed by compositing filler of high TC and matrix with high TC, high TC in composites can be achieved. However, in reality the TCs of these composites are not as high as expected, and interfacial thermal resistance was considered be predominantly responsible for this, since undesirable excessive formation of interface layer can hinder effective heat transfer due to low TC of interface layer itself (eg. the Al_4C_3 in Al/C system). In this study, a low-cost Al/C system was investigated to study the effects of interface, geometry and topology structures of carbon fillers on the TC of composites,

from both theoretical calculation and experiment study. The results show that the interface structure, geometry and topology structures of composites can be the key factors to determine the TC. Especially, a diffused interface structure is beneficial for the enhancement of the TC, and the effect of the structure factors were found to be as important as interfacial thermal resistance on the TC. Based on these results, low-cost Al/C composites have been designed and fabricated with TC as high as 630 W/m K.

1118 Design and fabrication low-cost Al/C composites with high thermal conductivity

Cunjuan Xia*, Zhe Chen, Shengyi Zhong
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In the microelectronics industry, the ever-increasing power density and continuing miniaturization of semiconductor and optoelectronic devices lead to mounting thermal management challenges. In order to realize high performance, long life and high reliability, it is imperative to develop very efficient electronic packaging materials with high thermal conductivity (TC) and low coefficient of thermal expansion (CTE). Towards this goal, different composite systems have been investigated. It is generally believed by compositing filler of high TC and matrix with high TC, high TC in composites can be achieved. However, in reality the TCs of these composites are not as high as expected, and interfacial thermal resistance was considered be predominantly responsible for this, since undesirable excessive formation of interface layer can hinder effective heat transfer due to low TC of interface layer itself (eg. the Al_4C_3 in Al/C system). In this study, a low-cost Al/C system was investigated to study the effects of interface, geometry and topology structures of carbon fillers on the TC of composites, from both theoretical calculation and experiment study. The results show that the interface structure, geometry and topology structures of composites can be the key factors to determine the TC. Especially, a diffused interface structure is beneficial for the enhancement of the TC, and the effect of the structure factors were found to be as important as interfacial thermal resistance on the TC. Based on these results, low-cost Al/C composites have been designed and fabricated with TC as high as 630 W/m K.

1119 Quantitative study of the microstructural evolution of FSPed TiB_2 nano-particles reinforced Al composites by EBSD, synchrotron and neutron analysis

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The microstructural evolution of friction stir processed TiB_2 nano-particles reinforced Al matrix composites was investigated by a combination of various techniques, including neutron and synchrotron X-ray diffraction, scanning electron microscopy and electron backscatter diffraction (EBSD). A detailed picture of microstructure evolution during this high temperature deformation was built up. The development of microstructure has been shown to be a ultrafine grain structure with homogeneous particle distribution, which results from a complex process

of particles/matrix interaction during the deformation and dynamic recrystallization. By comparing with its particles-free alloy counterpart, the thermomechanical response of composite materials at high temperature is discussed in details in term of aluminum's deformation and recrystallization mechanisms combined with nanosized particle effects.

1120 Quantitative study of the microstructural evolution of FSPed TiB₂ nano-particles reinforced Al composites by EBSD, synchrotron and neutron analysis

Cunjuan Xia*, Zhe Chen, Shengyi Zhong
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1121 Titania nanotubes grown at the inner surface of extremely long and slim titanium tubes

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Titania nanotubes have been widely studied for their intriguing performances in functional devices. It is well-established to grow nanotubes on planar titanium electrodes for controllable morphology, but less so for the study on titanium tubular counterparts. In this study, growth of titania nanotubes at the inner surface of extremely long and slim titanium tubular electrodes (tube length of 1000 mm and inner diameter of 10 mm) was investigated. It was found that uniform arrays of titania nanotubes are formed at inner surface of the electrodes via elaborately designing the experimental setups. The nanotubes exhibit a length limitation of 1 μ m and pore diameter of about 45 nm.

1122 A nanotwinned surface layer generated by high strain-rate deformation in a TRIP steel

Pan Xie*, Cuilan Wu, Yan Chen, Jianghua Chen, Xiubo Yang, Shiyun Duan, Ning Yan, Xueao Zhang, Jingyue Fang
Hunan university, Republic of China

Gradient nanotwinned layers (GNTLs) with high hardness of approximately 500 HV and good thermal stability were produced in a Fe–20Mn–3Al–3Si TRIP steel by means of a high strain-rate surface mechanical grinding treatment (SMGT). The effect of the strain-rate on the plasticity-enhancing mechanisms in steels was investigated. It is found that although it primarily exhibits transformation-induced plasticity (TRIP) when cold-rolled, this steel shows twinning-induced plasticity (TWIP) during the SMGT process, illustrating that the steel may undergo a transition from TRIP to TWIP under high-strain-rate deformation. The martensite induced by deformation is thermally unstable and can easily transform back to austenite during annealing. In contrast, the deformation twins (DTs) are thermally much stable, since de-twinning, which plays a key role in the recovery of DTs, is more difficult to occur during thermal annealing. As such, undergoing the same annealing at 600 °C for 1 h, the GNTLs containing a great many of DTs maintain high hardness, whereas the cold-rolled counterpart samples containing deformation-induced martensite softens drastically.

1123 Simultaneous strengthening and toughening of Mg alloys by {10-12} twins

Yunchang Xin*, Hong Zhang, Xiaojun Zhou, Qing Liu
Chongqing University, Materials Science and Engineering, People's Republic of China

{10-12} twinning constitutes one of the main deformation modes of Mg alloys at room temperature and is frequently used to tailor mechanical performance of Mg alloys. The present presentation involves the applications of {10-12} twins to simultaneously harden and toughen Mg alloys. The twins are used to improve mechanical properties of both a single phased Mg alloy and a precipitated one. The influence of twins on microstructure, texture and mechanical properties are systematically addressed. We also focus on how to prepare effective twin structure to acquire better mechanical properties. The hardening and toughening mechanisms of twins are discussed.

1124 Investigation on effect of centrifugal counter-gravity casting to solidification microstructure and mechanical properties of aluminum alloy

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To investigate the influence of Counter-Gravity Casting (C3) process on the solidification microstructure and mechanical properties of casting, aluminum alloy samples were produced by different process conditions of C3. The results show that C3 has better feeding capacity

compared with the vacuum suction casting, and the mechanical vibration and the convection of melts formed at centrifugal rotation stage suppress the dendrites growth, subsequently resulting in the refinement of grains and the improvement of mechanical properties, density and hardness. A finer grain and higher strength can be obtained in the A357 alloy with increasing centrifugal radius and rotational speed.

1125 Bioinspired Graphene-Copper matrix nanocomposites

Ding-Bang Xiong*, Mu Cao, Qiang Guo, Zhanqiu Tan, Genlian Fan, Zhiqiang Li, Di Zhang
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Metals can be strengthened by adding hard reinforcements, but such strategy usually compromises ductility and toughness. Natural nacre consists of hard and soft phases organized in a regular 'brick-and-mortar' structure and exhibits a superior combination of mechanical strength and toughness,[1] which is an attractive model for strengthening and toughening artificial composites, but such bioinspired metal matrix composite has yet to be made. Here we prepared nacre-like graphene reinforced Cu matrix composite based on a preform impregnation process, by which graphene was used as "brick" and inserted into ordered porous Cu preform, followed by compacting (namely "□-and-mortar").[2] This process realized uniform dispersion and alignment of graphene in Cu matrix simultaneously. The graphene-and-Cu artificial nacles exhibited simultaneous enhancement on yield strength and ductility as well as increased modulus, attributed to graphene strengthening, effective crack deflection and a possible combined failure mode of graphene. The artificial nacles also showed significantly higher strengthening efficiency than other conventional Cu matrix composites, which might be related to the alignment of graphene. References: [1] Wegst, U.G.K.; Bai, H.; Saiz, E.; Tomisa, A.P.; Ritchie, R.O. *Nature Mater.* 14 (2015) 23-36. [2] Xiong, D.B.; Cao, M.; Guo, Q.; Tan, Z.Q.; Fan, G.L.; Li, Z.Q.; Zhang, D. *ACS Nano*, 9 (2015) 6934–6943.

1126 Effect of continuous annealing temperature on microstructure and mechanical properties of a high strength cold-rolled DP980 steel

Kai Zhou, Ying Zou, YunBo Xu*, ZhiPing Hu, XiaoLong Yang, XiaoDong Tan, YongMei Yu, Hua Zhan
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Continuous annealing processes were applied to a 980MPa cold-rolled dual phase steel (Fe-0.11C-2.5Mn-0.5Si-0.4Cr) and the effect of continuous annealing temperature on microstructure and mechanical properties was investigated. The microstructures were observed and analyzed by optical microscopy (OM), scanning electron microscopy (SEM), electron probe micro-analyzer (EPMA) and transmission electron microscopy (TEM). The mechanical properties were measured by uniaxial tensile tests. The results revealed that the steel composed of a certain percentage of ferrite, martensite and a small amount of retained austenite. As the annealing temperature increased, the volume fraction of martensite reached to 43% from 25% and the morphology translated to lath-like from M/A island. As a consequence, the ultimate tensile strength (UTS) and yield strength had a moderate increase from 1070 to 1130 MPa and

580 to 650 MPa, respectively. Meanwhile, the fracture elongation rose to the maximum 12.6% firstly and then decreased to 9.0%. The optimizing mechanical properties with UTS up to 1090 MPa, yield ratio about 0.54 and fracture elongation about 13% could be obtained at the annealing temperature of 790°C for 120s.

1127 Characterization of electrodeposited manganese oxide layer for advanced capacitor electrode

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People's Republic of China

Manganese oxide has been considered a promising electrode material for supercapacitor due to its low cost, environmentally friendly and high specific capacitance. Many efforts have been made to improve the performance of manganese oxide for higher capacity and larger energy density. In the present study, we prepared MnO₂ layers with different microstructures by direct electrodeposition and investigated their performance and microstructures. The nanosheet of MnO₂ nanofiber bundles exhibited highest capacitance while the nanosheet of spherical MnO₂ particles exhibited most stable performance. A composite layer of MnO₂ and graphene was also obtained using direct electrodeposition. The effect of the addition of graphene on the performance of the MnO₂ electrode was evaluated.

1128 Fatigue endurance limit and crack growth behavior of a high-toughness Zr₆₁Ti₂Cu₂₅Al₁₂ bulk metallic glass

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We report the fatigue damage behavior of a Zr₆₁Ti₂Cu₂₅Al₁₂ (ZT1) bulk metallic glass (BMG), which was known to have a record-high fracture toughness. The ZT1 exhibits the highest fatigue endurance limit among all BMGs, with a normalized fatigue limit of $\sigma_a=440$ MPa (in four-point bending at a loading ratio of 0.1), or ~0.27 of its tensile strength. The crack-growth resistance in the stress/life tests arises from crack-tip plastic shielding due to prolific shear-banding, as well as deflected crack path following a “zigzag” pattern. The relation between the range of stress intensity factor (ΔK) and the crack-growth rate (crack extension per cycle, da/dN) was also determined. ZT1 shows a fatigue threshold, ΔK_{th} , of 2.8 MPa \sqrt{m} , which appears to be related to the onset of shear band nucleation. Three distinct regimes of fatigue crack growth were observed, with different slopes in the da/dN - ΔK curve, attributable to different micromechanisms. In the Paris regime, each fatigue striation on fracture surface is generated by a number of loading cycles, rather than by a single loading cycle. In general, the ΔK_{th} of a BMG approximately scales with its fracture toughness. At different levels of ΔK , shear bands appear to play different roles, either facilitating or blunting the crack growth.

1129 Weibull analysis of fracture strength for Zr₅₅Ti₂Co₂₈Al₁₅ bulk metallic glass: Tension-compression asymmetry and porosity effect

Jian Xu*, Hui-Li Gao, Yong Shen

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In this work, two- and three-parameter Weibull statistics were used for analyzing the variability of fracture strength for Zr₅₅Ti₂Co₂₈Al₁₅ bulk metallic glass (BMG), both in compression and tension testing. In contrast to the compression in which the specimens fail via the massive shear-off, however, failure mode in tension for the as-cast BMG is flaw-controlled crack opening (mode I or mixed mode) due to the presence of cast defects such as porosity. As a result, dispersion of compressive fracture strength is rather uniform. For the BMG rods of 6 mm in diameter, the three-parameter Weibull modulus m_{3p} and threshold stress (below which no failure occurs) is 3.4 and 1780 MPa, respectively. However, tensile fracture strength of the BMGs manifests a large variability, in a range of 310-1690 MPa. In terms of fracture surface morphology, the specimen failure at different stress is associated with two types of defects: large pores on/near the surface of specimens and small internal pores. Using bimodal and three-parameter Weibull analysis, the Weibull modulus m_1 and threshold at lower strength level is 1.8 and 250 MPa, respectively, suggesting a modest reliability. One should exercise caution, therefore, in interpreting the reliability of as-cast BMG materials only simply in terms of the compression tests, small-sized samples and two-parameter Weibull analysis. Like the conventional metal castings, controlling the processing conditions to minimize the cast defects is critical issue to ensure the reliability of BMG materials.

1130 Potential application in micro-forming technology with ultrafine-grained materials

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Grain size is the dominant factor determining the limiting size of the geometrical features that may be fabricated by micro-forming and this means that very small grain sizes, and especially materials having ultrafine grain sizes, are attractive for use in micro-forming operations. In this study, ultrafine-grained (UFG) materials including pure aluminum and a magnesium alloy were produced by equal-channel angular pressing (ECAP) and high-pressure torsion (HPT), respectively. The formability of UFG materials was evaluated by measuring the percentage of material flowing into the V-groove. The results show that refinement of grain size can significantly improve the formability by increasing the strain rate sensitivity by comparison with the coarse-grained materials. Micro-array channels with high dimensional accuracy and surface quality were successfully fabricated by micro-embossing processing using a silicon die. It is concluded that UFG materials exhibit excellent formability for fabricating micro-electro-mechanical system (MEMS) components. Keywords: micro-forming, size effect, ultrafine grains, formability, micro-embossing

1131 Microstructure of fiber laser deposited WC-Co cemented carbide and carbon steel

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A simple thermal-mechanical model is developed to explain semi-quantitatively the role of temperature gradient, process parameters, deposit shape, stray grain, and crack potential in laser deposition. Using a fiber laser welding head, crack-free WC-Co/Steel weld depositions are obtained with appropriate welding parameters. The microstructure, composition, phase, structure, and bend strength are analyzed using optical metallography, scanning electron microscopy, X-ray diffraction, transmission electron microscopy, and bend test. The influence of the microstructure, WC/WC, and WC/Co interfaces in the vicinity of joint failure at the cemented carbide side is discussed. It is evident that the deposit consists of austenite dendrites and interdendritic eutectic carbides. The austenite further transforms to martensite on-cooling. The flexural bend strength and yield strength of the joints attained 970 MPa and 876 MPa, respectively. Bend fracture occurs at the location of HAZ near cemented carbide of the deposition, characterized with cleavage fracture and quasi-cleavage fracture. TEM and HRTEM image of WC/WC and WC/Co interface verified the W₂C and eta phase formation.

1132 Transformation mechanism and microstructure optimization of a novel high strength high ductility hot rolled medium Mn steel

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The development of the 3rd generation high strength steels mostly concerns cold rolled steels and relies on the continuous annealing process. Being one of the most active concepts, the development of medium Mn steels mostly focuses on the intercritical annealing and revert austenite transformation, which results in a high product of tensile strength and total elongation, but relatively low yield strength. In order to develop high gauge 3rd generation high strength steels possessing high yield strength, the current work aims to explore fundamental aspects of a novel hot rolled medium Mn steels combining high yield strength and high product of tensile strength and total elongation. The work focuses on the hot rolling process and takes full advantages of the texture control and very slow cooling of a hot rolled coil. The prolonged (quasi) isothermal treatment allows the slow transformation to take place and results in microstructure of martensite/bainite embedded with retained austenite. The research carries out well-designed systematic dilatometer experiments and profound microstructure characterizations, in order to elaborate complex transformation mechanisms during a prolonged isothermal treatment between M_s and M_f. The resulting complex microstructure, promising mechanical properties and their correlations will be discussed. New proposal to further optimize the microstructure and mechanical properties will be presented.

1133 Catalytic properties of Ni₃Sn intermetallic nanoparticles fabricated by thermal plasma process

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Intermetallic nanoparticles are expected to be applied as catalysts for the applications to environmental cleanup and energy production. Recently, we found that Ni₃Sn intermetallic compound showed high catalytic activity and selectivity for methanol decomposition at high temperatures (above 800 K), whereas its activity at low temperatures was quite low. In our previous work, the Ni₃Sn powder samples were fabricated by mechanical milling of arc-melted ingots, which had a particle size larger than several micrometers in diameter. This led to a low specific surface area, which is considered to be the reason for the low activity at low temperatures. In this study, we attempt to fabricate Ni₃Sn intermetallic nanoparticles, and so as to enhance the catalytic properties of Ni₃Sn for hydrogen production from methanol at low temperatures. The Ni₃Sn (Ni-25at%Sn) intermetallic nanoparticles were fabricated using a high-frequency plasma method. The fabricated nanoparticles showed much higher catalytic activity for methanol decomposition below 800 K, compared to the Ni₃Sn powders fabricated by mechanical milling. The nanoparticles before and after reactions were characterized using XRD, TEM, SEM, and BET surface area measurement. Based on the characterization, the possible reasons for the enhancement of catalytic properties were discussed. The details of the results will be shown in our presentation.

1134 Creep study of martensitic steels developed within the project Z-ultra: Experiments and modelling

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In this work we summarize the main results obtained from the creep experiments and modelling carried out within the EU project Z-ultra. In this project, creep resistant martensitic/ferritic alloys have been developed. The new material concept is based on exploitation of Z-phase precipitates as strengthening particles. Creep experiments, both interrupted and continuous, have been carried out in order to determine the creep rupture strength of developed alloys. The first developed alloy ZL3 has shown better creep properties compared to grade P91 steel, but it is less creep resistant compare to the grade P92. Among the next set of developed alloys ZU1-3, ZU1 has shown very promising results in short term creep tests. A physical based model coupled with continuum damage mechanics (CDM) was developed to model the creep curves. The model describes the three types of dislocations: mobile, dipole and boundary dislocations. The precipitation kinetic is simulated using MatCalc software for uncrept condition and is assumed to remain unchanged during creep. Physical parameters were taken from literature and internal friction tests. The experimental creep curves of steel ZU2 were compared with model based creep curves at different stress levels. The predicted dislocation density and subgrain size have shown good agreement with empirical relationships.

1135 Framework structures for magnesium battery cathodes

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Rechargeable Mg batteries have received intensive attention as affordable rechargeable batteries with high electromotive force, high energy density, and high safety. Mg possesses two valence electrons and has the lowest standard electrode potential (ca. -2.36 V vs. SHE) among the air-stable metals. There is another advantage that Mg metal can be used as an active material because Mg metal hardly forms dendrites. However, the slow diffusion of Mg ions in solid crystals prevents the realization of active materials for Mg rechargeable batteries at room temperature. Although some complex oxides have been reported to work as active materials at higher temperatures, Chevrel compounds are still the gold standards, which work at room temperature. However, the working voltage of the Mg battery using a Chevrel compound for the cathode is only ca. 1.2 V, which is far below that of Li-ion batteries (3-5 V). Nevertheless, Chevrel compounds have the significant advantage that a relatively large space exists in the crystal structure, which allows for fast Mg ion diffusion. In the present study, we investigated some materials with framework structures as cathodes for Mg batteries, which can alleviate the electrostatic constraint between Mg ions and cathode constituents. Specifically, we investigated the redox behavior of the thin films of Prussian blue and Prussian blue analogs in electrolytes containing an Mg salt using electrochemical quartz crystal microbalance and X-ray absorption spectroscopy. In the presentation, we will also discuss the electrochemical insertion/extraction behavior of Mg ions and their solvation structures.

1136 Further study on the effect of environment on fatigue crack growth behavior of 2000 and 7000 series aluminum alloys

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The 7000 series alloys have the highest strength in the aluminum alloys, but lower fatigue properties than 2000 series alloys. Thus, 7000 series alloys are not applied to a large proportion of the aircraft components. However, the mechanism for this has not been elucidated yet. In humid air, hydrogen embrittlement based on intergranular cracking has been known to occur in 7000 series alloys. To date, in order to explain the difference in the fatigue crack growth behavior in the two series alloys, the effect of the test environment on the fatigue crack growth of the two series alloys has been investigated in this study. A 7075-type as well as 2024-type alloy with relatively coarse equi-axed grains was T6-tempered and subjected to fatigue crack growth test in humid and dry environments. Crack growth rate at low ΔK level seemed to be larger in the 7075-type alloy than the 2024-type alloy in the humid air, when assessed by means of gradually decreasing K method. In order to clarify this result, crack growth rate of the two alloys was assessed by means of gradually increasing K method as well as decreasing K method. Crack growth rate of the 7075-type alloy in moist air was concluded to be the largest in consistent with the previous study. Thus, the large fatigue crack growth rate of the 7075-type alloy is attributable to hydrogen embrittlement.

1137 Crystallographic features of electronic states in the highly-correlated electronic system $\text{Sr}_{1-x}\text{Sm}_x\text{MnO}_3$ around $x = 0.50$

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The highly-correlated electron system $\text{Sr}_{1-x}\text{Sm}_x\text{MnO}_3$ (SSMO) with the simple-perovskite structure has been found to exhibit fascinating electronic states accompanying antiferromagnetic and ferromagnetic orderings. It was, in particular, reported that the electronic state for $0.46 \leq x \leq 0.54$ was characterized by the coexistence state consisting of the A-type antiferromagnetic and ferromagnetic states. However, the features of the coexistence state in this Sm-content range have not been understood yet. We have thus investigated the crystallographic features of prepared SSMO samples with $0.46 \leq x \leq 0.55$, mainly by transmission electron microscopy. As a result, all prepared SSMO samples were first confirmed to have the orthorhombic-Pnma structure at 300 K. When the temperature was lowered from 300 K, in the case of $x=0.47$, the disordered-Pnma state was found to be transformed into an orbital-modulated (OM) state accompanying an incommensurate modulation. The notable feature of the OM state is that the state becomes unstable with increasing Sm contents at 100 K. In other words, the OM state was never changed into the CE-type state with the orbital and charge modulations. In addition, no orbital-ordered state for the A-type antiferromagnetic ordering was also found for $0.46 \leq x \leq 0.55$.

1138 Development of chalcogen-excess metal chalcogenides with using high-pressure synthesis technique

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High-pressure method is superior to conventional solid-state method for material search in some points, for example, stabilization of high-pressure phase, suppression of loss of volatile elements, and stabilization of unusual valence. Recently, we have developed some chalcogen-excess metal chalcogenides, BiSe_2 , BiS_2 , $\text{Nb}_{1-x}\text{Ta}_x\text{Se}_3$ ($x=0-1$) and others with using high-pressure technique. We will introduce our study of these metal chalcogenides. Possible existences of high-pressure phases, BiSe_2 and BiS_2 , have been suggested in 1970's, but no structure and properties were reported. We have lately stabilized these phases under 5.5GPa using cubic-anvil-type high-pressure apparatus[1]. X-ray structure refinements using single crystals of BiSe_2 and BiS_2 showed that they have isostructure with quasi-one-dimensional linking with monoclinic cell. The analysis suggests that Bi has trivalent $3+$ while Se/S has two types of simple chalcogen ion, $\text{Se}^{2-}/\text{S}^{2-}$ and dimerized ion, $(\text{Se-Se})^{2-}/(\text{S-S})^{2-}$. They are semiconductors and diamagnets. We also stabilized $\text{Nb}_{1-x}\text{Ta}_x\text{Se}_3$ ($x=0-1$) under high pressure of 3GPa. As is well known, NbSe_3 shows charge-density-wave (CDW) at 40K and 150K, and the suppression of CDW induces superconductivity with T_c of 2K and 6K. Though Ta concentration of x was limited up to 0.15 with conventional method, the concentration could be expanded with the range of x up to 1 by high-pressure method. It was proved that superconductivity was observed with wide range of x . We will discuss of the structure and

properties including other chalcogen-excess metal chalcogenides. [1] A. Yamamoto, Inorg. Chem., 54(2015), 4114

1139 Surface structural changes of Pd-Cu-Ge metallic glass thin films upon glass transition and crystallization

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The Pd₄₀Cu₂₀Ge₄₀ metallic glass exhibits glass transition at around 50 °C and crystallizes at around 100 °C, which are remarkably lower than those of typical metallic glasses. Because of low glass transition temperature and crystallization temperature, the surface oxidation of the Pd-Cu-Ge metallic glass can be suppressed upon heating to glass transition and crystallization temperature, when compared to the typical metallic glasses. Thus, Pd-Cu-Ge metallic glasses are useful for observation of structural changes upon heating. In this study, changes in density, thickness and surface roughness of Pd-Cu-Ge metallic glass thin films were examined by means of X-ray reflectometry. Pd-Cu-Ge metallic glass thin films were deposited on slide glass substrates. The thin films were divided into several pieces. Small pieces of the thin films were subjected to electric resistance measurement upon heating and cooling to confirm glass transition temperature and crystallization temperature. Large pieces were subjected to X-ray reflectivity measurements after heated up to glass transition temperature or crystallization temperature followed by cooling to room temperature. Density, thickness and surface roughness of the thin films were estimated though comparison between the experimentally measured reflectivity and theoretically calculated one. The density increased upon crystallization, the thickness decreased upon both glass transition and crystallization, and the surface roughness decreased upon glass transition and increased little upon crystallization.

1140 STEM and TEM observations of defects distribution of Ge/Si annealed by new heating method using plasma technique

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It is a well-known fact that Ge has a potential to be a base material for the high-speed transistors in a near future because of its high carrier mobility. In order to realize high-speed devices using Ge, formation of Ge/Si hetero-structure is a practical road rather than using a bulk Ge crystal. Therefore it is important to grow high quality Ge thin films onto Si substrate. The key point is how to produce a Ge film which has both flat surface and high crystallinity. Recently our group proposed a new heating method using plasma technique. This new method enabled us to heat specimens quite rapidly. For example, we could heat the specimen up to 1000 °C within 1 second. Thus we could anneal the Ge/Si specimen to remove defects located around the Ge surface before progressing the inter-diffusion between Ge and Si. In this study, we carried out TEM observations to evaluate the dislocation distribution of the Ge/Si specimens produced by this new heating method. We also carried out precise elemental analyses by using a super high sensitivity built-in EDX and make it clear whether or not Si was diffused into Ge. We also

examined a new STEM method, “Strain Analysis Using STEM Moire Method” which was proposed by other researchers, Yukihito Kondo and Noriaki Endo. (Kenbikyo, Vol. 49, 2014, 226.) These analyses exhibited that we were succeeded in localizing the most of the dislocation around the Ge/Si interface and forming a lattice-relaxed Ge with flat surface.

1141 Effect of nitrogen on age-hardening of metastable austenitic stainless steel after cold drawing

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Metastable austenitic stainless steels transform to the deformation-induced martensite by cold working. Especially, metastable stainless steel with high N content has high age-hardening property after aging treatment. In this work, effect of nitrogen on age-hardening of metastable austenitic stainless steel (SUS304: 0.04% N, type-SUS201: 0.18% N) after cold drawing was investigated, and age-hardening mechanism was discussed. Strength after cold drawing of SUS201 containing high N is higher than that of SUS304, and the age-hardening of SUS201 is significantly higher than that of SUS304 at the aging temperature of 200 ~ 500°C. It is suggested that strengthening mechanism of SUS201 is caused by aging products of N, because exothermal reaction in SUS201 is clearly recognized at low aging temperature by DSC analysis.

1142 Effects of Au and Pd additions on plastic deformation of Zr-Cu-Ni-Al bulk metallic glasses

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Zr₅₅Cu₃₀Ni₅Al₁₀ (Zr55), Zr₆₅Cu₂₀Ni₅Al₁₀ (Zr65) and Zr_{65+x}Cu_{17-x}Ni₅Al₁₀M₃ (Zr65+xM3, x = 0, 1.5 and 3 at.%, M= Au and Pd) bulk metallic glasses (BMGs) have been prepared by a tilt casting method. DSC results of the Zr65-BMG indicated the presence of a wide supercooled liquid region of 119 K between the glass transition temperature (T_g) of 644 K and the crystallization temperature (T_x) of 763 K. In contrast, by the Au/Pd addition BMGs, precipitation of an icosahedral quasicrystalline phase (I-phase) was observed in their supercooled liquid temperature regions. Compressive test was carried out at an initial strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at room temperature. By the Au/Pd additions, the yield stress was almost unchanged but the plastic strain at fracture was increased largely. The plastic strains at fracture of Zr55-, Zr65- and Zr65Au3/Pd3-BMGs were about 2 %, 10 % and more than 20 %, respectively. In the Zr66.5Au3/Pd3-BMGs, stress-increasing like a work hardening during plastic deformation was observed until more than 5% of compressive strain. By the transmission electron microscopy of the Au/Pd-addition BMGs, precipitation of nanocrystallites having about 5~10 nm in diameter was observed near the fractured surfaces of the compressive test specimens. So, the large increase of the plastic strain may be due to the work hardening by the dynamic precipitation of I-phase during plastic deformation.

1143 Effect of chlorine atoms for development of aluminum corrosion

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Aluminum and its alloy have good corrosion resistance upon exposure to the atmosphere. However, the corrosion in aluminum typically occurs at the microscopic level for which is known as pitting corrosion. Development and growth of the pitting area causes serious problem for application of aluminum in various fields such as structural and electrical engineering. Understanding of the fundamental mechanism of developing corrosion in atomic level is great important process to prevent the phenomena. It is known that existence of chlorine cause surface degradation significantly of aluminum. Preliminary computational density functional theory (DFT) modeling of adsorption chlorine to pure aluminum surface was performed. All calculations are performed employing ultra-soft pseudo-potential and plane wave method (GGA, PAW). The Al (111) slabs of (1x1), (Sqrt3 x Sqrt3) and (2x2) structures are five atomic layers thick and vacuum layer (25 angstrom) is oriented to z-direction. Adsorption energies of chlorine atom on-top, bridge, hcp and fcc sites on Al(111) slab are compared. Coverage dependence of the energetically most favorable site of adatom is examined in each configurations such as high coverage of chlorine tends to be stable on FCC site, while lower coverage to on-top site. Detailed analysis of fundamental process of pitting corrosion by using more realistic modeling which have water molecules are now in progress.

1144 Materials development for the realization of carbon-neutral energy cycles

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The CO₂ concentration of the atmosphere has been inadvertently increased by consuming finite energy resources of fossil fuels, and increased concerns about climate issues due to CO₂ emissions have spurred the development of alternative and sustainable energy cycle systems. We have proposed novel energy cycles where electrical power is generated by partial oxidation of alcohols without CO₂ emission, and fuels are regenerated from oxidized wastes by using renewable energies, resulting in CO₂-free power circulation ,i.e., carbon-neutral energy (CN) cycles. We herein demonstrate the successful synthesis of a well-mixed Fe-group nanoalloy (NA) catalyst, a carbon supported FeCoNi NA catalyst (FeCoNi/C), that exhibits selective ethylene glycol (EG), a divalent alcohol, electrooxidation to oxalic acid (OX) without CO₂ emission in alkaline media. Furthermore, an alkaline fuel cell fabricated with the FeCoNi/C anode catalyst and a solid oxide electrolyte was found to exhibit power generation from EG without any precious-metal catalysts. Renewing alcoholic compounds by direct electroreduction of OX is another challenge, which enables highly efficient electric power circulation. To the best of our knowledge, the electroreduction of carboxylic acids to alcoholic compounds has never been achieved. We could demonstrate OX electroreduction into glycolic acid, an alcoholic compound, with significantly high selectivity (>98%) using pure anatase-type titania spheres under mild conditions. These results are the first demonstration of a CO₂-free electric power circulation using carbon containing compounds.

1145 Initiation and propagation behaviors of small crack in a polycrystalline Ni-base superalloy under thermos-mechanical fatigue loading

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Propagation behaviors of naturally initiated small crack in a polycrystalline Ni-base superalloy, IN738LC, under thermo-mechanical fatigue loading were investigated. TMF and isothermal low-cycle fatigue (LCF) tests were carried out under a strain ratio of -1 at temperature ranged between 400 and 880 °C. In the former the tests were performed under the in-phase (IP) and out-of-phase (OP) conditions in which the phase difference between strain and temperature were 0° and 180°, respectively. The experimental results revealed that the initiation and propagation morphologies of the naturally initiated crack were affected by thermo-mechanical loading condition. The small cracks initiated and propagated at the grain boundaries perpendicular to the loading axis under the LCF and IP conditions. On the other hand, they initiated and propagated with the transgranular modes under the OP condition. The EBSD analysis and the finite element analysis considered with crystal orientation of the grain structure were carried out. Based on the results of the crystallographic investigations, the crack initiation and propagation mechanisms were discussed.

1146 Research on microstructure evolution in Al-9.8Zn-2.0Mg-1.8Cu alloy during solution treatment

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The microstructure of as-extruded Al-9.8Zn-2.0Mg-1.8Cu aluminum alloy and its evolution during solution treatment were investigated by means of optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), differential scanning calorimetry (DSC) analysis and electron back-scatter diffraction (EBSD). The results indicated that secondary phase of the as-extruded alloy mainly consisted of Mg(Zn, Cu, Al)₂ and Fe-rich phases. After solution treated at 475°C for 4h, Mg(Zn, Cu, Al)₂ phases were dissolved into the matrix, while Fe-rich phases still existed. Fe-rich phases cannot dissolve by prolonging solution time. The room temperature tensile strength gradually increased by prolonging solution time at 475°C. The ultimate tensile strength reached 700MPa treated by both single and two-step solution treatments, and the fracture toughness of the alloy is improved by two-step solution treatment.

1147 Crystal structure, microstructure and martensitic transformation path in Ni-Mn-In alloys

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Due to the giant magnetic-field induced strain and magnetocaloric effect, Ni-Mn-In alloys have received considerable attention for use as high performance actuator and magnetic refrigerant materials. The multi-functionality is found to be closely related to the strong coupling between crystal structure and magnetism during martensitic transformation. However, due to the complexity of the modulated structure, the crystallographic investigation of microstructure and martensitic transformation path are severely blocked. In this work, the crystal structure of martensite is first determined by means of Rietveld method in the frame of (3+1)-dimensional superspace theory [1]. Results show that the martensite has an incommensurate 6M modulated structure. Then, the martensitic transformation path is uniquely determined on the basis of the crystallographic orientation relationship between austenite and martensite and the measured martensite microstructure features. It is found that martensitic transformation obey Pitsch path, i.e. $\{110\} \rightarrow \{110\}$. Furthermore, the crystallographic features of martensite microstructure, such as variant organization and variant interface, are studied theoretically and experimentally. The martensite is organized in colonies. The maximum of 6 distinct colonies that are crystallographically equivalent can be generated within one initial austenite grain. Each martensite colony consists of 4 kinds of variants that are associated with the same or opposite transformation plane and direction. This study is expected to supply a fundamental information of crystal structure, microstructure and martensitic transformation path in Ni-Mn-In alloys, to understand the underlying mechanisms of their multifunctional magneto-responsive behaviors. [1] H.L. Yan, Y.D. Zhang, N. Xu, et. al, Acta Mater. 88 (2015) 375-388.

1148 Uniaxial tensile behavior of Cu-Al alloys subjected to low-cycle pre-fatigue deformation

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To explore the effect of pre-fatigue deformation on static mechanical properties of structural materials, Cu-7at.%Al and Cu-16at.%Al alloys, respectively with stacking fault energies (SFEs) of 24 and 6 mJ/m², are selected as the target materials in the present work, and the mechanical behavior, microstructures and deformation features after pre-fatigue at total strain amplitude ranging from 1.0×10^{-3} to 5.0×10^{-3} and subsequent uniaxial tension were investigated. The cyclic flow stress of two alloys increases with increasing strain amplitude, and a stress saturation phenomenon only occurs in Cu-7at.%Al alloy as the strain amplitude reaches as high as 2.3×10^{-3} . It is found that Cu-7at.%Al alloy presents mixed planar/wavy slip dislocation structures at higher strain amplitudes. In this case, an obvious strengthening effect is induced by low-cycle fatigue training due to the formation of a large quantity of deformation twins with a nano-size thickness, meanwhile, a slight expense in tensile ductility is produced as a result of pre-fatigue damage. For a low SFE material of Cu-16at.%Al alloy, pre-fatigue deformation introduces very typical planar slip dislocation structures, and a notable strengthening effect is produced at a low strain amplitude of 1.0×10^{-3} nearly at no expense in

ductility due to the combined effects of the increased dislocation density in planar slip bands and the formation of deformation twins. On the whole, a low-cycle pre-fatigue training on Cu-Al alloys at an appropriate strain amplitude can cause an obvious strengthening effect on their static mechanical properties.

1149 Microstructure and mechanical properties of Nb alloyed steel processed by hot equal channel angular extrusion

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Shear deformation is known to be effective to produce an ultra fine grain. However, the research of grain refinement by phase transformation after severe shear deformation for low carbon steel was limited in a torsion test. The grain size of 2 μ m was obtained by the torsion test. On the other hand, the single pass equal channel angular extrusion (ECAE) process was achieved for SUS 304 steel. Because a repetitive hot ECAE process and rapid ejecting the workpiece from die is difficult for 1 axis press. The research of phase transformation after hot ECAE process has not been conducted. Then we newly developed an ECAE process equipment which enabled the repetitive hot ECAE process without ejecting workpiece. This equipment has T-shape 3 axis in horizontal plane. A Nb alloyed low carbon steel was used in the study. The workpieces were heated up to the 960°C in the furnace for 10 min within the container block or without the container. The workpiece size is 8mm square \times 40mm length. A die with three channels intersecting at inner angle of 90° and outer angle of 0° was used. Before extrusion, the die was preheated to 400°C. The workpiece was cooled in the die after ECAE process. 1 pass and 2 passes via route C were conducted at a speed of 16mm/s, the inter-pass time is about 2 sec. The sample of average ferrite grain size of about 2 μ m, a tensile strength of 780MPa, a uniform elongation about 15% is produced after two passes process and subsequent cooling.

1150 Effect of Sr addition on the solidification structure in Al-6mass%Mg-3mass%Si alloy

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Al-Mg-Si system alloy have good strength and high ductility without heat treatment. Therefore, the application is expected as high-pressure die-casting alloy used for chassis and body components of automobile. However, there is a technical problem that hot-tearing is occurred in casting process. In our previous study, it was shown that the hot-tearing decreased by Sr addition because the shape of the solidification structure was changed. In this study, in order to clarify the mechanism that changes the crystallization shape by Sr addition, the effect of the Sr contents on the solidification structure in Al-6mass%Mg-3mass%Si alloy was investigated.

1151 Grain boundary engineering of ECAPed OFHC copper

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In this paper, we demonstrate a novel method for grain boundary engineering in OFHC (oxygen-free high-conductivity) copper using equal-channel angular pressing and strain annealing to modify the grain boundary character distribution (GBCD) for improved intergranular corrosion (IGC) resistance. The GBCD and its effect on the intergranular corrosion resistance of copper after solid solution and ECAP deformed annealing were investigated by means of electrochemical test, static weight loss method, and electron back-scattered diffraction (EBSD). The possible mechanism of the effect of low Σ coincidence site lattice (CSL, $\Sigma \leq 29$) on intergranular corrosion resistance were discussed. The experimental results showed that ECAP can refine the grain size of copper and enhance its hardness, but reduce its resistance to intergranular corrosion simultaneously. However, annealing treatment at 1133K for 2 min after 1 pass ECAP could improve the intergranular corrosion resistance of copper remarkably, the reason could be attributed to increased fraction of special grain boundaries whilst decreasing the connectivity of random grain boundaries in the altered microstructure. A discontinuous random grain boundary network and a large amount of low CSL boundaries, which arrest the propagation of the corrosion cracks. ECAP, followed by annealing, may therefore be used to control the grain boundary character distribution and improve the intergranular corrosion resistance of copper.

1152 Directional solidification structures and room temperature mechanical properties of Mg-Gd magnesium alloys

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Directional solidification of Mg-xGd (x=1.38, 2.35, 4.38) (wt.%) magnesium alloy was carried out to investigate the effects of the solidification parameters (growth rate V and temperature gradient G) on the microstructures and room temperature mechanical properties. The specimens were solidified under steady state conditions with different temperature gradients ($G=20, 25$ and 30 K / mm) at a wide range of growth rates ($V=10 - 200$ $\mu\text{m} / \text{s}$) by using a Bridgman directional solidification furnace with liquid metal cooling (LMC) technology. The cellular microstructures are observed in Mg-1.38Gd and Mg-2.35Gd alloys. The cellular spacing λ decreases with increasing V for constant G of 30 K / mm or with increasing G for constant V of 10 $\mu\text{m} / \text{s}$. The measured values are in good agreement with the corresponding values calculated by Trivedi model. However, for Mg-4.38Gd alloy, the microstructure changes from cellular structure to dendrite structure when V is up to $100 \sim 200$ $\mu\text{m} / \text{s}$ under the fixed G of 30 K/mm. The main reason for this may be the joint effects of the higher solute content and the interfacial tension. It is also found that the directionally solidified experimental alloy exhibits higher strength than the non-directionally solidified alloy under the same cooling rate, and the tensile strength of the directionally solidified experimental alloy was also improved, while the

corresponding elongation decreased with increasing V for constant G of 30 K / mm.

1153 Formation of hierarchical intra-splat crack patterns in plasma sprayed ceramic splats

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The intra-splat cracks in plasma sprayed ceramic splats arises from the large stress during rapid cooling of ceramic splat. The final crack patterns are often regarded as extremely complex and irregular, and the detailed formation mechanism of intra-splat crack patterns is scarcely concerned. In this study, the formation mechanism of intra-splat crack patterns in splats is presented. The crack patterns approximately present hierarchical structure with four sides and six neighbors, which indicates they arise from successive domain divisions due to sequentially residual stress produced by substrate constraint during splat cooling. The experiment value of minimum crack spacing for TiO_2 splat is about $4.54 \mu\text{m}$ which is in good agreement with theoretical value $4.43 \mu\text{m}$. In addition, the difference of crack spacing at splat center and splat periphery was also clarified to comprehensively understand the whole crack patterns. These results would shed light to the further coating microstructure tailoring towards high performance and durable plasma sprayed coatings.

1154 Novel structures of TiO_2 films prepared by modified hydrothermal method

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Anatase TiO_2 has attracted much attention because of its unique electronic, optical and catalytic properties. The high energy (001) surfaces showed much higher chemical activities than the normal (101) surfaces in anatase TiO_2 crystals, therefore, it is desirable to prepare TiO_2 crystals with more reactive {001} facets exposed. In this study, we provided an environmental-friendly method, a modified hydrothermal method, to prepare anatase TiO_2 film with exposed {001} facets. By this method, the direct contact of ATONAs with HF solution was avoided, the reaction between gas phase HF (but not acid HF solution) and solid ATONAs played a key role in the transformation process from amorphous to anatase TiO_2 , accordingly porous anatase TiO_2 film with exposed {001} facets up to 76.5% could be successfully prepared at temperature as low as 130°C . After the surface F^- ions were removed by 600°C annealing for 2 h, the obtained F^- free TiO_2 films with exposed {001} nanofacets showed improved photocatalytic activities for methyl orange (MO) degradation.

1155 Damage healing and twinning mechanisms of a nano-grained high-nitrogen austenitic stainless steel processed by electropulsing treatment

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A nano-grained high-nitrogen austenitic stainless steel (HNS) obtained from high-pressure torsion (HPT) technique was processed via electropulsing treatment (EPT). The microstructural evolution, especially the damage healing behaviors were studied through various characterization techniques. Rapid recrystallization and suppressed grain growth occurred in the material during EPT due to the fast heating and cooling rate. In the same time, the deformation defects like dislocation, shear band, nanovoid and micro-crack were dramatically reduced and a uniform ultrafine grained microstructure with high density of nanoscale twins were then formed. The damage healing and twinning behavior were illustrated by a selective recrystallization mechanism and a physical model was then proposed.

1156 Quantitative understanding of anomalous slip in bcc metals

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Hexagonal dislocation networks (HDNs) formed by the reaction of $\langle 111 \rangle/2$ screw dislocations are tightly correlated with anomalous slip in body centered cubic metals, but its role assigned in anomalous slip remains obscure due to the absence of quantitative description of its response to uniaxial loading. Here systematic atomistic simulations are performed in Mo and other metals to study the responses of a HDN to different loadings. The results not only confirm that anomalous slip occurs by the HDN motion, but provide an quantitative yield criterion for the HDN motion, from which the transition from primary to anomalous slips with the loading direction is predicted to be consistent with experimental observations.

1157 Formation of transformation textures enhanced by deformation

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Orientation relationship often exists during transformation. The crystallographic symmetries lead to abundant variants and the weakness of texture in new phase. However, some factors may promote variant selection during transformation, thus the texture in new phase may become stronger and will influence the mechanical properties of alloys. Such factors may be the deformation, the surface effect, the anisotropic elasticity or un-uniform distribution of crystal

defects. In this paper the evolutions of transformation textures in Ti-alloys, high manganese TRIP steels and Fe-Mn electrical steel are presented and interpreted using EBSD technique and matrix analysis. The forged Ti alloy bars often contain a strong $\langle 100 \rangle$ fiber texture in the center before the transformation from BCC beta-phase to HCP alpha-phase, and different variant selection tendencies are detected depending on the state of recovery in beta-phase and alloy compositions. In the case of high Mn- TRIP steels, strong orientation dependence can be detected, namely, the $\langle 100 \rangle$ BCC alpha-martensitic grains preferentially form in compressed $\langle 100 \rangle$ austenitic grains, rather than the $\langle 110 \rangle$ grains according to K-S orientation relationship. A $\langle 100 \rangle$ transformation texture is thus resulted. The third example occurred due to surface-effect induced transformation in Fe-0.5Mn electrical steel sheet subject to cold rolling and subsequent controlled transformation in hydrogen atmosphere to produce $\{100\}$ columnar grains at surface together with a lot of sigma-3 relation between ferritic grains. The influencing factors related with the variant selection during transformation are discussed in terms of matrix analysis. Such information is important for transformation texture control.

1158 The effect of grain size on oxidation resistance of pure titanium

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The effect of grain size on high temperature oxidation resistance for pure titanium was experimentally investigated with two different grain sizes of 219 micron and 118 micron. Results exhibited faster oxygen diffusion in the substrate of the small grain sample. In the small grain size sample, the weight gain during oxidation and the penetration depth of oxygen from metal surface were larger than those in the large grain sample. These differences may be attributed to the grain boundary diffusion of oxygen. A steep change in oxygen concentration was observed at a grain boundary. Our simulation results suggested that the slower oxygen diffusion into the inner grain from surface through the grain boundary with high diffusivity causes that steep change in oxygen concentration.

1159 Laser direct metal deposition of M2 high speed steel: Microstructure evolution and crystallization behavior during annealing

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In the current investigation, the M₂ high speed steel (HSS) sample was produced by an additive manufacturing (AM) process, laser direct metal deposition (DMD). The observed microstructure from SEM and XRD consisted of a cellular or dendritic structure of ferrite, martensite, retained austenite and fine carbides. The overall microstructure from LOM was characterised to be a gradual transition from bottom to top due to the continuous decrease of the cooling rate. Post- annealing at 860 °C leads to spheroidization of carbides and recrystallized. The carbides were examined by XRD to be M₆C and MC. With prolonging the

annealing holding time, the more homogenous microstructure could be acquired. These studies demonstrate that post-annealing can improve microstructures of M₂ HSS produced by DMD.

1160 Mg-Zn-Y alloys with long-period stacking ordered phases: deformation, creep, solute segregation and strengthening mechanisms at elevated temperatures

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In this talk, the mechanical performances, plastic deformation and creep at elevated temperatures, as well as the atomic origin of superior mechanical properties of a Mg-Zn-Y alloy with long-period stacking ordered (LPSO) phases will be presented. The LPSO-strengthened Mg alloys have superior strength at temperatures as high as 473 K, which is in stark contrast to commercial AZ Mg alloys. The creep strain is only 0.01% under a tensile stress of 70 MPa at a testing temperature of 473 K. We investigated the microstructures using various electron microscopy techniques, in order to obtain a comprehensive understanding of the underlying mechanisms for the superior high-temperature strength and creep resistance. Kinking of the LPSO phases plays an important role in strengthening and toughening the alloy. Kinking of the LPSO is a result of generation, motion and rearrangement of basal dislocations, which strengthens the alloy due to microstructural refinement, and affords remarkable plasticity and simultaneously strong resistance to propagation of microcracks. Atomic-resolution imaging demonstrated the occurrence of Suzuki segregation in Mg matrix during both plastic deformation at a strain rate of $1 \times 10^{-3} \text{ s}^{-1}$, and a creep rate of $1.5 \times 10^{-7} \text{ s}^{-1}$ at 573 K. Suzuki segregation not only impeded the motion of the dissociated dislocation, but also could act as obstacles for other moving dislocations. Therefore, Suzuki segregation has also important contribution to the superior mechanical performances of this Mg alloy at elevated temperatures. In addition, Cottrell atmospheres were observed in both the LPSO phases and Mg grains in samples plastically deformed samples.

1161 Research on the crystallization kinetics and glass-forming ability of a Ti-based bulk metallic glass

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Ti-based bulk metallic glasses (BMGs) are very attractive due to their low density, high strength and high specific strength. However, if compared with other bulk glassy alloy systems, such as Zr-based or Pd-based BMGs, the glass-forming ability (GFA) of Ti-based BMGs is relatively low, despite that many progresses have been obtained in recent years. Then, improving the glass-forming ability, thermal stability and mechanical property of Ti-based glassy alloys is important and meaningful. In present work, the non-isothermal crystallization kinetics and glass-forming ability of Ti₄₁Zr₂₅Be₂₈Fe₆ glassy alloy have been investigated by differential scanning calorimetry. The activation energies corresponding to the characteristic temperatures have been calculated by Kissinger and Ozawa equations. Based on reported models, it has been

found that the local activation energy is higher at the beginning of the crystallization process for the first exothermic peak. The calculated result of the fragility index shows the Ti₄₁Zr₂₅Be₂₈Fe₆ glassy alloy is a “strong glass former.” The studied alloy also possesses a critical size up to centimeter order, and the high GFA has been discussed based on the thermal stability of the alloy melt.

1162 Flow behavior of severely deformed titanium at elevated temperatures

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There has been a great deal of research concentrating on ultra-fine grained (UFG) microstructures via severe plastic deformation (SPD) with the ultimate goal of obtaining superior physical and mechanical properties. In probing the mechanical characteristics of UFG materials, flow behavior at high temperatures deserves attention both for enlarged operational envelopes and hot working studies. In this effort, compression experiments were performed at a strain rate of 0.001 to 0.1 s⁻¹ and in the range of 600°C to 900°C to investigate the high temperature deformation behavior of commercial purity titanium after SPD. Preliminary results underlining the effect of purity levels are also presented. It was revealed that the deformation temperature and strain rate are significant in dictating the steady state flow stress levels. Flow accompanied by thermal softening was observed due to a combination of dynamic recovery and recrystallization mechanisms for deformation at or above 600°C. Microstructural evolutions of the as-processed and hot deformed material were examined as well. Based on various constitutive equations, the flow behavior was modeled and reasonable predictions were obtained.

1163 Small-angle X-ray scattering contrast imaging in grating-based X-ray interferometry

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Grating-based X-ray phase imaging techniques have attracted much attention in the last decade because they allow for high-sensitive X-ray phase imaging with a low-brilliance laboratory X-ray source[1]. We have developed an imaging method using this interferometry that makes it possible to quantitatively visualize information on unresolvable microstructure for each pixel: the method makes it possible to generate an image with contrast due to small-angle X-ray scattering (SAXS) arising from unresolvable microstructures in a sample and obtain structural parameters characterizing the unresolvable microstructure at each pixel[2,3]. Our technique can be realized with a large size X-ray beam, while conventional scanning X-ray imaging techniques require a highly parallel X-ray microbeam. In this presentation, our previous and recent research on this method, including three-dimensional tomographic reconstruction of the unresolvable microstructures[4] and surface/interface characterization in grazing-incidence geometry[5], will be reported. [1] J. Als-Nielsen and D. McMorrow, “Elements of Modern X-ray Physics”, 2nd Edition (Wiley, 2011). [2] W. Yashiro, Y. Terui, K. Kawabata, and A. Momose, Optics Exp. 18 (2010) 16890-16901. [3] W. Yashiro, S. Harasse, K. Kawabata, H.

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1164 Photo-excitation-induced silicides formation in Pt/SiO_x bilayer film

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When materials are atomic species-, site- or electronic orbital-selectively excited by photons with variable energy, bond breaking or reaction between specific atoms take place. It was confirmed in our group that a platinum silicide, Pt₂Si, was successfully formed at the platinum/silicon oxide interface kept at room temperature under 25–200 keV electron irradiation. This result shows that the reaction cannot be induced by simple thermal annealing under no-electron-irradiation conditions. In the present study, the synthesis of platinum silicide at the platinum/silicon oxide interface by photo-excitation was investigated using synchrotron-radiation PES and TEM. Spectrum of silicon valence band and Pt4f_{7/2} core level did not change after photo-excitation by 80 eV photons. On the other hand, in the case of photo-excitation by 140 eV photons, peak near the Si 3p level in the Si valence band spectrum shifts to higher energy, and a peak originated from Si3p-Pt5d bond appears near the E_F. In Pt4f_{7/2} core level spectrum, the peaks shift to higher energy by 1.2 eV and remarkably change during Pt₂Si formation. It was confirmed that Si 2p core level excitation plays an important role in Pt₂Si silicide formation by reaction between silicon and platinum on Pt/SiO_x thin film interface, because the binding energy of Si 2p is approximately 99 eV. In order to produce Si-Pt bonds preferentially from Si-O-Pt bonds, simultaneous breaking of Si-O and O-Pt bonds and the consequent desorption of oxygen atoms and formation of Si-Pt bonds may be required by photo-excitation.

1165 Deformation behavior of Fe-Al-Co-Ti single crystals containing Co₂AlTi precipitates

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Fe-15Al-18Co-3Ti (at.%) single crystals containing the Co₂AlTi precipitates was examined. In the single crystals furnace-cooled (FC) from 1373 K to room temperature, coarse Co₂AlTi phase with the L2₁ structure was precipitated in the bcc matrix. The L2₁ phase showed a cuboidal shape with a misfit strain of 0.59%. It is also noted that large amount of Fe substituted for Co in the Co₂AlTi precipitates. The single crystals exhibited high yield stress above 600 MPa up to 823 K while further increase in temperature resulted in a decrease in yield stress. In the FC crystals, 1/2<111> dislocations in the bcc matrix bypassed the coarse L2₁ precipitates due to their large misfit strain, resulting in high strength. In contrast, the fine L2₁ precipitates about 30 nm in diameter were observed in the crystals after solutionization and annealing at 823 K. The crystals with the fine L2₁ precipitates demonstrated high yield stress above 1200 MPa at room temperature. Paired 1/2<111> dislocations cut the fine L2₁ precipitates, which led to high strength. The dependence of the yield stress on the precipitate size was also discussed.

1166 Drawing Fe-6.5wt.%Si wires with enhanced formability

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Fe-6.5wt.%Si alloy is hard to deform due to its low formability at room temperature. It hinders its industry application although the Fe-6.5wt.%Si alloy shows extremely low iron loss and much low noise when being used as iron core in transformer or electrical engine. This embrittlement results from the appearance of ordered phases, like B2 and D0₃ structures. It has been discovered that the formability of the Fe-6.5wt.%Si alloy can be enhanced by heat treatment and thermal mechanical processing which reduced the content of the ordered phases. Thin sheet of 0.05-0.3 mm in thickness can be obtained by cold rolling. Some special treatments can also be employed to enhance the formability, including electroplastic effect by electro pulse, and the chemomechanical effect in a electrochemical environment. The electro-pulse passing a specimen during deformation results in a softening and ductilizing. The electrochemical environment accelerates the dislocation moving so that its workability is improved. The technique was applied to drawing wires. Fe-6.5wt.%Si high silicon steel wires with a diameter of 0.5-2 mm are fabricated successfully by drawing. The as drawn wires show much better ductility than sheets. A tensile elongation above 4% was measured at the room temperature. The microstructure analyses show that the elongated grains after drawing and the disordering by deformation in the wires might contribute to its good ductility. Bs value of 1.437 T and Hc value of 16.96 A/m are obtained for the wire after proper heat treatment for the wires.

1167 Texture development during static recrystallization of Mg-Sn-Al-Zn alloys sheets

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By altering a strong basal-type texture which is unfavourable to accommodate the deformation by activation of basal <a> slip, the formability of Mg alloys sheet can be considerably improved. A number of studies have shown that the texture weakening induced by alloying addition of rare earth elements (RE) or Ca results in a high ductility and formability at room temperature. Different mechanisms of formation of more randomised texture have been proposed, such as higher activation of non-basal dislocations slip induced by RE addition, nucleation at various twins or shear band, and particle stimulated nucleation. In the present study, the recrystallization behaviour of two Mg alloy sheets with different alloying amount of Sn were examined to reveal the twin band nucleation on the texture development. The rolled sheets of these RE-free Mg alloys, which contain a lot of various twins, were recrystallized in different heat treatment schedules. A basal-type texture is developed by recrystallization annealing at the (Mg) single phase area on the phase diagram, while a rapid growth of equiaxed grains is observed. The annealing at (Mg) + Mg₂Sn dual phase area results in a significantly retarded grain growth due to Zener pinning, such that new recrystallized grains triggered at the twin (deformation) bands can be easily distinguished from the other grains. Based on the orientation changes during the recrystallization with respect to their origins, e.g. twin or matrix grains, the contribution of various mechanisms to the texture and microstructure development will be discussed, especially regarding texture weakening.

1168 Electrochemical behaviors of biomedical nano-grained β -type titanium alloys

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β -type Ti-29Nb-13Ta-4.6Zr (TNTZ) alloy have been extensively investigated as a structural material for surgical implant devices. Recently, microstructural control at ultrafine-grained (UFG) or nano-grained (NG) level for TNTZ has been investigated using high-pressure torsion to ensure long term healing. HPT has been proved incredibly effective method for fabricating the nano-grained TNTZ, which has been employed to obtain remarkable mechanical biocompatibility such as high mechanical strength (1100–1400 MPa) and hardness (300–450 HV) with an adequate ductility (7–12 % compared to Ti-6Al-4V ELI (1000 MPa, 310 HV, 25 %). The surface properties of TNTZ in the human body are important aspects to consider with regard to its corrosion resistance. However, the electrochemical behavior of such NG TNTZ has still not been investigated completely. Therefore, the electrochemical behavior of NG TNTZ has been investigated by potentio-dynamic scanning and electrochemical impedance spectroscopy in vitro conditions in this study.

1169 Molten mold flux technology for continuous casting of TWIP steel

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Molten mold flux technology so called, POCAST process has been studied at pilot plant CMM in Pohang POSCO. Steel grades have been selected mainly for ultra low carbon with C 0.06~0.1%, and hook formation and oscillation mark along with the consumption rate of molten mold flux are investigated to compare with conventional powder type mold flux casting process. TWIP(TWin Induced Plasticity) steel with C 0.6~0.7% Mn 15~18%, Al 1.5~2% is also tested to investigate the castability using POCAST process. As a result, continuous molten mold flux feeding makes possible to eliminate the formation of slag bear in the mold, enhancing the mold flux consumption rate to produce clean steel slab. Furthermore, molten mold flux behaviors affecting surface cracks on slabs are discussed.

1170 Crystallographic features of the states appearing in the multiferroic material $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ around $x = 0.25$

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Two simple-perovskite oxides, BiFeO_3 and LaFeO_3 , can form a mixed oxide system $\text{Bi}_{1-x}\text{La}_x\text{FeO}_3$ (BLFO). The former BiFeO_3 with the R3c structure is one of multiferroic material showing ferroelectric and antiferromagnetic properties, while only antiferromagnetic order was

reported in the latter LaFeO_3 with the Pnma symmetry. The point to note here is that, in addition to the R3c and Pnma states, both the Pnam state having the PbZrO_3 -related superstructure and the state characterizing an incommensurate modulation were proposed to exist around $x=0.20$. However, the detailed features of these states still remain obscure yet. We have examined the crystallographic features of BLFO samples with $0.20 \leq x \leq 0.30$, prepared by the solid state reaction method, mainly by transmission electron microscopy. Among the experimental data obtained in this study, for instance, it was found that there were two kinds of areas with an average size of about 200 nm in samples at room temperature around $x=0.25$. Electron diffraction patterns with various electron beam incidences, taken from these two areas, indicated that one area had the Imma structure with the R_{25} -type rotational displacements of oxygen octahedra about two $\langle 100 \rangle_c$ directions in terms of the pseudo-cubic notation. It was also found that a commensurate modulation was involved in the crystal structure of the other area. From the careful analysis of experimentally-obtained patterns, the modulated structure was understood to be characterized by the presence of two modulated waves with wave vectors of $q = [1/4 \ 1/4 \ 0]_c$ and $q = [0 \ 0 \ 1/4]_c$.

1171 Enhanced sintering densification of yttria ceramics by means of field-assisted and flash sintering techniques

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Y_2O_3 ceramics have special chemical and physical properties such as high resistance to halogen-plasma corrosion and thermal stability. Conventional sintering, however, requires very high temperatures typically $>1600^\circ\text{C}$ and a vacuum or hydrogen atmosphere because of the low sinterability. We have demonstrated that undoped Y_2O_3 can be sintered nearly instantaneously to almost full density by flash sintering, where densification occurs in a few seconds under a threshold condition of temperature and applied field (H. Yoshida, Y. Sakka, T. Yamamoto, J.-M. Lebrun, R. Raj, J. Eur. Ceram. Soc., 34 (2014) 991). The Y_2O_3 shows flash-sintering at the fields above 300 V/cm. For instance, full densification is achieved at 1133°C under a field of 500 V/cm. The flash sintering in Y_2O_3 is preceded by gradually accelerated field-assisted sintering (FAST). This hybrid behavior differs from earlier work on Y_2O_3 -stabilized ZrO_2 where all shrinkage occurred in the flash mode. The microstructure of flash sintered specimens indicated that densification was accompanied by rapid grain growth. High-resolution transmission electron microscopy (HRTEM) observation indicated that the flash sintered Y_2O_3 exhibited single-phase microstructure without amorphous layers even at grain boundaries and multiple junctions. It is postulated that densification and grain growth were enhanced by accelerated solid-state diffusion, resulting from both Joule heating and the generation of defects under the applied field.

1172 Influence of grain size on work-hardening behavior in 12Cr stainless steel

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Grain refinement is gathering attention as a strengthening method which does not depend on the alloying elements added to steels. There are many reports on manufacturing methods and mechanical properties of ultra-fine grained steels. In the ultra-fine grained steel, it is well known that yielding stress drastically increases in accordance with Hall – Petch relationship, while uniform elongation significantly decreases. These tendencies imply that the grain size affects not only yielding but also work-hardening behavior. However, the influence of grain size on work-hardening behavior has not been clearly understood. In this study, work-hardening behavior during tensile deformation in 12Cr stainless steel with various grain sizes was investigated. Grain refining was conducted by cold-rolling of the steel quenched after annealing, and the recrystallization annealing. Grain size decreased with the increase of cold-rolling reduction ratio, and the minimum grain size obtained by the method was approximately 5 micrometers. 0.2% proof stress increased with the decrease of grain size. On the other hand, the strain which reached the plastic instability condition decreased with the decrease of grain size. In the session, we report dislocation accumulation behavior estimated by grain hardness and XRD, and dynamic recovery behavior assessed by Kocks – Mecking model.

1173 Local structure investigation of in Mg-Zn-Gd alloys by XAFS

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Magnesium based alloys in Mg-Zn-Gd with long period stacking ordered (LPSO) structure have superior mechanical properties. The specific structure is formed by the aging procedure at high temperature (623 K <). Additionally, this system has many crystal structures and morphologies depending on aging temperatures and holding times. In this study, our final goal is to determine the local structure behaviors around Zn and Gd on precipitation process of LPSO phase from Mg₉₇Zn₁Gd₂ cast alloy. We adopt x-ray absorption fine structure (XAFS) method with using a micro-beam. Specimens are firstly scanned across an x-ray beam which has a diameter of several hundred nanometers. After that, XAFS are measured at selected positions in the elemental distribution images. The shapes of Zn K-edge x-ray absorption near edge structure (XANES) at the highly Zn concentrated regions are changed by aging. These XANES spectrum differences between as-cast and annealed samples suggest for the local structure changes of Zn from the as-casted sample.

1174 Evaluation of parameters effect on microstructure and mechanical properties in TIG welding of A105 to A106 steels

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Abstract. Welding parameters such as current intensity, voltage, number of passes can affect the mechanical properties of the weld. In this paper the effect of these parameters on structure and mechanical properties of welded A105 and A106 steels has been evaluated. According to the mechanical and microstructure test results, increasing in welding pass number causes reduction in grain size and increasing in average hardness of HAZ. Also inter-pass slag inclusion defect occurred in high number of passes.

1175 Quantitative evaluation of reheat cracking susceptibility by in-situ observation and measurement using laser confocal microscope

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In the post weld heat treatment (PWHT) process, the reheat cracking which might occur in the weldments of low alloy steels has been a serious problem. So, it is considered to be important to predict the possibility of occurrence of the reheat cracking in these steels. It is however recognized as time consuming procedure to evaluate quantitatively the susceptibility to this type of cracking. In the present study, a new quantitative evaluation method of reheat cracking susceptibility by in-situ observation and measurement using a laser confocal microscope has been proposed. Through this new method, the reheat cracking susceptibility of any kind of steels can be evaluated with the same standard. Moreover, because the position of initial crack can be focused and the critical ductility to initiate crack is measured by in-situ observation, the reheat cracking susceptibility can be evaluate by using only one specimen. Therefore, the newly developed method can provide efficient quantitative assessment of the reheat cracking sensitivity with high accuracy.

1176 Research and development of an antibacterial biodegradable Mg alloy for orthopedic applications

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Magnesium (Mg), a potential biodegradable material, has recently received increasing attention due to its unique antibacterial property. However, rapid corrosion in the physiological environment and potential toxicity limit clinical applications. In order to improve the corrosion resistance meanwhile not compromise the antibacterial activity, a novel Mg alloy, Mg-Nd-Zn-Zr (Hereafter, denoted as JDBM), is developed by alloying with neodymium (Nd), zinc (Zn),

zirconium (Zr). The JDBM showed high strength and ductility balance, and the pH value, Mg ion concentration, corrosion rate and electrochemical test show that the corrosion resistance of JDBM is enhanced in comparison with the commercial pure Mg. A systematic investigation of the in vitro and in vivo antibacterial capability of JDBM is performed. The results of microbiological counting, CLSM, SEM in vitro, and microbiological cultures, histopathology in vivo consistently show JDBM enhanced the antibacterial activity. In addition, the significantly improved cytocompatibility is observed from JDBM. The results suggest that JDBM effectively enhances the corrosion resistance, biocompatibility and antimicrobial properties of Mg by alloying with the proper amount of Zn, Zr and Nd, indicating a promising clinical prospect.

1177 Load partition and microstructural evolution during hot tensile tests of unreinforced and TiC particle reinforced in Ti-6Al-6V-2Sn

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Load partition in multiphase materials can be studied by means of in-situ high energy synchrotron diffraction (HEXD). An example of the capabilities of this technique was applied to an unreinforced and two particle reinforced Ti-6Al-6V-2Sn alloys with 12 vol.% and 20 vol.% of TiC particles, with higher E and better wear resistance than the matrix. This matrix contains larger amount of beta phase than Ti-6Al-4V and its service temperature is raised up to 315°C due to the addition of Sn. The alloys, produced by a powder metallurgy route, exhibit lamellar microstructure of the alpha phase with inhomogeneous distribution of the TiC particles for the composites. In-situ HEXD experiments were carried out during tensile tests at 750°C to determine the load partition between alpha, beta and TiC and their plastic deformation mechanisms. The reinforced alloys show higher ultimate strength than the matrix owing to the smaller size of the alpha lamellae. Nevertheless, the subsequent softening is more noticeable for the composites. The in-situ method allows following the rotation of the alpha phase and the increment of the misorientation in the beta phase within individual grains during the deformation. The small gauge volume of the synchrotron beam compared to the grain size allows studying regions with high particle concentration which are capable of bearing load, although in average the reinforcement is not efficient for strengthening owing to its non-homogeneous distribution. The stress concentration between particle clusters and debonding between matrix and particles during deformation are also studied.

1178 First-principles study on thermodynamic stability of Mg-based LPSO phases revisited from short-range order

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Mg-based ternary alloys can form so-called long-period stacking ordered (LPSO) structures, which is a candidate for light-weight structural materials in next generation, due to its high

tensile strength and ductility. In order to address thermodynamic stability of the Mg-based LPSO phases, we examine the short-range order (SRO) for competing metastable disordered phases on multiple stacking sequences using first-principles-based statistical thermodynamics calculations. To systematically and efficiently evaluate the SRO over possible combination of constituent elements, we employ lattice statistical information (LSI) approach recently developed by our research group, which enables quantitative estimation of SRO using information about merely a single special microscopic structure that is common for any combination of elements and is independent of temperature. We find that anisotropy in in-plane and inter-plane SRO along nearest-neighbor coordination shows different behavior among Mg-based alloys where L12-type cluster is observed, those where not observed, and those where LPSO phases themselves are not observed. These facts strongly indicate that tendency of SRO for competing metastable disordered phase can be one of the key role to systematically understand the LPSO phase stability. Other tendencies for SRO will also be discussed.

1179 Analysis of hydrogen solubility and diffusivity toward the design of V-based alloy membranes with high hydrogen permeability and strong resistance to hydrogen embrittlement

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Ultra high purity hydrogen can be produced efficiently with high hydrogen flux rate by utilizing hydrogen permeable metal membrane. Vanadium and its alloys are promising materials for advanced hydrogen permeable membrane to be substituted for currently used Pd-based alloys, e.g., Pd-Ag. On the other hand, a consistent description of hydrogen permeation through metal membrane based on hydrogen chemical potential has recently been proposed [1], and it is found that the hydrogen flux through the membrane is directly linked with the shape of the pressure-composition-isotherms (PCT curves). The hydrogen flux changes proportionally with the PCT factor. In this study, from a series of PCT measurements, the alloying effects on hydrogen solubility of vanadium have been investigated systematically. Also, the mechanical properties of vanadium membrane in hydrogen atmosphere at high temperature has been examined [2]. It is found that a ductile-to-brittle transition occurs drastically at the hydrogen concentration around 0.2 (H/M). Combined the results of these two experiments, V-based alloys with hydrogen permeability and strong resistance to hydrogen embrittlement have been designed and developed. In addition, the alloying effects on the mobility of hydrogen atom under hydrogen permeation have been analyzed applying the consistent description of hydrogen permeation. It is found that most of the alloying elements enhance hydrogen diffusivity at low temperature below about 700 K, except for V-Al system. [1] A.Suzuki et. al., Int'l J. Hydrogen Energy, 39 (2014) 7919. [2] Y.Matsumoto et. al., Proc. 2nd Int'l Conf. SSTT, (2012) 132.

1180 Prediction of carbide coarsening and its effect on the fretting wear behavior of an Inconel 690 SG tube for nuclear power plants

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Prediction of carbide coarsening and its effect on the fretting wear behavior of an Inconel 690 steam generator (SG) tube for nuclear power plants was investigated in this study. Based on a prior study, increasing carbide size and spacing increase the wear resistance of Inconel 690. Therefore, we reasoned that, if carbide coarsening were to occur within the operating temperature of an SG, the wear behavior of the material would change during operation. We set up a time-temperature equation to predict carbide size at various temperatures based on the Lifshitz-Slyozov-Wagner (LSW) coarsening model. The calculated activation energy in this study well fit the diffusion activation energy value for Cr. SEM observations and wear test results revealed that during the operation of SG carbide coarsening can occur and have an effect on the wear resistance of Inconel 690.

1181 Friction stir welding of a 5024 alloy subjected to cold rolling

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Effect of friction stir welding (FSW) on microstructure and mechanical properties of Al-4.57Mg-0.35Mn-0.2Sc-0.09Zr sheets was studied. FSW was carried out on sheets of AA5024 subjected to cold rolling with a total reduction of 70 pct. Following parameters of FSW were used: the rotation speed of 400 rpm, the traverse speed of 75 mm/min and the tilt angle of 2.5°. FSW leads to the formation of fully recrystallized microstructures with average grain sizes about 2 μm and a moderated dislocation density of $\sim 10^{13} \text{ m}^{-2}$ in the stir zone. No evidence of abnormal grain growth was found in the heat affected zone of the weld. The joint efficiency in the cold rolled condition for both yield strength and ultimate tensile was found to be 83 pct. and 82 pct., respectively. Relationships between microstructure and mechanical properties of in different zones of weld joints are discussed.

1182 A 3D DDD modelling and simulations of precipitate strengthening at high temperatures

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A behaviour of structural materials operating under high temperatures and multiaxial loading can be investigated by a 3D discrete dislocation dynamics (DDD) model. Our model describes the dislocation structure by short straight segments which simplifies calculation of stress field from dislocations. The total stress is given by a superposition of contributions from individual segments and an applied stress and determines the Peach-Koehler (P-K) force and the kinetics

of individual segments. The segments move by vacancy diffusion controlled dislocation climb and dislocation slip. A scaling factor is employed, which depends on the displacement mechanism and the orientation of the dislocation segment and P-K force relative to the crystal lattice. The model incorporates rigid incoherent spherical particles. The simulations run in two regimes: the particles either interact with the dislocations only by a geometrical condition (impenetrability), or the dislocations encounter reaction forces normal to interface. The dislocations may surpass the precipitates by combination of glide and climb, or by creating dislocation loops at the interface. A system consisting of a low-angle tilt boundary with misorientation angle about 1° and an array of spherical particles was treated. The migration of the dislocation boundary is driven by an external shear stress. The simulations were done for distinct dislocation densities and particle sizes. The results indicate that precipitate hardened alloys exhibit a threshold shear stress. The DDD method also allows calculating creep curves and estimate stress exponents. Depending on the dislocation density, the stress exponents may reach high values, even above 15.

1183 Multiphysics and multiscale modeling of solidification in casting processes

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Solidification of the molten metal during casting sets the basic crystalline microstructure of the material. During solidification several types of defects form, such as heterogeneities of chemical composition: microsegregation at the microstructure scale, macrosegregation at the scale of the product; heterogeneities of structures in terms of grain orientation, size and morphology; or microporosities. The reduction of these defects is an important issue for the industry because they impair the final properties of products. Process-scale models of solidification need to account for couplings of different physical phenomena across several length scales. This requires a multiscale description of crystal growth, of formation of dendritic growth morphologies, and of multiphase heat and mass transfer and fluid flow across several regimes of multiphase transport. Microscopic phenomena can be incorporated into macroscale models by various homogenization techniques; the volume averaging method is often used. This requires a careful simplification and averaging of the microscale phenomena and interactions. In our presentation we provide an overview of existing models and their fundamental principles. We present the most recent applications of these models to industrial solidification processes (casting of steel ingots, direct-chill casting of aluminium alloys). We demonstrate how they contribute to the understanding of complex phenomena, such as the competition between nucleation and growth of crystal grains in the presence of convection of the liquid and of grain motion, and we discuss their predictive capabilities. Finally, we address the key remaining questions for future research.

1184 The influence of nitrogen on the precipitation kinetics in microalloyed medium carbon steel

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Three melts with different N contents, 35 to 85 ppm wt%, are produced via vacuum induction melting and casting. Target of the present work is to investigate the influence of traces of N on the precipitation kinetics of Nb-Ti microalloyed, medium carbon steel during processing.

The material is investigated experimentally by means of light optical microscopy, scanning electron microscopy, electron microprobe analysis and for the fine scaled particles transmission electron microscopy is applied. Numerically, the precipitation kinetics is calculated with the thermokinetic software package MatCalc based on the time-temperature profile of the solidification and rolling process of the material and the final quench and tempering heat treatment. The inhomogeneous chemical composition and microstructure of the as-cast condition is considered via a Scheil-Gulliver Simulation to estimate the chemical composition of the steel after casting. This information is the starting condition for the diffusion simulation where the change of the microsegregation and the precipitation kinetics during further processing is investigated. The experimental findings and simulation results are in good agreement.

1185 Effects of deformation induced vacancies in SPD processed nanomaterials

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Since the introduction of methods of Severe Plastic Deformation (SPD) [1], non-equilibrium vacancies induced by plastic straining [2, 3] have become very important. Because of the high plastic strains as well as the high hydrostatic pressures applied in SPD processing [1], the vacancy concentrations can reach values till 10^{-3} [2, 3]. On the one hand, these can affect strongly the diffusion in SPD processed alloys thus achieving significant changes in the stability of phases [3]; on the other hand the vacancies can form agglomerates which act as barriers for the dislocation motion thus causing extra hardening by up to several tens of % [2, 5]. Models are presented which simulate the phase changes [4] as well as the hardening effects, and examples are given which suggest the two effects not only for mechanical but also for functional applications [5]. [1] M. Zehetbauer, H.P. Stuewe, A. Vorhauer, E. Schafner, J. Kohout, Adv.Eng.Mater. 5, 330-337 (2003) [2] M. Zehetbauer, Key Eng.Mater. 97-98, 287 (1994), Mater.Sci.Forum, 503-504, 57-64 (2006) [3] D. Setman, E. Schafner, E. Korznikova, M.J. Zehetbauer, Mater.Sci.Eng. A 493, 116-122 (2008); D. Setman, M. B. Kerber, H. Bahmanpour, J. Horky, R. Scattergood, C. C. Koch, M. J. Zehetbauer, Mech.Mater. 67, 59-64 (2013) [4] see, e.g., B. Straumal, A.R. Kilametov, Y. Ivanisenko, A.A. Mazilkin, O.A. Kogtenkova, L. Kurmanaeva, A. Korneva, P. Zieba, B. Baretzky, Int.J.Mater.Res. 106, 657-664 (2015) [5] R. Valiev, Y.Estrin, Z. Horita, T. Langdon, M. Zehetbauer, Y. Zhu, Mater.Res.Lett., sections 4.3 & 4.7, published online DOI: 10.1080/21663831.2015.1060543

1186 Characterization of phase transformations occurring in Ti-15Mo by in-situ methods

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Phase transformations occurring in metastable Ti-15Mo alloy were studied in-situ during linear heating by electrical resistance measurement, dilatometry and differential scanning calorimetry. The alloy was solution treated at temperature above beta-transus for 4 hours and then water quenched. After quenching the material consists of beta phase matrix and omega phase particles. The transitions occurring in the material during heating are omega and alpha phase precipitation and dissolution in beta phase matrix. The methods allowed to study effect of detected processes on physical properties of the material. Different heating rates were employed in order to determine kinetics of ongoing transitions. The evolution of the microstructure was observed on samples heated up to temperatures obtained from in-situ measurements by scanning and transmission electron microscopy.

1187 Deformation induced strong and stable nanolaminated structures in nickel

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Heavy plastic deformation can substantially refine the grain size of metals into submicron regime (0.1-1 μ m), leading to ultrafine grained (UFG) structures with high strength. However, further straining is unable to reduce the grain size, while the structural stability decreases with an increase of strength. How to break the restriction of saturation grain size and to simultaneously enhance strength and structural stability has been one of the difficult technical problems for nanometals. A polycrystalline Ni with a purity of 99.88 wt% was subjected to surface mechanical grinding treatment (SMGT). Nanometer-scale flat and straight lamellae, i.e. 2-dimensional nanolaminated (NL) structures, were fabricated in the surface layer of 10-80 μ m thick. The NL structure shows a boundary spacing about 20 nm that is one order's smaller than the UFG counterparts and mainly consists of low angle boundaries. The hardness of the NL structures is 6.4 GPa that is two times UFG Ni. More interestingly, the coarsening of the NL structure during post annealing takes place at a relatively lower rate and higher onset temperature of 506°C that is 40°C higher than that of the UFG Ni. The good combination of strength and stability breaks the traditional trade-off relationship between strength and thermal stability. The formation mechanism of such NL structure by plastic deformation with high strain, high strain rate and high strain gradient was analyzed. A new generation of nanostructure with high performance was proposed.

1188 Microstructure and property of Fe-based alloy modified layer on 304 stainless steel by high-energy pulse laser-like cladding(HPLC)

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Fe-based alloy modified layers were prepared on 304 stainless steels by high-energy pulse laser-like cold welding cladding technique. The microstructure, composition and phase constituents of the cladding layers were analyzed using SEM, EDS and XRD, respectively. The microhardness, friction-wear and cavitation erosion resistance were also investigated using microhardness tester, pin-on-disk wear-testing machine and ultrasonic vibrator. Experimental results showed that Fe-based alloy modified layer was mainly composed of α -Fe matrix phase and skeleton-like Cr_{23}C_6 , Cr_7C_3 carbide reinforced phase, which was dispersively distributed into a-Fe matrix. The microhardness and friction coefficients of Fe-based alloy modified layer were 600HV and 0.4, respectively, indicating an improved wear resistance. The weight loss rate and average erosion depth of the modified layer was 1/5 and 1/10 that of 304 stainless steel in 3.5% NaCl solution after 5-h cavitation erosion test, respectively. The erosion crater depth of the modified layer was uniform, indicating that the cavitation erosion resistance of the modified layer was much better than that of the 304 stainless steel.

1189 Effect of cooling rate and austenite grain size on Ar₃ in low alloy steels

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The growths of ferrite with different prior austenite grain sizes were simulated with DICTRA software under different cooling rates in Fe-C-M₁ and Fe-C-M₁-M₂ (M₁ and M₂ denote substitutional alloying elements) low alloy steels. The starting temperatures of proeutectoid ferrite transformation (Ar₃), defined as 1% transformation temperature, were compared with those obtained from published CCT diagrams. It is found that both the increasing of cooling rate and austenite grain size will decrease Ar₃ and the calculation results agree relatively well with the experimental Ar₃ under slow cooling rate. However, the delay of experimental Ar₃ becomes larger with the increase of cooling rate, which is considered to be the result of incubation time and nucleation. During incubation period, grain boundary diffusion of alloying element could occur in the nucleus.

1190 Annealing effect on ambient ductility of a high Nb containing TiAl alloy

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Refining the lamellar microstructures by thermal mechanical treatments has shown the most significant effect in improving the ductility in both $\gamma+\alpha_2$ two phase and $\gamma+\alpha_2+\beta$ multi-phase high-Nb containing TiAl alloys. Whereas, the parallel heat treatment methods for modifying cast microstructures are less effective for lack of the deformation stored energy. Recently, cast TiAl alloys containing higher Nb contents are developed for the applications at higher temperatures but suffered even increased difficulty in gaining moderate ductility. Based on the understanding that residual stresses arisen during solidification significantly influence the mechanical performance of cast materials, the stresses in the as-cast and annealed test bars of the newly developed high Nb containing cast TiAl alloys are measured using Engine-X at ISIS neutron source. Current experiments present a coincident result that the residual stresses in annealed specimens are significantly reduced and meanwhile the ductility after annealing improved from 0.5 lower up to 1.9%. Accordingly, the relief annealing at moderate temperatures is developed to mitigate the brittleness of the newly developed cast TiAl alloys.

1191 Observation of interactions between crystal defects by applying in situ nanoindentation in a TEM

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In recent years, in situ nanoindentation technique has been widely used to investigate the mechanical behavior and properties dominated by geometric and structural features at nanometer scale. By applying this technique in a TEM, some fundamental mechanisms with respect to the interactions between crystal defects can also be quantitatively investigated in real time. In this work, in situ TEM nanoindentation has been performed in bcc iron and fcc Al with sample sizes around several hundred nanometers. The interactions between grain boundaries and dislocations are analyzed and the results are compared between iron and Al to understand their different yield and flow behaviors.

1192 Revisiting the effect of solidification cooling rate on microstructure of cast magnesium alloys

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Microstructure of as-cast metals is governed by solidification cooling rate, chemical composition (solute additions), effective heterogeneous nucleation sites and associated external forces. It is generally considered that higher cooling rate results in finer and equiaxed grains due to the large undercooling. Considerable research in the last a few decades also indicates

that additions of solutes and/or proper nucleants can also promote the formation of fine and equiaxed grains. However, the combined effects of two or more these factors are more complicated. Significant grain coarsening and increase fraction of columnar structure were observed in some Mg binary alloys at high cooling rate in our recent work. In these alloys, fine and equiaxed grains were more easily obtained at slow cooling. In addition, it was also found that adding some solutes or particles into the binary Mg alloys may lead to chemical reaction before the solidification starts. The as-cast microstructure of Mg alloys is closely related to these reaction products. The possible mechanisms of grain coarsening and refining will be discussed in the presentation.

1193 Preparation and its application of high performance plasma electrolytic oxidation (PEO) and its compound coatings on magnesium alloy

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Plasma electrolytic oxidation (PEO), as a relatively new and effective surface treatment technique derived from the conventional anodic oxidation, has been widely investigated and used to form functional ceramic coatings on Mg alloys. In this paper, a new PEO coating (with higher PBR, 1.44), instead of MgO (PBR, 0.81), was formed on magnesium alloy in acidic KF solution with dihydric phosphate by controlling the bio-polar pulse voltage. Results show that the coating was uniform and compact without big fissures, provided with low porosity level, 4.8%; The presence of dominated KMgF_3 and MgF_2 phase was found in the outer layer, whereas, in the inner barrier layer, the co-existence of MgF_2 , $\text{Mg}_3(\text{PO}_4)_2$, MgHPO_4 and MgO species was observed. Compared with the bare metal, the corrosion resistance of new coating was increased by about five orders. Based on the PEO technology, some compound coatings have been prepared and forward magnesium alloy to application in industrial field. (1) plasma electrolytic oxidation (PEO) treatment plus a top coating with sealing agent using multi-immersion technique under low-pressure conditions (2) an electroless plating (EP) on an insulated protective film on magnesium alloy, which presents the advantages of PEO and EP for magnesium alloy AZ91D (3) the PEO/Nano self-assembly (SAM) metal ceramic coatings has been successfully prepared on magnesium alloy that provide multivariate properties for substrate, such as anti-wear, high hardness and high protective properties.

1194 Reversion during continuous heating in martensitic Fe-2Mn-1.5Si-0.3C alloy

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The reversely transformed austenite structure and alloying element partitioning behaviors during the reversion have great effects on the kinetics of subsequent austenite decomposition, thus, the final mechanical properties of steels. Two morphologies of austenite: acicular and globular were observed during the austenitization from lath martensite. However, the effects of heating rate on the reverse transformation behavior, such as, kinetics, alloying elements partitioning have not been well understood yet. In this study, the reverse transformation

behavior as a function of the heating rate were examined both experimentally and numerically. It was found that the globular and acicular austenite bear random and K-S orientation relationship with the tempered martensite, respectively. The partitioning behavior of Mn and Si is strongly depended on the morphology of austenite. High heating rate leads to high density of globular austenite and thinner acicular austenite. Both experimental and numerical results reveal that the degrees of Mn and Si partitioning between acicular austenite and tempered martensite decrease with the increase in heating rate.

1195 Effects of T4 heat treatment on residual stress in friction stir welding metal matrix composites: neutron diffraction and multiscale modeling

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Residual stress in the Friction stir welding (FSW) metal matrix composites (MMCs) significantly affects the weld performance. Therefore, it is important to tailor the residual stress. Post-welding treatment is a way to modify the residual stress and additionally to improve the mechanical properties. In this work, the effects of T4 heat treatment on the residual stress in the FSW MMC joints are studied via neutron diffraction and multiscale modeling. After the T4 heat treatment, the distribution range and magnitude of high longitudinal (L) total and macroscopic residual stresses decrease in both the matrix and reinforcement in the nugget zone and heat affected zone (HAZ); the distribution range and magnitude of high L elastic mismatch residual stress increase in the matrix in the nugget zone and HAZ, while they decrease in the reinforcement; the magnitudes of transversal (T) total, macroscopic and elastic mismatch residual stresses in both phases increase; the variation of the normal (Z) total, macroscopic and elastic mismatch residual stresses in both phases is small; the T and N elastic mismatch and the thermal misfit residual stresses have slight variations after the T4 heat treatment. For accurate multiscale modeling of macroscopic and microscopic residual stresses in the FSW MMC plate and the FSW + T4 plate, it is important to take into account the residual stress from previous T4 heat treatment of the parent plates. Besides, applying temperature history dependent constitutive models for both macroscale and microscale models can also improve the prediction accuracy significantly.

1196 High temperature optical spectroscopy characterizations of semiconductor materials

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1197 Microstructure and property of three-wire submerged arc welded joint of shipbuilding steel EH36

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Three wire submerged arc welding was applied to butt weld the industrially produced 32 mm thick shipbuilding steel plate, and microstructure and property of the weld joint was investigated. The weld metal contains mostly of acicular ferrite grains plus few grain boundary ferrites. The coarse grained heat affected zone contains 10~20 micrometer polygonal ferrite and 3~10 micrometer acicular ferrite, which takes up 30-40% in volume fraction. A microstructure of polygonal ferrite and pearlite was found in the fine grained heat affected zone, as compared to the base metal consisting of quazi-polygonal grains and bainite. The weld metal shows the highest hardness of 180~190 HV5 across the weld joint, while the lowest value of 150~170 HV5 falls in the HAZ, as compared to the base metal with a value of 170~180 HV5. The weld joint failed at base metal during transverse tensile test, and exhibits a tensile strength larger than 516 MPa. The HAZ shows an absorbed energy larger than 100 J during Charpy v-notch impact test at -20°C.

1198 Identification of pre-transformations of beta phase in metastable beta titanium alloy

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The characteristics of nucleation and growth of alpha phase in beta titanium alloys have been extensively investigated in the past several decades. However, the structure of supersaturated beta phase has not been well explored. In the present work, the striations of the beta phase in Ti-5553 alloys evidenced by TEM diffraction contrasts have been studied in depth by HRTEM. Results reveal that nano-scaled striations form in the beta matrix in the alloy solution treated followed by fast cooling. The microstructure consists of tweed nano-structure formed by the interweaving of two directional striations that have no clear boundaries. They are distributed homogeneously within almost all the beta matrix. Analyses reveal that these striations originate from the pre-transformations of beta to alpha and to omega. The transformed structures are between the cubic structure of the parent beta phase and the respective hexagonal structure of the alpha and the omega phase. The pre-transformations are realized by atomic displacements on each second {110} plane in <1-10> direction for alpha, and on each second and third {112} plane in opposite <11-1> directions for omega. These results are expected to provide new understanding on the structural nature of beta phase in Ti alloys.

1199 Exploring the interface-induced phenomena in thin film materials using advanced transmission electron microscopy

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Important properties of materials are strongly influenced or controlled by the presence of solid interfaces, i.e. from the atomic arrangement in a region which is a few atomic spacing wide. Characterizing the localized atom behaviors is critical to fully understanding the functional properties. The precise quantitative study on local atomic structure at and adjacent to the interface and induced relevant phenomenon is now made possible with the advent of the spherical aberration corrected transmission electron microscopy. Here, I will show that with quantitative measurements of atom column positions and theoretically calculations the intrinsic behavior of the localized atoms at or adjacent to the interfaces can be uncovered. Some examples will be presented. At the interface of VN-MgO (or metal-ceramic interface, i.e. Cu-MgO) the oscillations of interplanar spacing were observed, being confirmed by the theoretical calculations. In the second example, a diffuse interface with a composition-gradient and induced by N vacancies in CrN was also studied. Extensive spectra and structural analysis lead to some generalized conclusions. The relationship of the lattice constant and N vacancy concentration show a near linear under a certain condition. The relationship between the ionicity in CrN and nitrogen vacancy concentration is further revealed. A relation between the elastic deformation and electronic structure change in CrN is also experimentally derived.

1200 In-situ Atomic resolution Transmission Microscopy Study on mechanical property of Low Dimensional Materials under Strain Manipulation

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The microstructure of advanced materials at low dimension often exist abnormal mechanical and/or physical properties. For study on the property variation of nanowires and ultra thin films result from external strain introduced microstructure evolution, we developed an in-situ electron microscopy method that enables tensile and/or bending tests with simultaneous atomic-lattice resolution observations. We shall introduce some recent In-situ atomic resolution TEM work on mechanical property study of f.c.c and b.c.c metals as Au-, Cu-, Pt-, Ag-, and W-thin film, particles and nano-wires. The variation of mechanical properties reported here are raging from brittle-ductile transition, deformation mechanism transition from dislocation to nano-twins, theoretical strength approaching, and some other new phenomena which never appear in their bulk counterpart. We shall address on the size impact of deformation mechanism for metallic materials in nano-scale.

1201 Fracture and strength of bulk metallic glasses

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Based on the analysis of substantial experimental observations of fracture behaviors of metallic glasses and other high-strength materials, here we developed a new fracture criterion and proved it effective in predicting the critical fracture conditions under complex stress states. The new criterion is not only a unified criterion which unifies the three classic fracture criteria, i.e., the maximum normal stress criterion, the Tresca criterion and the Mohr-Coulomb criterion, but also a universal criterion which has the ability to describe the fracture mechanisms of a variety of different high-strength materials under various external loading conditions. Furthermore, in terms of the universal fracture criterion above, we show that the fracture of metallic glasses originates from the thermodynamic destabilization of the amorphous structure driven by imposed mechanical energy. The fracture behaviors and properties of metallic glasses can be predicted precisely and comprehensively just according to their elastic constants. This endows deep insights on the fracture nature of materials and makes it possible to compute fracture non-destructively without destroying the materials and free from a computer.

1202 Ion irradiation effects on nanocluster precipitation in steels

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Newly-developed precipitate-strengthened ferritic steels with and without pre-existing nanoscale precipitates were irradiated with 4 MeV protons to a dose of ~5 mdpa at 50 °C and subsequently examined by nanoindentation and atom probe tomography (APT). Irradiation-enhanced precipitation and coarsening of pre-existing nanoscale precipitates were observed. Proton irradiation induces the precipitation reaction and coarsening of pre-existing nanoscale precipitates, and these results are similar to a thermal aging process. The precipitation and coarsening of nanoscale precipitates are responsible for the changes in hardness. The observation of the radiation-induced softening is essentially due to the coarsening of the pre-existing Cu-rich nanoscale precipitates. The implication of the precipitation on the embrittlement of reactor-pressure-vessel steels after irradiation is discussed.

1203 Mg impact upon the generalized stacking fault energy of Al

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Both theoretically and experimentally, Mg is demonstrated to decrease the intrinsic stacking fault energy of Al, and is considered as a potential alloying element for the implication for fabricating nano twins in Al alloys. However, deformation twinning has been seldom reported in Al-Mg alloys even subjected to various kinds of severe plastic deformation techniques,

including ECAP (Equal Channel Angular Pressing), DPD (dynamic plastic deformation), HPT (High-Pressure Torsion) etc. A systematic first-principles study of the Mg solutes effect upon the generalized stacking fault energy of Al reveal that Mg alloying cannot efficaciously decrease the intrinsic stacking fault energy of Al, and is quite limited in promoting the twinning tendency of Al alloys. Calculations also indicate that Mg possesses a segregation tendency towards the stacking fault of Al, which is actually known as a Suzuki segregation effect. On basis of the predictions, the linear decrease in the intrinsic stacking fault energy along with increasing Mg content as reported in the literature is not observed, and noticeable increase in twinnability of Al originated from Mg alloying is not found, even at high Mg content. It is predicted that the local Mg content and local Mg arrangement has a strong influence on the intrinsic stacking fault energy as well as twinning propensity of Al.

1204 Solidification of Al-Pb alloy under the effect of micro-alloying element Ti and C

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Experiments were carried to investigate the effect of TiC on the solidification process and microstructure of Al-Pb alloys. It is demonstrated that TiC particles are effective inoculants for the nucleation of the Pb-rich droplets during cooling an Al-Pb alloy in the miscibility gap. A model describing the kinetic behavior of TiC particles in the melt and the liquid-liquid decomposition of Al-Pb were developed. The dissolution, coarsening and precipitation processes of TiC particles as well as the microstructure evolution during the liquid-liquid phase transformation of an Al-Pb alloy were calculated. The numerical results indicate that what determines the refinement efficiency of TiC particles on the Pb-rich droplets/particles is the number density of TiC particles in the melt cooled to the binodal line temperature of the Al-Pb alloy. If the number density of TiC particles in the melt before the beginning of the liquid-liquid decomposition is high enough, the addition of TiC causes a refinement of the Pb-rich droplets/particles and promotes the formation of Al-Pb alloys with a well dispersed microstructure.

1205 The Portevin–Le Chatelier effect and kinematics of deformation bands in an Al-Mg-Sc alloy: Effect of grain size

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Stress serration patterns and kinematics of deformation bands associated with the Portevin-Le Chatelier (PLC) effect were examined for an Al–6%Mg–0.35%Mn–0.2%Sc–0.08%Zr–0.07%Cr (in wt.%) alloy with two grain sizes: 22 μm and 0.9 μm . The fine-grained structure of the alloy was obtained using equal-channel angular pressing (ECAP) at 320°C up to a total strain of ~ 12 . Tensile tests were carried out at room temperature and strain rate ranging from $4 \times 10^{-5} \text{ s}^{-1}$ to $2 \times 10^{-2} \text{ s}^{-1}$. In addition, high-frequency local extensometry technique was applied to monitor the evolution of the axial strain distribution during deformation. Depending on the

strain rate, conventional A, B, C, or mixed types of serrations were observed on the stress-strain curves. These types of behavior usually correspond to different kinematics of the PLC bands, including band propagation and localization. However, the propagation regime was found to dominate in the investigated alloy in the entire strain-rate range. This unusual behavior of deformation bands and their features depending on the grain size are discussed.

1206 Cellular automaton modeling of ferrite growth in ternary Fe-C-Mn alloys

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Using the mixed-mode phase transition concept, a modified 2D cellular automaton model is developed to study the austenite-to-ferrite transformation kinetics in ternary Fe-C-Mn alloys. In contrast to the diffusion-controlled phase transformation models, the mixed-mode nature of the transformation model takes the effect of the finite interface mobility into account during the ferrite growth. A new treatment for ferrite growth proposed by Hutchinson et al (Metall Mater Trans A.2004;35A:1211) that allows for the local transport of Mn atoms across the reaction interface is included in this model. Thus, both the fast initial ferrite growth and the transition to much sluggish kinetics that were observed in many ternary Fe-C-X alloys can be accurately described. This CA model is also allowed effect of the annealing temperature and different Mn alloying content on the transformation kinetics and solute variation to be studied.

1207 The effect of process parameters of a novel interdendritic-melt solidification control technique on the microstructure and properties of a Ni-base superalloy

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A novel technique called interdendritic-melt solidified control (IMSC) was developed to manufacture equiaxed superalloy castings in the efforts of reducing porosity. The basic idea of IMSC is to preheat and maintain the shell mold at a specific temperature in the liquidus and solidus range. At this temperature, the dendritic skeletons have been built and the volume fraction of residual liquid remained at definite level so that the melt in the interdendritic zone could connect by capillary network. Once the molten alloy is poured into the preheated shell mold, the mold is withdrawn downward with a certain speed out of the heating zone and cooled to room temperature to ensure that the shrinkage is fed by the melt in interdendritic channel and an equiaxed structure can be built. Currently, the effect of parameter, such as withdraw rate and shell mold temperature, of the IMSC on the porosities and mechanical properties of a Ni-base superalloy was investigated compared with conventional investment casting (CC) technique. The IMSC and CC samples were characterized by quantitative metallography. In addition, the minor phases, such as MC carbides (~1vol.%), γ' phases and trace amount of η phase, were identified by synchrotron X-ray diffraction at Diamond Light Source (UK). The results indicate

that proper withdraw rate for IMSC can produce castings with much reduced porosity and equivalent mechanical properties compared to conventional investment casting. However, fast withdraw rate will produce casting with much severe porosity, more brittle phase η phase and worse properties.

1208 Investigations on hot tearing susceptibility and its mechanism of Mg-Zn-Y alloys

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Hot tearing sensitivity of $\text{MgZn}_{2.5}\text{Y}_x\text{Zr}_{0.5}$ ($x=0.5, 1, 2, 4, 6$) alloys were predicted by using thermodynamic calculations based on Clyne-Avrami's model, and verified experimentally by using a "T"-type hot tearing permanent-mold equipment. The hot tearing mechanism with different Y content of $\text{MgZn}_{2.5}\text{Y}_x\text{Zr}_{0.5}$ alloys was investigated. The results demonstrated that the predicted values are consistent with that of measured values. The hot tearing susceptibility of the alloys from high to low are: $\text{MgZn}_{2.5}\text{Y}_2\text{Zr}_{0.5} > \text{MgZn}_{2.5}\text{Y}_{0.5}\text{Zr}_{0.5} > \text{MgZn}_{2.5}\text{Y}_4\text{Zr}_{0.5} > \text{MgZn}_{2.5}\text{Y}_1\text{Zr}_{0.5} > \text{MgZn}_{2.5}\text{Y}_6\text{Zr}_{0.5}$. Among the studied alloys, the hot tearing sensitive of the alloys of $\text{MgZn}_{2.5}\text{Y}_1\text{Zr}_{0.5}$ and $\text{MgZn}_{2.5}\text{Y}_6\text{Zr}_{0.5}$ is less than that of the commercial alloy AZ91.

1209 Microstructure evolution in titanium alloys during large deformation in a wide temperature interval

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The effect of temperature on the mechanical behavior and mechanisms of microstructure formation in titanium alloys during large deformation was studied in the present work. In case of single-phase commercial pure titanium (CP Ti) three temperature intervals, each of which was associated with different mechanisms of microstructure formation, were revealed in the interval $-196 \dots 850^\circ\text{C}$. The microstructure evolution of titanium during the hot working was characterized by the development of discontinuous dynamic recrystallization consisting of nucleation and growth of the new grains. In the interval of warm working conditions, the microstructure evolution of CP Ti was associated with twinning following by continuous dynamic recrystallization, when the new grains result from gradual transformation of strain-induced subboundaries into high-angle grain boundaries. In the cold working interval four consecutive stages of CP Ti microstructure evolution developed, including: twinning, increase in dislocation density and the formation of substructure, formation of deformation-induced high-angle boundaries and finally appearance of new grains. In two-phase titanium alloys the microstructure evolution during warm and hot deformation was usually associated with transformation of a lamellar microstructure into globular one via the classical boundary splitting mechanism followed by further spheroidization of a particles by means of termination migration. Decrease in deformation temperature increased the contribution of shear deformation into formation of intraphase boundaries. The processes of microstructure evolution in titanium alloys were quantified and discussed in terms of kinetics and controlling mechanisms.

1210 Needle like Fe-Containing Intermetallic Compounds of High Silicon Aluminum Alloy with Fe Modified by Mn and Ultrasonic Vibration

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In this study, the modification effects and mechanism of manganese (Mn) and ultrasonic vibration (USV) on the needle-like Fe-containing intermetallic compounds of Al-20Si-xFe-2.0Cu-0.4Mg-1.0Ni (x=1, 2 wt.%) alloy have been studied respectively. The effect of Fe-containing phases on volume fraction of hard phases is also investigated. The results show that the mechanism and effect of Fe-containing intermetallic compounds improved by Mn are in close relationship with Fe content. Mn can promote to form less harmful α -Al₁₅(Fe,Mn)₃Si₂ phase, or replace some Fe atoms of β -Al₅FeSi and δ -Al₄FeSi₂ according to different Fe content. When USV was applied to this alloy containing 2%Fe near liquidus temperature, most of the acicular β phases formed in traditional process are substituted by fine plate δ phases. With the combined effects of 0.5%Mn and USV, the acicular β phases are almost repressed and the Fe-containing phases exist in form of fine Al₄(Fe,Mn)Si₂ and Al₅(Fe,Mn)Si particles about 20~30 μ m. Consequently, the total volume fraction of hard phases which are composed of primary silicon particles and Fe- containing phases increases significantly.

1211 Development of low expansion coatings of reactive element modified Ni+CrN+AlN nanocomposite for high temperature protection

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The mechanical degradation mechanism of high temperature coatings is the cracking and spallation of thermally grown oxide (TGO) scales during cooling stages, as well as erosion of TGO scales by foreign particles. The cracking and spallation of TGO scales results from the thermal stresses generated during cooling stages, which is associated with the large mismatch between coefficients of thermal expansion (CTE) of the TGO scales and the underlying coatings. A low expansion coating of reactive element modified Ni+CrN+AlN nanocomposite for substrates of superalloy and stainless steels was developed in order to the resistance of TGO scales from cracking and spallation. The coating was prepared by either reactive vacuum arc evaporation or reactive magnetron sputtering from a single reactive element modified NiCrAl alloy target in gas mixture of argon and nitrogen. Cyclic oxidation resistance of the nanocomposite coating was demonstrated much better than the NiCrAlYSi coating at 1000 °C. A thin AlN interlayer was found to be effective for preventing the inward diffusion of N from the coating into the substrate and beneficial for improving the performance of the coating at high temperatures. Based on such a nanocomposite coating, a multilayer coating of Ni+CrN+AlN/AlN was prepared and tested at higher temperature of 1100 °C. The mechanisms of oxidation and microstructural evolution of the coatings are discussed in this talk.

1212 Direct mapping of a periodic array of flux-closure quadrants in strain-mediated ferroelectric PbTiO₃ films

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Nanoscale ferroelectrics are expected to exhibit various exotic domain configurations, such as the full flux-closure pattern that is well known in ferromagnetic materials. Here we observe not only the atomic morphology of the flux-closure quadrant but also a periodic array of flux closures in ferroelectric PbTiO₃ films, mediated by tensile strain on a GdScO₃ substrate. Using aberration-corrected scanning transmission electron microscopy, we directly visualize an alternating array of clockwise and counterclockwise flux closures, whose periodicity depends on the PbTiO₃ film thickness. In the vicinity of the core, the strain is sufficient to rupture the lattice, with strain gradients up to 10⁹ per meter. Engineering strain at the nanoscale may facilitate the development of nanoscale ferroelectric devices.

1213 X-ray diffraction in nano-objects: effect of electron density modulation in the surrounding media

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Modulation of electron density due to atomic interactions in close proximity to interfaces between two media is well established phenomenon, which has been investigated by advanced electron microscopy and grazing incidence X-ray diffraction. May be most known is the electron density modulation on a monolayer scale in a liquid near its interface with a solid crystalline body. In this case, diffraction signal from modulated layer is enhanced with respect to that from liquid layer, in which molecules are not ordered. Interesting situation arises when characteristic crystal size is comparable with the modulation length in the surrounding, so substantial modification of total diffraction signal from entire structure can be expected. We have uncovered this problem when studying the structure of biogenic alpha-chitin crystals being few nm in size. Measurements were carried out in pristine chitin crystals stabilized by proteins and water, as well as upon deproteinization and dehydration. We found that this processing causes substantial shift of the (020) diffraction peak, while preserving angular positions of other diffraction peaks practically unchanged. These experimental findings were attributed to the modulation of the surrounding protein and water electron density along the b-direction of the chitin unit cell. Using the developed analytical diffraction model, we were able to fit the modified experimental diffraction profiles and to extract the protein and water modulation lengths, as well as their averaged fluctuations over the variety of chitin crystals. The extracted modulation periods can be used for characterizing the protein/chitin and water/chitin interaction lengths.

1214 Microstructure and mechanical properties of medium manganese steel plate with high strength and toughness

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An intercritical annealing process was applied to a medium manganese steel plate (Fe-0.01C-5.3Mn-1.53Si) after the thermo-mechanical controlled processing (TMCP) and ultrafast cooling (UFC). The microstructures were observed by scanning electron microscopy (SEM) equipped with electron backscatter diffraction (EBSD), electron probe micro-analyzer (EPMA) and transmission electron microscopy (TEM). The retained austenite was measured by XRD and mechanical properties were measured by uniaxial tensile and impact tests. The influence of different annealing temperature was compared and the relationship between microstructures and mechanical properties was investigated. Results showed that the microstructures of the medium manganese steel plate were characterized by ultrafine grained lath-like ferrite and retained austenite and the excellent mechanical properties could be obtained at the annealing temperature of 640°C for 5 h. The volume fraction of the retained austenite reached up to 21%, which could significantly increase the elongation compared with the traditional steel plate. The mechanical property results revealed that the steel possessed adequate ultimate tensile strength of 865MPa and excellent impact energy of 121J (-20°C). The outstanding combination of strength and toughness indicates that the steel has a bright application prospect.

1215 Influence of second phases on the superplasticity of Mg-TM-Y-CeMM alloys containing LPSO-phases

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The effect of the nature of the second phases in extruded Mg-TM-Y-CeMM (TM refers to a transition metal) alloys reinforced by intermetallic MgRE (RE refers to a rare earth element) compounds and LPSO-phase on their superplasticity has been evaluated between 300 and 400°C at the strain rate of 10^{-4} s^{-1} . The data have been compared with those of alloys containing a similar volume fraction of the LPSO-phase. The results evidence that no superplasticity below 350°C was found in the alloys containing exclusively the LPSO-phase while the alloys containing both MgRE compounds and LPSO-phase deform superplastically by grain boundary sliding from 300°C. These differences are related to the different behaviour of MgRE compounds and LPSO-phase in the course of superplastic regime. MgRE compounds assist to accommodate the deformation more easily than LPSO-phase, reducing tendency to develop cavities and extending the time for the occurrence of necking. The size and volume fraction of the respective phases are critical in order to promote enhanced superplastic behaviour. Maximum elongations are attained in the alloys combining similar volume fractions of MgRE compounds and LPSO-phase in which their size is reduced to the maximum. An increase in the particle size of the second phases, especially in the case of the LPSO-phase, hinders the grain boundary sliding mechanism in the alloys.

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