## 2010 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Electrical Properties of Nanomaterials

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee

*Program Organizers:* David Stollberg, Georgia Tech Research Institute; Nitin Chopra, University of Alabama; Jiyoung Kim, University of Texas - Dallas; Seong Jin Koh, University of Texas at Arlington; Navin Manjooran, Siemens Corporation; Ben Poquette, Keystone Materials; Jud Ready, Georgia Tech

Monday AM Room: 214
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Jud Ready, Georgia Tech; Seung H. Kang, Qualcomm Inc

**8:30 AM Introductory Comments**

**8:35 AM Invited**

**Schottky Diodes on Nanowires of Cadmium Telluride and Copper Indium Diselenide Embedded in Porous Alumina Templates**: *Vijay Singh*[1]; [1]University of Kentucky

Nanowires of copper indium diselenide (CuInSe2 or CIS) and cadmium telluride (CdTe) are of interest in solar cells of relatively high efficiency that can be produced inexpensively. These nonowires were electrodeposited inside porous alumina templates and characteristics of Al/CIS and Au/CdTe nanowire Schottky diodes were studied. The X-ray diffraction of the nanowires showed nanocrystalline cubic phase structures with the grain size equal to the diameter of the pores. The EDX analysis confirmed that both a and ß-phases of CdTe were present. For CIS, analysis of the current-voltage characteristics of these devices yielded diode ideality factor and reverse saturate current density values similar to those reported in the literature for bulk CIS/Al junctions. For CdTe, Analysis of Schottky diodes yielded a diode ideality factor of 10 in the dark and under "one sun" illumination. Reverse saturation current density was 34.9µA/cm2 in the dark and 39.7µA/cm2 under illumination.

**8:55 AM**

**Electronic Transfer in Molecular Nanostructures**: *Karel Kral*[1]; [1]Inst. Phys. ASCR, v.v.i.

The transfer of electrons or holes between states localized on individual molecules is considered theoretically. The mechanism considered can apply to molecular crystals, polymer solids, DNA molecule, etc. The electronic transfer is studied within a model with inter-molecular electron tunelling accompanied by the coupling of charge carriers to intra-molecular vibrations. The multiple electronic scattering on phonons is taken into account in the self-consistent Born approximation to the nonequilibrium Green's function self-energy. The corresponding kinetic equation shows the diffusion mechanism of electrons along the molecular solid. This mechanism will be compared to the well-known Marcus theory of electronic transfer.Using the above mechanism we shall discuss earlier experimental data on photoconductivity in charge-transfer crystals, recent data on electric conduction of DNA molecules and also the electric conduction in polymers.

**9:15 AM**

**Effect of the Nanocrystalline Structure of Metallic Cathodes on the Efficiency of Ion-Induced Electron Emission**: *Radik Mulyukov*[1]; Rinat Khisamov[1]; Konstantin Nazarov[1]; Yulai Yumaguzin[2]; Ayrat Nazarov[1]; [1]Institute for Metals Superplasticity Problems, Russian Academy of Sciences; [2]Bashkiria State University

Cold cathodes which are based on ion-induced electron or secondary electron emission are widely used in vacuum and discharge devices, optical quantum generators, displays, ion sources etc. Lowering their work function is an important fundamental task. Traditionally it is solved by a variation of the chemical composition of cathodes, coating, and giving a special shape for the improvement of emission characteristics. Recent investigations have shown that the work function of metals can be lowered by their nanostructuring. On an example of pure nickel and Al-6%Mg alloy it will be shown that nanostructuring results in a decrease of the work function by 0.4 to 0.8 eV and enhancement of the ion-induced electron emission current by 30% and more. This allows an increase of the efficiency of ion sources by using nanocrystalline materials as cold cathodes. Prospects of application of nanocrystalline materials for manufacturing highly effective cold cathodes will be outlined.

**9:35 AM**

**Electrical Conductance of Single TiO2 Nanotube Devices**: *Jie Huang*[1]; Dongkyu Cha[1]; Mingun Lee[1]; Moon Kim[1]; Jiyoung Kim[1]; [1]University of Texas at Dallas

Titanium dioxide (TiO2) nanotubes attract increasing attention because of their wide range of potential applications: photocatalysts, gas sensors, solar cells and bio-sensors, etc. In our research, TiO2 nanotubes were fabricated using atomic layer deposition (ALD) combining with an anodic aluminum oxide (AAO) template technique. Materials characterization was done by both XRD and TEM. Single nanotube devices were made by focus ion beam (FIB) as well as e-beam lithography. As fabricated TiO2 nanotubes with different wall thicknesses were annealed either in vacuum or O2 with various temperatures in order to control the oxygen vacancy doping rate. Conductivity was measured by both four-probe measurements and in-situ characterization in TEM. In this presentation, we will also propose a potential conduction mechanism. This research was supported by a grant (code #: 2009K000469) from 'Center for Nanostructured Materials Technology' under '21st Century Frontier R&D Programs' of the Ministry of Education, Science and Technology, Korea.

**9:55 AM**

**Enhancing Electrical Performance of Solution Processed Doped Zno Film by Nanowire Alignment**: *Sujay Phadke*[1]; Jung-Yong Lee[1]; Jack West[2]; Alberto Salleo[1]; [1]Stanford University; [2]Stanford University/Sequoia High School

ZnO is a wide band gap semiconductor with potential to replace ITO as transparent electrode material. Low cost ZnO thin films can be fabricated by depositing ZnO nanowires synthesized using low temperature solution based processes. To improve the conductivity of intrinsic ZnO, it is n-doped with Gallium atoms during synthesis. In order to study the electrical properties of ZnO nanowire films, the effect of directional alignment of doped ZnO nanowires on film sheet resistance was studied. Nanowires are aligned by shearing a nanowire dispersion between two silicon wafers. Directional sheet resistances of ZnO films have been measured with a four point probe. The sheet resistance observed in the direction of alignment is lower than that observed perpendicular to that. Computer simulations used in conjunction with sheet resistance measurements on aligned films are used to quantitatively determine the effect of inter-wire contact on conductivity of ZnO nanowire mat films.

**10:15 AM Break**

**10:25 AM**

**3D Carbon Nanotube Based Photovoltaic Devices**: *Jack Flicker*[1]; Jud Ready[2]; [1]Georgia Institute of Technology; [2]Georgia Tech Research Institute

Although photovoltaic technology has been around for over fifty years, the use of a nanostructured, three dimensional morphology in these types of devices has occurred only relatively recently. We introduce a three dimensional photovoltaic device with carbon nanotube pillars coated with photoactive materials to create a solar cell. The extra dimensionality of this cell added by the nanotubes has been theorized to increase the relative energy generated over planar cells by up to four times. The energy increase is due to an increase in the interactions between photons and the photoactive material as the sun is at an off normal angle to the cell substrate. Prototypes of these cells have been made and, although suffering from a low overall efficiency, do show an increased energy production in the same manner that theory predicts when the light source is at an off normal angle.

**10:45 AM**

**CuO Nanowire-Co3O4 Nanoparticle Heterostructures for the Development of Multi-Functional Photocatalyst**: Wenwu Shi[1]; *Nitin Chopra*[1]; [1]The University of Alabama

Heterostructures comprising of nanowires and nanoparticles with controlled compositions are critical for the development of future devices. In this regard, nanostructured CuO and Co3O4 are significantly important because of their semiconducting and catalytic properties. Here, we report a simple method to synthesize CuO nanowire-Co3O4 nanoparticle heterostructures and study these novel materials as photocatalysts. Our synthetic approach couples chemical vapor deposition (CVD) growth of CuO nanowires (diameters ~78.3±17.9 nm) with wet-chemistry method to result narrow size distribution Co3O4

nanoparticles (diameter ~7.0±1.5 nm) uniformly decorated on CuO nanowires. A series of characterization steps (SEM, HRTEM, XRD and EDX) are undertaken to observe size, morphology, phases, and composition of heterostructures. The absorption spectrum of as-prepared materials is collected by UV-vis-NIR spectrometer. It has been found that after nanoparticles decoration, absorption in visible region enhanced greatly. Experiments of using them as photocatalyst under visible light show photocatalytic properties have been improved by ~4.5 times.

**11:05 AM**

**CNT Based Thermoelectric Devices for Energy Harvesting**: *David Lashmore*[1]; Tom VanVechten[1]; Jennifer Mann[1]; Cory Timoney[1]; Ian Wilson[1]; [1]Nanocomp

Carbon nanotube sheets can be doped to create n and p type regions with Seebeck coefficients from +60 µV/k for p type and -50 µV/k for n type. Devices fabricated from these sheets will be described along with optimization of thermoelectric performance along with ZT, Watts/gram, Watts/m2 and Dollars per Watt. Two parameters limiting thermoelectric behavior are electrical conductivity, which can be made very high, ~ 2x 106 S/m, and the thermal conductivity which can be made very as low as 10 W/m oK. Thermal conductivity and electrical conductivity can be independently controlled. Direct solar conversion to electricity, a kind of thermoelectric solar cell, will be described. Carbon nanotube material acts much like a black body from near UV to almost 12 microns so that 98% of the solar radiation can very efficiently be converted to heat.

**11:25 AM**

**Synthesis of Tin Filled Carbon Nanotubes and Their Application to Lithium Ion Batteries**: Raj Das Gupta[1]; Carsten Schwandt[1]; *Derek Fray*[1]; [1]University of Cambridge

There is a need to increase the charge capacity of the anodes in lithium ion batteries and this can be achieved by using tin, aluminium or silicon particles but the particles decrepitate, due to the large volume changes on charging and discharging. In order to overcome this problem, carbon nanotubes were filled with tin by placing graphite electrodes into a bath of molten lithium chloride – tin chloride and applying a slow alterating current. It was found that the yield of carbon nanotubes was high and the majority were filled with tin. Anodes were made from this material and it was found that the charge capacity levelled off at 430 mAh/g after about 400 charge-discharge cycles at which point it became constant. TEM examination of the used anode showed that the nanotubes had expanded due to the volume changes of the tin but still encapsulated the tin particles.

**11:45 AM**

**Radar Absorption Properties of Radar Absorbing Structures Composite Filling with Carbon Nanotubes**: *Zhengquan Zhang*[1]; Tiehu Li[1]; [1]Northwestern Polytechnical University

The developments of evading radar detection have become increasingly more important. By reducing the radar cross section,they could evade radar detection. Radar absorbing structures are the structure that has both the function of load bearing and the EM energy absorbing capability. The construction of polymeric composite RAS could be achieved by regulating the electric property or magnetic property of material. Nowadays studies on investigating RAS using fiber reinforced polymeric composite materials are becoming popular research field.In the study, Radar absorbing structures laminate composites composed of glass fibers, carbon fibers and epoxy resin filled with carbon nanotubes, were fabricated in a press mould. The absorbing properties were simulated based on electromagnetic theory. Two optimal double-layered radar absorbing structure and one three-layered radar absorbing structure was obtained. Reflection loss of a three-layered radar absorbing structure was less -10dB in whole frequency range of 8.2-12.4GHZ and has double absorbing peaks.

**12:05 PM**

**Study of the AC Conductivity Affected by Crystallization and CNFs for PVDF/CNF Composite Thin Films**: *Lili Sun*[1]; Bin Li[1]; Weihong Zhong[1]; Yan Zhao[2]; [1]Washington State University; [2]Beihang University

Poly (vinylidene fluoride) (PVDF) is an important ferroelectric polymer with complicated phase behaviors and the polar functional group, -C-F-. In this paper, carbon nanofiber (CNFs) filled PVDF thin films were prepared via a solution casting method. The effects of CNFs on the crystallization of PVDF and the AC conductivity of CNF/PVDF nanocomposites were investigated. The results revealed that the introduction of CNFs not only affected the conductivity directly, but also caused indirect effects to the conductivity through influencing the size of crystal grains, the degree of crystallinity and the crystal phase transformation of PVDF. In particular, a phase transformation from a-phase to ß-phase in PVDF was found to affect conductivity of the nanocomposites due to the increment of CNF concentration in the nanocomposites.

**12:25 PM Concluding Comments**

## Advances in Composite, Cellular and Natural Materials: Natural Materials and Polymer Matrix Composites

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee

*Program Organizers:* Yuyuan Zhao, The University of Liverpool; David Dunand, Northwestern University

Monday AM Room: 305
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Lorna Gibson, Massachusetts Institute of Technology; Amy Johnson, University of Illinois at Urbana-Champaign

**8:30 AM**

**Alpha-helical Protein Networks Are Self-Protective and Flaw-Tolerant**: *Markus Buehler*[1]; [1]Massachusetts Institute of Technology

Alpha-helix based protein networks as they appear in intermediate filaments in the cell's cytoskeleton and the nuclear membrane robustly withstand large deformation of up to several hundred percent strain, despite the presence of structural imperfections or flaws. Here we report a series of molecular simulations with a coarse-grained multi-scale model of alpha-helical protein domains, explaining the structural and mechanistic basis for this observed behavior. We find that the characteristic properties of alpha-helix based protein networks are due to the particular nanomechanical properties of their protein constituents, enabling the formation of large dissipative yield regions around structural flaws, effectively protecting the protein network against catastrophic failure. We show that the key for these self-protecting properties is a geometric transformation of the crack shape that significantly reduces the stress concentration at corners. Our findings help to explain the ability of cells to undergo large deformation without failure while providing significant mechanical resistance.

**8:50 AM**

**Mechanical Behavior of Natural Sisal Fibers**: Flavio Silva[1]; *Nik Chawla*[1]; Romildo Toledo Filho[2]; [1]Arizona State University, School of Mechanical, Aerospace, Chemical, and Materials Engineering; [2]Civil Engineering Department, COPPE, Universidade Federal do Rio de Janeiro

Environmental awareness and an increasing concern with the greenhouse effect have resulted in the development of sustainable materials in construction, automotive, and packing industries. Natural fibers are a good alternative since they are ready available in fibrous form and can be extracted from plant leaves at a very low costs. They are also biodegradable materials. In this work we present a systematic experimental investigation on the mechanical performance of sisal fibers. Tensile tests were performed at four different gage lengths. The measured Young's modulus was corrected for machine compliance. Weibull statistics were used to quantify the degree of variability in fiber strength, at the different gage lengths. Fatigue tensile tests were performed at stress-levels between 80 to 400 MPa. The failure mechanisms will be described and discussed in terms of the fiber microstructure as well as the defect population in the fibers.

**9:10 AM**

**Development of Eco-Friendly Brake Friction Composites**: *Yafei Lu*[1]; Baoting Suo[1]; Hui Wang[1]; Yimei Lu[1]; [1]Beijing University of Chemical Technology

Eco-friendly brake friction composites with good friction performance were developed. The raw materials utilized were selected according to eco-friendly criterion that natural products should be preferably chosen. The formulations are composed of plant flax, mineral basalt, and wollastonite fibers as reinforcements, natural graphite as solid lubricant, zircon as abrasive, vermiculite and baryte as

functional and space fillers, and cardanol based benzoxazine toughen phenolic resin as binder. To enhance the heat resistance of flax fiber, chemical and physical methods including drying, room temperature alkaline solution, acid steam, high temperature alkaline solution were performed and after the treatments, micro-fibrils were formed. A new cardanol based benzoxazine was synthesized by the reactions among cardanol, aniline, and formaldehyde. The effects of the content of treated flax fiber and friction temperature on friction performance, friction coefficient and specific wear rate, of the composites were evaluated.

**9:30 AM**

**Energy Attenuation Capability of Woven Natural Silk (WNS)/Epoxy Composites Plates Subjected to Drop-Weight Impacts**: *Albert Uchenna Ude*[1]; Che Husna Azhari[1]; Kamauzzaman Sopian[1]; [1]The National University of Malaysia (UKM)

The impact energy attenuation and damage characterisation of woven natural silk (WNS)/epoxy laminated composite plates were evaluated. The samples which were prepared in configurations of sandwich WNS/epoxy/honeycomb, WNS/epoxy/coremat WNS/epoxy/foam and reinforced WNS/Epoxy laminate plates were subjected to low velocity impact loading at energy level of 32J, 48J and 64J respectively. Impact parameters like load–deflection, load–time and absorbed energy–time behaviour were measured for evaluating the impact performance in terms of load bearing capabilities, energy absorption and failure modes. Evaluations of the results showed WNS/epoxy/coremat as possessing better load bearing capability qualities. In general energy absorption decreases as impact energy increases in all the composite samples; WNS/epoxy/foam was seen as better energy absorber. Damage areas increases as impact energy increases while time decreases with increase in impact energy in all the configurations. SEM micrographs show mode of failure as matrix crack, delamination and fibre brakeage.

**9:50 AM**

**Strain Rate Effects on the Deformation Behavior of Particles in Epoxy-Based Composites**: *Bradley White*[1]; Jonathan Spowart[2]; Jennifer Jordan[3]; Naresh Thadhani[1]; [1]Georgia Institute of Technology; [2]AFRL/RXLMD; [3]AFRL/RWME

The micromechanical mechanisms controlling stress transfer between particles and matrix in a composite are fairly well understood. However, the influence of microstructural details on the amount of local stress/strain the individual particles experience and the strain rate dependence of the stress transfer are not well understood. In order to determine the bulk material-particle deformation relationship for particulate composites, particle strain measurements were carried out on cast epoxy-based materials containing metal particles that were subjected to compressive loads across a large range of strain rates ($10^{-4}$ - $10^{4}$). These composite materials varied by particle loading fraction (10-50 Vol%), size (5-50 \956m), and type (Al or Al+Ni) to produce microstructures with varied distributions of particle reinforcement. Individual particle deformations were evaluated, and correlated with quantitative characteristics of the distribution of reinforcement phase(s) within the matrix. In this presentation the approach as well as current findings and results will be discussed.

**10:10 AM Break**

**10:30 AM**

**Reactive Polymeric Nanocomposites**: *Christopher Crouse*[1]; Christian Pierce[1]; Jonathan Spowart[1]; [1]Air Force Research Laboratory

Metal nanoparticles have attracted much interest over the past decade as fuel sources for energetic materials due in part to their high surface areas and large energy densities. In combination with the appropriate oxidizer component, or secondary metal, these mixtures are capable of liberating energy through either a thermitic process or intermetallic formation, respectively. Without a strong interface between the component metal particles, these composites are typically very brittle, and offer little in terms of structural integrity. To this end, we have pursued monomer functionalization of the particles subsequently followed by an in situ polymerization to integrate a polymeric component (i.e. binder) into these systems. This approach affords a tunable energetic response through manipulation of the particle content and polymer selection. A detailed assessment of the chemistry employed to develop these composites along with an evaluation of their energetic performance will be presented.

**10:50 AM Invited**

**B/SiOx Nanonecklace Reinforced Nanocomposites by Unique Mechanical Interlocking Mechanism**: Xinyong Tao[1]; Jie Liu[1]; Goutam Koley[1]; *Xiaodong Li*[1]; [1]University of South Carolina

We demonstrate that nanonecklaces with SiOx beads on boron strings can be synthesized via a facile and environment-friendly method under atmospheric pressure. The continuous crystalline boron strings possess a thousand times higher electrical conductivity than bulk boron. The SiOx beads demonstrate good compatibility with the epoxy resin matrix. Due to the mechanical interlocking between the SiOx beads and the surrounding matrix, the reinforcement effect of this kind of nanonecklaces in epoxy nanocomposites is even better than normal carbon nanotubes. The marriage of boron strings and SiOx beads in the form of nanonecklaces is expected to exhibit unique electrical and mechanical properties for constructing nanodevices and nanocomposites. These nanoneckalce fiber reinforcement concepts are expected to have significant applications in the design and optimization of intricate nanostructures and nanocomposites with superior mechanical properties.

**11:10 AM**

**A Review of Fiber Waviness and Its Effect on Material Properties**: *Bryan Allison*[1]; Jeff Evans[1]; [1]University of Alabama in Huntsville

Fiber waviness is a common defect in fiber reinforced polymer (FRP) composites. Waviness is generally created during the manufacturing process. It negatively affects the material properties in most cases, particularly the ultimate compressive strength. This reduction has been reported to be over 80% in certain cases. This is not always the case, however. One case where waviness can actually increase the strength is buckling. This presentation is a review of how fiber waviness affects the mechanical properties of FRP, and a look at what future work might be on the horizon.

**11:30 AM**

**Structure and Capability of TiB2/UHMWPE Composite Shielding Material for Nuclear Radiation**: Xiaozhou Cao[1]; Xiangxin Xue[1]; Ting'an Zhang[1]; *Xianwei Hu*[1]; [1]Northeastern University

A new style TiB2/UHMWPE Composite Shielding Material for Nuclear Radiation was prepared by hot pressing method. The microstructure, mechanical property and radiation shielding of composite material were analyzed. The results indicate that each component was distributed continuously and evenly and the interface was combined closely. Hardness and tensile strength of the composite improved with TiB2 content increased. The absorption ration for thermal neutron increased with the thickness of composite increased.

## Aluminum Alloys: Fabrication, Characterization and Applications: Development and Application

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
*Program Organizers:* Subodh Das, Phinix LLC; Steven Long, Kaiser Aluminum Corporation; Tongguang Zhai, University of Kentucky

Monday AM Room: 615
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* John Chinella, U.S. Army Research Laboratory

**8:30 AM**

**High Strength and High Temperature Aluminum Alloys for High Performance Applications**: *Awadh Pandey*[1]; Jonathan Spowart[1]; [1]Pratt & Whitney Rocketdyne

New high strength and high temperature aluminum alloy has been developed to provide weight reduction benefit for rocket engines. The present aluminum alloy is based on a novel Al-Sc based alloy system. Aluminum-scandium alloy forms Al3Sc based precipitate that has an L12 structure which is strong and thermally stable up to very high temperatures. This material was made by a powder metallurgy technique consisting of ingot fabrication, powder production, compaction and extrusion. The proposed alloy has demonstrated very high strength for a range of temperatures up to 600F. High cycle fatigue and creep tests of these alloys were also conducted. Fatigue result of this alloy showed good endurance limit at room temperature. Creep resistance of this alloy was found to be significantly superior to the existing aluminum alloys. The

dominant strengthening mechanisms in this alloy is grain size strengthening, Orowan strengthening and antiphase boundary energy strengthening.

**8:50 AM**

**Scandium/Zirconium Modified Aluminum Alloys for Improved Mechanical and Corrosion Properties**: *Jennifer Gaies*[1]; [1]NSWC Carderock Division

Due to the need for high speed, lightweight ships there is an increased use of aluminum alloys in ship construction. These applications require materials with the following combination of properties: high welded strength, ductility and elongation, corrosion resistance, and fatigue and corrosion fatigue resistance. The most commonly used aluminum alloy families in marine applications are the Al-Mg (5XXX series) alloys which derive their strength from work hardening and are not heat-treatable. Reduced as-welded strengths limit their practical structural applications. The high temperatures introduced during the welding process reverse some of the effects of the strengthening mechanisms so aluminum alloys which can retain the greatest amount of post-welded strength are, therefore, greatly desired. Small additions of Sc and Zr have been shown to improve mechanical and corrosion properties of aluminum alloys and their effect as additions to commercial 5XXX alloys are investigated.

**9:10 AM**

**TRIMAL 52® - A New Aluminium Alloy for High Performance Spaceframe Construction**: *Marcel Rosefort*[1]; Horst Gers[2]; Thomas Koehler[1]; Jana Ehrke[1]; Dirk Schnapp[2]; Hubert Koch[1]; [1]Trimet Aluminium AG; [2]Honsel AG

Aluminium especially extrusions of the 6xxx series have considerable potential to make cars lighter and thus more economical. Trimet Aluminium AG has been supplying aluminium and services for the aluminium industry since 1988. TRIMET Essen is producing aluminium billets for high performance profiles especially in automotive applications since 1994. Over the time requirements has been changed to higher strength for profiles with lower wall thickness including better energy absorbing behaviour. Therefore TRIMET and Honsel started an innovative alloy and process development to give these profiles a considerably greater strength and better folding behaviour under load compared to other alloys available. Extrusions made out of this new alloy, trimal®-52, fulfil the highest demands of the OEMs. This paper describes the most important aspects of the production chain of aluminium for spaceframe application from the alloy development to the casting process and at last to test results of the final product.

**9:30 AM**

**High-Temperature Fatigue Deformation Behavior of Heat-Resistant Aluminum Alloy for Automobile Parts**: Jong-Soo Park[1]; Si-Young Sung[2]; Bum-Suk Han[2]; Chang-Yeol Jung[3]; *Kee-Ahn Lee*[4]; [1]Center for Advanced Green Materials Technology, Andong National University; [2]Korea Automotive Technology Institute; [3]Korea Institute of Industrial Technology; [4]Department of Advanced Material Science and Engineering, Andong National University

In this study, fatigue samples were processed from cylinder head parts that are actually used in domestic and foreign automobiles; high-temperature, high-cycle, and low-cycle fatigue characteristics were then evaluated and compared. The result of the tensile strength test on material B at 250° was higher by 15.9MPa compared to material A. On the other hand, elongation was 9.5% higher for material A. At 130°, material B exhibited high fatigue life given high-cycle fatigue under high stress, whereas material A showed high fatigue life when stress was lowered. With regard to the low-cycle fatigue result (250°) showing higher fatigue life as ductility is increased, material A demonstrated higher fatigue life. Through the observation of the differences in microstructure and the fatigue fracture surface, an attempt to explain the high-temperature fatigue deformation behavior of the materials was made.* supported by Fundamental R&D Program for Core Technology of Materials, Korea

**9:50 AM Break**

**10:05 AM**

**Optimized Heat Treatment Sequence for AA 6061**: *Christian Zelger*[1]; Josef Schnitzlbaumer[1]; Ramona Prillhofer[1]; Josef Enser[1]; Carsten Melzer[1]; [1]AMAG Rolling

Increasing material requirements regarding formability and mechanical properties sometimes force customers to apply either 5xxx alloys for better formability or 2xxx/7xxx alloys for higher strength in cases where standard 6xxx alloys and heat treatment processes fail. In this paper optimized heat treatment processes are presented, which allow to (i) form parts in T4 temper and reduce the subsequent time-consuming and costly artificial ageing step, or (ii) reach strength levels >10% above standard T6 temper or (iii) combine these two benefits. The advanced heat treatment processes enable 6xxx alloys to reach T6 temper within <1 hour compared to about 12 hours in standard processes. For the costumers this means significant time-, energy- and cost savings. Additionally a new heat treatment sequence allows reaching strength levels, which reduce the gap between known mechanical property levels of 6xxx alloys and 2xxx/7xxx alloys, without reducing high formability and corrosion resistance.

**10:25 AM**

**Microstructure, Mechanical Characterization and Hot Tensile Behaviour of Al-Zn-Mg Modified Alloys**: Paola Leo[1]; *Emanuela Cerri*[1]; Hugh J. McQueen[2]; [1]University of Salento; [2]Concordia University

Two Al-Zn-Mg alloys (Zr modified and not) were investigated by optical microscopy, hardness and tensile tests. The alloys were solutionized (2h at 490°C) and artificial aged at 130, 160, 190 and 220°C. Tensile tests were performed at room temperature on as-cast, solutionized and peak aged and the deformed samples annealed at 500°C. Tensile tests at 250°C-350°C and 10-5s-1 to 10-3s-1 were run on as-cast alloys. For both the alloys at room temperature, the solutionized samples exhibited the best ductility, the peak aged the highest strength and the as-cast alloy the highest strain-hardening rate. Hot tensile behaviour of both alloys is similar. The higher average grain size of the Zr alloy leads to lower hardness both in the as-cast and heat treated state and to lower peak stresses during room temperature tensile tests. Recrystallization of Zr-modified alloy is incomplete and not homogeneous as compared with unmodified alloy.

**10:45 AM**

**Cast Aluminum Housings in Electrical Fires**: *Joel Liebesfeld*[1]; [1]Countermeasure Security, Inc.

Cast aluminum and its alloys are often used as enclosures for electrical appliances and similar devices. Electrical faults can often be analyzed by the melting patterns of aluminum and its alloys. The properties of the various types of cast aluminum can assist the forensic investigator/engineer/scientist in forming accurate hypotheses that can be used for adherence to the "scientific method" standards needed for the proper procedural de-layering processes called for in loss analysis. The deployment of appropriate metallurgical test techniques and evaluations lends great credibility to the needed conformance to the scientific method. Aluminum castings are usually combined with other materials with differing melting temperatures and properties as components within enclosures, however in electrical applications the aluminum housing may have higher relevance as aluminum is an excellent electrical conductor and frequently used as an integral electrical grounding path for the subject encasements.

**11:05 AM**

**Influence of the Grain Size on the IGC, Crack Propagation and Fracture Toughness Behaviour of Aa2024-T3 Sheet Material**: *Josef Berneder*[1]; Reinhard Rachlitz[1]; Carsten Melzer[1]; Helmut Antrekowitsch[2]; Peter Uggowitzer[3]; [1]AMAG Rolling; [2]University of Leoben; [3]ETH Zürich

The alloy AA2024 is the most important sheet material for aerospace applications. Alloy AA2024 sheet material in T3 temper with variations in grain size and grain shape was produced on industrial practice. A significant difference between globular fine grain and in rolling direction elongated coarse grain material was observed regarding intergranular corrosion resistance (IGC) as well as fatigue crack propagation behaviour. In the present work we (i) describe the appropriate rolling procedures aiming at variations in grain's shape and size, (ii) investigate the secondary precipitates present at the grain boundaries after solution heat treatment and water quenching by means of transmission electron microscopy (TEM), and (iii) discuss the TEM results in view of IGC behaviour, fatigue crack propagation, and fracture toughness behaviour.

**11:25 AM**

**Microstructure and Mechanical Properties of Age-Hardening Al-Li-Sc-Yb, Al-Li-Sc, and Al-Sc Alloys**: *Matthew Krug*[1]; David Seidman[1]; David Dunand[1]; [1]Northwestern University

Three Al-based alloys were cast, homogenized, and aged producing strengthening L12 precipitates. A quaternary Al-5.31 Li-0.0448 Sc-0.0089Yb (at %) alloy was aged at 325°C, yielding core-shell Al3(Li,Sc,Yb) precipitates with a Yb-rich core and a Sc-rich shell, which confer strength at ambient and

elevated temperatures (>300°C). This was followed by an aging treatment at 170°C, resulting in precipitation of an Al3Li shell on the Al3(Li,Sc,Yb) precipitates. The additional shell yielded a large increase in volume fraction and strength. Precipitation-strengthened Al-2.90 Li-0.106 Sc (at %) and Al-0.106 Sc (at %) alloys were also aged at 325 °C, allowing direct investigation of the effect of Li addition on the mechanical properties and microstructural development of the Al-Sc alloy. Room-temperature Vickers microhardness measurements and compression creep of the three alloys at 300°C were interpreted on the basis of strengthening and creep models informed by microstructural observations by atom-probe tomography and TEM.

## Biological Materials Science: Bio-inspired Materials Design and Processing I: Macromolecular Concepts and Applications

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee

*Program Organizers:* John Nychka, University of Alberta; Jamie Kruzic, Oregon State University; Mehmet Sarikaya, University of Washington; Amit Bandyopadhyay, Washington State University

Monday AM Room: 205
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* John Nychka, University of Alberta

**8:30 AM Introductory Comments by John A. Nychka, 2010 Lead Organizer**

**8:35 AM Keynote**

**From Materials Science to Medicine Using Genetically Engineered Peptides**: *Mehmet Sarikaya*[1]; [1]University of Washington

With the recent developments of nanoscale engineering in physical and chemical sciences and the advances in molecular biology, molecular biomimetics is combining genetic tools and biological evolutionary approaches with synthetic nanoscale constructs to create a new hybrid methodology, genetically designed peptide-based molecular materials. Following the fundamental principles of biology-based design, molecular recognition, and self-assembly in nature, we can now use recombinant DNA technologies to design single or multifunctional peptides and peptide-based molecular constructs. These GEPIs, genetically engineered peptides for inorganics, have been making significant impact as inorganic material synthesizers, nano-particle linkers, and molecular assemblers, simply as molecular building blocks, in a wide range of fields from chemistry to materials science to medicine with practical implementations in biosensing, targeted assembly, hard tissue regeneration, probe and implant materials. This review presents a synopsis of the developments, current challenges, and future prospects. The research supported by NSF-MRSEC, -BioMat, and -IRES programs.

**9:15 AM**

**Electronic Transport through Solid-Binding Peptides**: *Yuhei Hayamizu*[1]; Marketa Hnilova[1]; Ersin Emre Oren[1]; Chao Zhong[1]; Candan Tamerler[1]; Marco Rolandi[1]; Mehmet Sarikaya[1]; [1]University of Washington

Genetically engineered peptides for inorganics (GEPIs) could be used as novel bio-molecular linkers in bioelectronic devices because of their materials-specific binding properties. We characterized their electronic transport using scanning tunneling microscopy and atomic force spectroscopy. As typical platforms, gold binding peptides (AuBPs) and ITO (indium-tin-oxide) binding peptides (ITOBPs) with specific binding affinity to gold and ITO surfaces, respectively, are used. The electronic transport properties of AuBPs, which were self-assembled and immobilized on the Au (111) surface, were investigated. Similarly, ITOBPs assembled on a highly smooth ITO were investigated. Several types of peptides which have different sequence of amino acids were used. The binding versus transport characteristics were studied using AuBPs on ITO and ITOBPs on Au (111) substrate to quantitatively investigate binding versus electron conductivity of these dodecapeptides. The research was supported by Genetically Engineered Materials Science and Engineering Center (GEMSEC), an NSF-MRSEC at the UW and an NSF-BioMat grant.

**9:35 AM**

**Computational Biomimetic Design of Materials Specific Peptides**: *Ersin Emre Oren*[1]; Ram Samudrala[1]; John S. Evans[2]; Malcolm L. Snead[3]; Martha J. Somerman[1]; Candan Tamerler[4]; Mehmet Sarikaya[1]; [1]University of Washington; [2]New York University; [3]University of Southern California; [4]Istanbul Technical University

Solid/protein interactions are at the heart of biology-inspired materials engineering, biomineralization, and bionanotechnology. Adaptations of biological principles relies on our ability to manipulate peptide/solid interfaces. Recently adapted biocombinatorial techniques permit isolation of peptides recognizing inorganic solids that are used as key molecules in the synthesis, assembly and formation of functional inorganics. To explore propensity rules connecting sequence and function that play major role in peptide/solid interaction, we developed a bioinformatics approach that further enables us to design novel peptides with specific affinities and multiple functionalities, e.g., peptides capable of binding to only silica or gold, both or neither. These peptides have utility in developing surface engineering of solids, combining several nanoentities, or molecular erectors when conjugated to other proteins, or to each other via a linker. Experimental verifications of designed peptides confirm our predictions with high accuracies. Supported by GEMSEC an NSF-MRSEC at the University of Washington, and NSF-BioMater Program.

**9:55 AM**

**Bridging Inorganic Nanoparticles and Biomolecules via Genetically Engineered Peptides**: *Turgay Kacar*[1]; Marketa Hnilova[1]; Banu Taktak[2]; Yuhei Hayamizu[1]; Ersin Emre Oren[1]; John Evans[3]; Candan Tamerler[1]; Mehmet Sarikaya[1]; [1]University of Washington; [2]Istanbul Technical University; [3]New York University

Genetically engineered peptides for inorganics (GEPIs), isolated through biocombinatorial approaches, were used for nanoparticle and protein immobilization on inorganic surfaces. Gold binding (AuBP) and quartz binding (QBP) peptide sequences were chemically linked, constituting bi functional GEPIs. The assembly of silica and gold nanoparticle was successfully carried out on gold and glass substrates, respectively, that were each functionalized with bi-GEPIs. Also, AuNPs were attached to the larger silica NPs yielding an optically active platform that enabled detection of a target biomolecule, i.e. Anti-Maltose Binding Protein (Anti-MBP), using AuBP-MBP as probe. Our results demonstrate that GEPIs can be an attractive approach for immobilization of inorganic NPs and proteins on a given solid substrate provided that the appropriate solid-specific peptide is used in the bifunctional entity. The novel molecular bi-GEPI platform including their protein fusion products have enormous potential in practical applications in nanobiotechnology. Supported by NSF-MRSEC and -IRES programs at the UW.

**10:15 AM Break**

**10:25 AM**

**Peptide-Mediated Formation of Hybrid Metallic Nanostructures**: *Marketa Hnilova*[1]; Hanson Fong[1]; Banu Taktak[2]; Turgay Kacar[1]; Candan Tamerler[1]; Mehmet Sarikaya[1]; [1]University of Washington; [2]Istanbul Technical University

Syntheses of metallic nanostructures mediated by combinatorially selected peptides and carried out at ambient conditions are potentially appealing as environmental- and bio-friendly alternatives to conventional chemical syntheses methods. We specifically probe gold-binding peptide (AuBP)-mediated gold crystal growth morphologies and kinetics in solution. We find that both AuBP sequences catalyze gold crystal growth in aqueous solution resulting in formation of stabile and dispersed peptide-capped gold-nanoparticles in a single reaction step. Additionally, we use novel recombinant maltose-binding proteins genetically fused to inorganic-binding peptides i.e., AuBPs, and demonstrate formation of multifunctional water-dispersible metallic nanostructures via gold-binding sequence motif. The peptide-based biomimetic approaches of synthesis metallic nanostructures described here have implications in a wide range of potential practical applications such as controlled bottom-up assembly of hybrid nanostructures, nanobiophotonic and biosensing platforms. The research was supported by GEMSEC, an NSF-MRSEC at UW, NSF-BioMat grant,and NSF-IRES, TUBITAK-NSF Joint Project, TR-SPO at MOBGAM-ITU.

**10:45 AM**
**In Situ Biomineralization Using Peptides via SPR and QCM**: *Brandon Wilson*[1]; Eugene Ngai[1]; James Park[1]; Mustafa Gungormus[1]; Marketa Hnilova[1]; Candan Tamerler[2]; Mehmet Sarikaya[1]; [1]University of Washington; [2]Istanbul Technical University

There is an ever increasing interest in the understanding of mechanism of hydroxyapatite (HA) formation in both biological and synthetic environments. Practical methods are sought for synthesizing HA with controlled structures using a variety approaches. Here we use bifunctional peptide to mineralize a HA film on a titanium substrate. The experiments were carried by a combining titanium-binding peptide (TiBP) and HA-binding peptide, HABP, sequences into a bi-functional construct. Biomineralization was catalyzed by immobilized in various solution conditions. The kinetics of the biomineralization process was monitored by liquid-mode QCM. Scanning electron microscopy (SEM) was used to examine the film morphology. The results will be discussed in enzymatic and morphogenesis effects of the peptide. The process developed here may be used in wide ranging applications in biotechnology from tissue engineering to creating biocompatible surfaces. This research is supported by GEMSEC, and NSF-MRSEC at the University of Washington, NSF-IRES, and NSF-BioMat programs.

**11:05 AM**
**Self-Mineralized/Self Assembled Peptide-based Hydrogels as Scaffolds for Tissue Regeneration**: *Mustafa Gungormus*[1]; Monica Branco[2]; Hanson Fong[1]; Candan Tamerler[1]; Joel Schneider[2]; Mehmet Sarikaya[1]; [1]University of Washington; [2]University of Delaware

We developed an in situ forming, self assembling peptide hydrogel for hard tissue-engineering with a high, inherent propensity for calcium phosphate mineralization. The hydrogel possesses a genetically engineered hydroxyapatite binding peptide (HABP). In vitro solution and cell mineralization studies demonstrated that the HABP retains the mineralization capability when attached to the hydrogel resulting in a hybrid scaffold with an inherent ability to regulate the HA formation. The mineral deposited on the hybrid hydrogel was elongated, biological-like apatite while poorly crystalline calcium phosphate was deposited on the native hydrogel. This study demonstrates that inorganic-binding peptides conjugated to self assembling peptide hydrogels participate in the biomineralization process similar to a native extra cellular matrix. The ability to engineer such three-dimensional assemblies with spatial control and programmed functions may find substantial use in tissue engineering applications for successful restoration or regeneration. The research supported by NSF-MRSEC and-IRES programs at the University of Washington.

**11:25 AM**
**Peptide-Based Biofunctionalization of Implant Materials**: *Dmitriy Khatayevich*[1]; Mustafa Gungormus[1]; Christopher So[1]; Sibel Cetinel[2]; Hong Ma[1]; Alex Jen[1]; Candan Tamerler[2]; Mehmet Sarikaya[1]; [1]University of Washington; [2]Istanbul Technical University

Genetically engineered peptides for inorganics (GEPIs) offer unique advantages for biomaterials functionalization over the conventional molecular immobilization methods. They require no hostile environments to bind, are specific to their materials, can be adapted to carry various active entities, and are unlikely to exhibit toxicity. In this study we used peptides selected for gold, platinum and glass to modify surfaces with Poly(ethylene glycol) anti-fouling polymer and RGD integrin binding sequence. Using this approach we imparted anti-fouling properties to gold and platinum via 3GBP1 and PtBP1 binding sequences. We also induced a 2.5 fold increase in the number and a 4 fold increase in the spreading of osteoblast cells on glass using the QBP1-RGD construct. Work is ongoing to take advantage of GEPI selectivity for simultaneous functionalization of multi-material substrates. The research was supported by Genetically Engineered Materials Science and Engineering Center (GEMSEC), an NSF-MRSEC at the UW and an NSF-BioMat grant.

**11:45 AM**
**Directed Assembly and Fabrication by Materials Selective Fusion Protein-Peptides**: *Candan Tamerler*[1]; Mehmet Sarikaya[2]; [1]Istanbul Technical University; [2]University of Washington

Bio-based building blocks are explored for establishing novel routes to develop new performance materials, manufacturing processes and novel biological materials. In our approach, we design, and utilize peptides and multifunctional proteins as molecular tools. Current biotechnological applications such as biosensors, protein arrays and microchips require oriented immobilization of enzymes. The characteristics of recognition, selfassembly and ease of genetic manipulation make inorganic binding peptides an ideal molecular tool for site-specific enzyme immobilization. Here, we provide examples on the use of peptides as fusion partners to different proteins in creating biocompatible nanomaterials, self immobilization on material surfaces, and formation of scaffolds for tissue restoration and regeneration. Among them, hydroxyapatite binding peptide linked to green fluorescence protein to target biomineralization, and laccase fused with gold and silica binding peptides as biosensors and as a biocathode for fuel cell application will be discussed. Research Supported by GEMSEC-UW, NSF-MRSEC, NSF-BIOMAT and TUBITAK-NSF Joint Project, TR-SPO

## Bulk Metallic Glasses VII: Structures and Mechanical Properties I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
*Program Organizers:* Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Monday AM Room: 213
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Takeshi Egami, University of Tennessee; Peter Liaw, University of Tennessee

**8:30 AM Keynote**
**Mechanical Failure and Glass Transition**: *Takeshi Egami*[1]; Pengfei Guan[2]; Mingwei Chen[2]; [1]University of Tennessee; [2]WPI-AIMR, Tohoku University

It is not easy to elucidate the properties of metallic glasses based upon the atomic structure. In the absence of the established theory many use the free-volume model to explain the experimental results, even though it does not work well for metallic liquids. We developed an alternative theory based upon the concept of atomic level stresses to explain the glass transition and the glass transition temperature. We now expand the theory to account for mechanical yield and failure. We see failure as the glass transition induced by shear stress, and explain it in terms of percolation of the atomic sites made topologically unstable by applied stress. We also relate the stress sensitivity of the glass transition to the shear thinning phenomenon in supercooled liquids. We show the formation of shear bands and local heating as consequences of the stress-induced glass transition, not the cause.

**9:00 AM**
**Embrittlement of Bulk Metallic Glasses**: *Golden Kumar*[1]; Dale Conner[2]; Jan Schroers[1]; [1]Yale University; [2]California State University

Embrittlement of bulk metallic glasses (BMGs) was studied after annealing at temperatures below and above Tg, for time scales comparable to structural relaxation and crystallization. The effect of annealing on the bending ductility, the isoconfigurational elastic constants, the structure, and the thermal stability is examined. The embrittlement during sub-Tg annealing originates from structural relaxation and can be reversed by subsequently annealing above Tg. The embrittlement kinetics correlate with the structural relaxation. However, only a fraction of relaxation time at a given temperature (below Tg) is sufficient to significantly embrittle the Zr-based BMGs. Above Tg, plasticity is retained for annealing far beyond the relaxation time but instead embrittlement is caused by crystallization. The values of $\upsilon$ and $\mu/B$ during annealing-induced embrittlement remain well above and below the respective critical values (0.31 for $\upsilon$ and 0.41 for $\mu/B$) previously proposed to denote the transition from a tough to brittle behavior.

**9:10 AM Invited**
**Anelastic Deformation of a Metallic Glass**: *Michael Atzmon*[1]; Jong Doo Ju[1]; Dongchan Jang[2]; [1]University of Michigan; [2]Caltech

We report on our ongoing studies of anelastic deformation in amorphous Al86.8Ni3.7Y9.5. Using a combination of bend stress relaxation and nanoindenter cantilever measurements, time scales ranging from 10 to 10^7 s were probed. We had previously reported on multiple time constants for anelastic

relaxation for this alloy. Recent improvements in precision have allowed us to refine our results, with attention to the state of relaxation and deformation history. While plastic deformation and annealing are generally assumed to have opposite effects on the atomic structure metallic glasses, our results indicate that they affect the anelastic response at short time scales in a similar manner. This suggests that both deformation and annealing lead to removal of similar deformation sites. In separate experiments, using surface profiling, we observe time-dependent flow at shear bands.

**9:30 AM**

**In situ X-Ray Diffraction Investigations of Cu-Zr Bulk Metallic Glasses under Applied Stress**: *Norbert Mattern*[1]; Jozef Bednarcik[2]; Gang Wang[1]; Juergen Eckert[1]; [1]IFW Dresden; [2]DESY Hamburg

The structural behaviour of Cu64.5Zr35.5, Cu50Zr45Al5, Cu50Zr45Ti5 bulk metallic glasses was investigated in situ under compressive stress by means of high energy X-ray synchrotron diffraction. The changes of the structure factor in reciprocal space and in the atomic pair correlation function in real space were analyzed in detail upon loading and unloading. The components of the elastic strain tensor were determined by the shift of the position of first maximum of the structure factor as well as by the higher order maxima of the atomic pair correlation function. The obtained elastic moduli agree qualitatively with those measured macroscopically. The atomic pair correlation functions indicate changes of the nearest neighbourhood under stress even in the elastic regime. The number density of Cu-Zr and Zr-Zr nearest neighbour atomic pair becomes oriented along the loading direction, pointing to changes in topological and chemical short-range order. Possible mechanism will be discussed.

**9:40 AM Invited**

**Structure – Property Relationship in Bulk Metallic Glasses**: *Evan Ma*[1]; [1]Johns Hopkins University

For monolithic BMGs that are completely and invariably amorphous, an understanding of how the internal structures influences strength, ductility, relaxation dynamics remains elusive. Here we resolve the structure of typical BMG-forming Zr-Cu, and Zr-Cu-Al [PRL (2009)], in relationship with the properties [PRB (2008)]. We identify the structural disordering processes responsible for the initiation of shear flow [Acta Mater. (2008)] and localization [Acta Mater. (2009)], and the fertile and resistant sites for carrying shear transformations. The composition-dependent and processing(cooling rate)-dependent local order is monitored in a quantitative manner, in lieu of arguments based on free volume. This structural perspective offers an explanation to the reported (e.g., Zr-rich) BMGs with large plasticity, and allows the prediction of ductile BMGs [Acta Mater. (2009)]. We also illustrate the apparent correlation between the elastic constants (such as G, B) and the behavior of BMGs [Acta Mater. (2009)], in terms of the structural origin.

**10:00 AM Break**

**10:10 AM Invited**

**Defects and Plastic-Deformation Modes of Bulk-Metallic Glasses**: *Yuri Petrusenko*[1]; Alexander Bakai[1]; Ivan Neklyudov[1]; Igor Mikhailovskij[1]; Sergij Bakai[1]; Peter K. Liaw[2]; Lu Huang; Tao Zhang[3]; [1]National Science Center - Kharkov Institute of Physics and Technology; [2]Department of Materials Science and Engineering, The University of Tennessee; [3]Department of Materials Science and Engineering, Beijing University of Aeronautics and Astronautics

This report describes the results of theoretical and experimental investigations of mechanisms of the plastic deformation of bulk-metallic glasses (BMGs) by taking into account of their heterogeneous polycluster structures. It will be shown that the intercluster boundaries and vacancies are the most important defects of BMGs controlling the small-scale creep processes, shear-band formation, and diffusion-viscous flow. The boundary structure determines initial slipping processes under shear stresses. The boundary structure can be changed under repeating loading-unloading processes or ultrasonic vibrations (USV). The impact of USV on the Kaiser effect is chosen as a tool for studying the boundary-slip initiation and effect of vibrations on the boundary structure. The generation and stability of vacancies under the electron irradiation in BMGs are investigated. The map of the plastic-deformation states of metallic glasses is studied and discussed.

**10:30 AM Invited**

**Structural Features of BMG under Mechanical Deformation**: *Wojciech Dmowski*[1]; Andrew Chuang[1]; Peter Liaw[1]; Yang Ren[1]; Jon Almer[1]; Takeshi Egami[1]; [1]University of Tennessee

We investigated changes in the atomic structure in metallic glasses that had been deformed in a homogeneous and inhomogeneous way and in the elastic regime. We attempted to resolve elastic, inelastic and anelastic contributions. We examined structural changes induced by high temperature creep followed by high temperature recovery. The X-ray diffraction was carried in-situ during loading, after creep and recovery. The data was analyzed by expanding the scattering function and PDF in terms of Legendre polynomials. We observed the development of a bond anisotropy that supported apparent macroscopic shear strain after high temperature creep. High temperature recovery reduced bond anisotropy; however, some plastic strains were irreversible. We compare changes in the atomic structure of BMG due to different modes of deformation: homogeneous creep, inhomogeneous plastic deformation and elastic deformation.Work was supported by the U.S. DOE under DE-AC05-00OR-22725 and NSF-DMR-0906744.

**10:50 AM**

**(Fe,Co)-Based BMGs with Small Cu Additions**: *Mihai Stoica*[1]; Ran Li[1]; Stefan Roth[1]; Jürgen Eckert[1]; Gavin Vaughan[2]; Alain Yavari[3]; [1]IFW Dresden; [2]ESRF Grenoble; [3]INP Grenoble

Recently, (Fe-Co)-B-Si-Nb bulk metallic glasses (BMGs) were produced. Such BMGs exhibit high glass-forming ability (GFA) as well as good mechanical and magnetic properties. These alloys combine the advantages of functional and structural materials. The soft magnetic properties can be enhanced by nanocrystallization. To force the nanocrystallization, small content of Cu was added to the starting composition. In this paper, $\{[(Fe_{0.5}Co_{0.5})_{0.75}Si_{0.05}B_{0.20}]_{0.96}Nb_{0.04}\}_{100-x}Cu_x$ glassy alloys ($x$ = 1, 2 and 3) were chosen for investigation. The GFA and the thermal stability of these alloys were evaluated. The effects of crystallization during heat-treatment processes on the phase evolution, the kinetic parameters and the magnetic properties, including $M_s$, $H_c$ and $T_c$, in these alloys were investigated. The phase analyses were done with the help of the X-ray diffraction patterns recorded in-situ by using the synchrotron radiation in transmission configuration.

**11:00 AM Invited**

**Irreversible Structural Changes with Cyclic Loading in Zr Based Amorphous Alloys**: *Despina Louca*[1]; Peng Tong[1]; Gongyao Wang[2]; Peter Liaw[2]; Yoshihiko Yokoyama[3]; [1]University of Virginia; [2]University of Tennessee; [3]Tohoku University

Bulk amorphous metallic alloys possess unique mechanical properties, suitable in a wide range of engineering applications. However, they tend to exhibit low ductility and are particularly vulnerable to fatigue damage as irreversible changes occur through the formation of shear bands during the deformation process. The physical mechanism leading to failure under fatigue-loading conditions is currently not well understood. To determine the deformation behavior at the microscale, the local atomic structure of Zr-based glasses subjected to cyclic fatigue tests is investigated. In order to provide a direct description of the three-dimensional structure and associate the atomic environment to physical properties, the pair density function analysis of neutron and X-ray diffraction data is used. Our results indicate that the cyclic-fatigue effects are not elastic. At the same time, Zr glasses show improved ductility upon cooling and the results from the local structure analysis will be additionally presented.

**11:20 AM**

**Effect of Plastic Deformation History on the Subsequent Mechanical Behavior of a Bulk-Metallic Glass: A High-Energy Synchrotron X-Ray Scattering Study**: *Feng Jiang*[1]; Dongchun Qiao[1]; Yang Ren[2]; Wojtek Dmowski[1]; Gongyao Wang[1]; Yangdong Wang[3]; Takeshi Egami[1]; Peter Liaw[1]; Hahn Choo[1]; [1]University of Tennessee; [2]Argonne National Laboratory; [3]Northeastern University

The elastic-strain tensors of a (Zr55Cu30Al10Ni5)99Y1 bulk metallic glass with and without deformation history have been studied at ambient temperature by using high-energy synchrotron x-ray diffraction facility with an in-situ mechanical loading device. The results show that the specimens with plastic deformation history, introduced by performing four-point bending fatigue, exhibits more compliant elastic behavior than the specimen without plastic

deformation history. Furthermore, more pronounced changes in the diffraction peak widths were observed in the elastic-scattering intensity, I(q), of the specimen with the deformation. The results will be discussed in terms of the increases in the residual-stress and/or free-volume during the plastic deformation and their roles in the subsequent deformation behavior of the BMG alloys.

**11:30 AM Invited**

**Atomic-Scale Mechanisms of Tension-Compression Asymmetry in a Metallic Glass**: Lianyi Chen[1]; B. Z. Li[1]; X. D. Wang[1]; Feng Jiang[2]; H. Franz[3]; Ren Yang[4]; Peter Liaw[2]; *Jianzhong Jiang*[1]; [1]Zhejiang University; [2]University of Tennessee; [3]HASYLAB at DESY; [4]Argonne National Laboratory

Materials exhibit tension-compression asymmetry in terms of plasticity and/or strength. This phenomenon is usually interpreted by continuum mechanics, which neglects the stress-induced structure change. Here we investigated the structure change of a metallic glass of Cu45Zr46Al7Ti2 under both the tension and compression stresses by in-situ loading high-energy X-ray diffraction. A correlation of the stress-induced structure change with the extraordinary tension-compression asymmetry in the metallic glass will be discussed. The results obtained in this work might provide a new perspective on the tension-compression asymmetry from the structural aspect, which is useful for understanding the deformation mechanisms of metallic glasses and may guide the development of plastic metallic glasses.

**11:50 AM**

**Thermomechanical Behavior of Cu50Hf41.5Al8.5 Bulk Metallic Glass Following Cyclic and Static Elastic Compression in Different Loading Directions**: Arif Mubarok[1]; *Rainer Hebert*[1]; [1]University of Connecticut

Recent temperature-modulated thermomechanical analyses have demonstrated that cyclic deformation of bulk metallic Cu50Hf41.5Al8.5 glass tends to reduce the non-reversible length decrease during heating while static elastic compression for the same amount of time as the cyclic testing and at the peak stress of the cyclic test enhanced the length decrease in the supercooled liquid region. As a continuation of the static and cyclic elastic tests, cubic-shaped bulk metallic Cu50Hf41.5Al8.5 samples were exposed to static and cyclic elastic compression tests, followed by thermomechanical analysis to determine the isotropy of the length changes relative to the compression direction and the axial direction of the initial cast bar. Anisotropic thermomechanical behavior was observed for the as-cast samples compared to the structurally relaxed samples that reveal isotropic thermomechanical behavior. Sustained elastic deformation emerges as a novel approach for changing the level of structural relaxation of metallic glasses and imparting directionality on the thermomechanical behavior.

**12:00 PM**

**Static and Dynamic Observation of Shear Bands in Metallic Glasses**: *Eun Soo Park*[1]; Frans Spaepen[2]; [1]Seoul National University; [2]Harvard University

Understanding how shear bands from and propagate in amorphous metals is essential for the eventual use of these materials in structural applications. However, only little quantitative information has been collected on their dynamics. Furthermore, the structure and density of these shear bands has been studied mostly after the fact. In the present study, using self-designed experimental setup, nucleation and density of shear bands depending on strain rate has been clearly observed in bend test on thin ribbons. We will compare the static and dynamic observation results for shear bands in mainly iron-based metallic glasses.

**12:10 PM**

**Effect of Stress State on Flow at Bulk Metallic Glass Interfaces**: *Nicholas Hutchinson*[1]; Dan Campbell[1]; Katharine Flores[1]; [1]The Ohio State University

Plastic deformation in metallic glasses is a dilatational process that depends on both the shear and normal stress on the flow plane. It is therefore interesting to consider the effect of stress state on material mixing and mechanically assisted diffusion across an interface as a mechanism for solid state joining. We evaluate the effect of interface orientation relative to the loading axis on material flow and joint formation via a thermo-mechanical joining technique. Post-test characterization of the interfaces was conducted using X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Surprisingly, although plastic flow is commonly considered to be a shear driven process, preliminary experiments with 45° interfaces indicate that the addition of shear stresses actually impedes joint formation relative to that observed for pure compression. This suggests that normal stress-driven diffusion plays a much larger role in joint formation than shear stress-driven mechanical mixing.

## Characterization of Minerals, Metals and Materials: Characterization of Iron and Steel I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee

*Program Organizers:* Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF; Jiann-Yang Hwang, Michigan Technological University

Monday AM Room: 307
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Jiann-Yang Hwang, Michigan Technological University; Donato Firrao, Politecnico di Torino

**8:30 AM Introductory Comments**

**8:35 AM**

**Nano-Scale Characterization of Carbon Partitioning from Supersaturated Plates of Ferrite**: *Francisca Caballero*[1]; Mike Miller[2]; Carlos Garcia-Mateo[1]; [1]CENIM-CSIC; [2]ORNL

A new processing concept has been proposed for designing ductile steels. The 'quenching and partitioning' process involves quenching austenite below the martensite-start temperature, followed by a partitioning treatment to enrich the remaining austenite with carbon, thereby stabilizing it to room temperature. It is important to understand the partitioning of carbon from supersaturated plates of ferrite. Carbon partitioning is also critically relevant for fundamental reasons associated with the bainite transformation mechanisms. In these processes, the supersaturation must be relieved either through precipitation of carbides, or by partitioning to austenite. This atom probe tomography study investigated the redistribution of carbon during the bainite formation and during subsequent tempering in a nanocrystalline steel that transforms at abnormally low temperatures and exhibits extremely slow transformation kinetics. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

**8:55 AM**

**Nondestructive Characterization of Microstructure and Properties of Steel Products**: *Jagdish Pandey*[1]; Manish Raj[1]; Nikhiles Bandyopadhyay[1]; [1]Tata Steel Ltd.

The paper highlights the experimental investigations to characterize texture and plastic strain ratio r-Bar in auto grade steel sheets by ultrasonic measurements, whereas attempts were made to characterize interlamellar spacings in pearlite, tensile stresses and torsional properties in prestressed concrete wires by eddy current measurements. Good correlations were obtained between the testing parameters (such as ultrasonic attenuation and velocity in case of steel sheets as well as eddy current out put voltage and phase angle in case of steel wires) and the materials properties (such as texture, plastic strain ratio r-Bar, interlamellar spacings in pearlite, tensile stresses and torsional properties). 15 MHz normal direct contact and 20 MHz delay line normal shear wave probes were used for ultrasonic measurements. Eddy current measurements were done using an absolute coil probe with variable frequency selection. The experimental results show the possibility of nondestructive on line measurement of the above microstructures and properties.

**9:15 AM**

**Role of Microstructure and Composition in Resisting Hydrogen Embrittlement of Fastener Grade Steels**: *Nicholas Nanninga*[1]; Lloyd Heldt[2]; Karl Rundman[2]; [1]NIST; [2]Michigan Technological University

The degree of hydrogen embrittlement for several fastener grade steels has been determined. The technique used to assess the resistance to hydrogen embrittlement of the steels was the rising step load test on notched tensile specimens. While microstructural alteration resulted in some improvement in resistance to hydrogen embrittlement, the overriding factor contributing to susceptibility of the steel was strength. The susceptibilities of the microstructures

to hydrogen embrittlement, ranked in decreasing order, are as follows: tempered martensite, bainite, fine pearlite. The effect of alloying was also assessed by comparing rising step load results from different fastener grade steels with similar microstructures. In most cases, the alloy chemistry had little effect on rising step load results, presumably due to trap saturation associated with this testing technique.

**9:35 AM**

**2D Imaging of Nano-Hardness and Phase Characterization in Steel by Noncontact Optical Photodeflection Microscopy**: *Nelida Mingolo*[1]; Oscar Martínez[1]; Ulises Crossa Archiopoli[2]; [1]Universidad de Buenos Aires; [2]Tolket SRL

We describe a technique that allows rapid imaging of the thermal diffusivity of the sample by a modified photo-thermal technique. The thermal diffusivity in hardened steels strongly correlates to the hardness. Hence in a noncontact manner and with optical microscope resolution, a 2D map exhibiting the crystallographic phases and hardness of the sample can be retrieved. Two almost collinear laser beams scan the surface, one as a modulated pump and a second as a CW probe. The deflection of the probe carries information on the thermal expansion of the sample and with it, on the thermal diffusivity. It is shown that a single determination of the time delay of the modulation of the probe beam can be used to retrieve the thermal diffusivity.

**9:55 AM**

**Characterization of AISI 4340 Steel Formed by Direct Metal Deposition Process**: *Jyotirmoy Mazumder*[1]; Sudip Bhattacharyya[1]; [1]University of Michigan

Materials generated by Direct Metal Deposition, a rapid prototyping technique for manufacturing near-net shape components, usually have very fine microstructure and residual stresses due to extremely high cooling rates. AISI 4340 is extensively used in many applications including submarine connecting rods. In this investigation microstructure of 4340 steel deposited on a mild steel substrate using a 6 kW continuous CO2 laser was investigated using optical, scanning electron microscopy (SEM) and x-ray diffraction (XRD) techniques. Ferrite, martensite and cementite phases were determined by XRD. Transmission electron microscopy (TEM) corroborated the findings and change in crystal structure e.g. lattice parameters due to fine microstructure and residual stresses. Bright field TEM images and corresponding selected area diffraction (SAD) patterns confirmed the ferrite and cementite phases and show that there is an approximately 0.5-2% reduction in the lattice parameters of these phases. No retained austenite phase was found in either XRD or TEM results.

**10:15 AM**

**Classification and Rating of Inclusions in a High Carbon Steel**: M. Faraji[1]; R. Thackray[1]; I. Todd[1]; *P. Tsakiropoulos*[1]; [1]The University of Sheffield

Inclusions formed in a high-carbon, manganese-silicon killed plain steel (Fe-0.77C wt%) were studied. Various techniques were used to characterise the inclusions, and the latter were classified according to their chemical composition, size and morphology. The main constituent elements entering the inclusions were Mn, S, Al, and oxygen. Alongside MnS and some Al2O3 inclusions many non-metallic inclusions appeared in complex forms, consisting of silicates, sulphides and oxides. Duplex oxy-sulphides, mainly MnS combined with Al2O3, or SiO2 were the most common complex multiphase inclusions in this steel. The nanoindentation technique was used to assess and rate different types of inclusions and data of the hardness and elastic modulus of individual inclusions at room temperature are presented.

**10:35 AM**

**Inspecting and Analyzing Microstructural Homogeneity in Grey Cast Iron**: *M. David Hanna*[1]; [1]General Motors R&D Center

There is a great deal of emerging evidence that microstructural homogeneity of grey cast iron has significant effects on mechanical and physical properties that are directly influenced by the amount, size, and morphology of graphite flakes present throughout the casting. The large mismatch in physical and mechanical properties between graphite flakes and the iron matrix is significant. Hence, it has become important to analyze and inspect microstructural homogeneity in grey cast iron used for many applications with one fundamental motivation being to obtain a uniform microstructure. In this study a wide range of grey irons having different compositions and casting mold designs were analyzed using an image analyzer to reveal the extent of graphite flake size (GFS) homogeneity. A review of the literature and published work concerning homogeneity of the microstructure was conducted and information about effects on physical properties was gathered.

**10:55 AM**

**An Approach for Graphite Nodules Detection in Ductile Cast Iron**: *Ali-Reza Kiani-Rashid*[1]; S.A. Rounaghi[1]; [1]Ferdowsi University of Mashhad

The present study has been conducted to the as-cast samples. The aim of this work is to acquire the structural information about graphite detection in experimental cast iron so there is no doubt between this kind of phases with foreign particles such as inclusions and impurities which are reported occasionally instead of graphite in Metallography of cast irons. In this paper the problem of identification and characterization of the graphite nodules have been considered and a novel qualitative analysis procedure has been developed based on a chemical etchant technique. This is a kind of methodology for image analysis of the material metallographic specimen's pictures that provide a reliable and efficient separation of the elements of interest from the background and the evaluation of their morphological characteristics.

**11:15 AM**

**As-Cast Microstructures of Aluminum Containing Ductile Cast Irons**: *Ali-Reza Kiani-Rashid*[1]; A Shayesteh-Zeraati[2]; H. Naser-Zoshki[3]; M.R. Yousef-Sani[1]; [1]Ferdowsi University of Mashhad; [2]Sharif University of Technology; [3]Iran University of Science and Technology

In this paper, the effect of aluminum content on the formation mechanism, volume fraction, morphology and particle size distribution of graphite has been investigated. Addition of aluminum on ductile iron causes some fundamental changes in iron-carbon phase-diagram and as a result, improves the graphite formation during eutectic transformation. The results reveals that aluminum compounds have been formed in the core of graphite nodules, thus aluminum plays an important role in the formation of graphite nodules. Furthermore, it is indicated that an increase in the aluminum content also leads to an increase in the number of graphite nodules and a decrease in the nodules size. By using EPMA, the segregation of aluminum and silicon between graphite nodules has been studied.

**11:35 AM Concluding Comments**

**11:40 AM Question and Answer Period**

## Coatings for Structural, Biological, and Electronic Applications: Processing Techniques and Characterization

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS: Thin Films and Interfaces Committee

*Program Organizers:* Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Roger Narayan, Univ of North Carolina & North Carolina State Univ; Choong-un Kim, University of Texas at Arlington; Nancy Michael, University of Texas at Arlington

Monday AM — Room: 309
February 15, 2010 — Location: Washington State Convention Center

*Session Chair:* Gregory Krumdick, Argonne National Labs

**8:30 AM Introductory Comments**

**8:35 AM Invited**

**Morphological Evolution during the Annealing and Growth of Polycrystalline Films**: *Ramanathan Krishnamurthy*[1]; Mikko Haataja[2]; [1]Purdue University; [2]Princeton University

Polycrystalline films are commonly used in materials applications; however, growth models rarely include time-dependent lateral grain size effects. We employ a thermodynamics-based method to effectively handle grain grooving / lateral grain growth. We model the annealing of a film (zero deposition flux) with polydisperse grain sizes and successfully predict several experimental observations on surface-diffusion dominated grooving and boundary motion (ghost lines, groove asymmetry effects). We also predict experimentally observed, grain aggregate behavior, such as the non-monotonous time-variation

of film roughness. We discuss simulation results vis-a-vis the assumed values for surface diffusivity / grain mobility. For film growth (non-zero deposition flux), the greater chemical potential driving grooving when a large average deposition flux (compared to surface diffusivity /grain mobility) is employed results in enhanced film grooving / impeded grain growth. We discuss the effects of spatially varying deposition fluxes on film growth, with a form chosen to simulate electrodeposition.

**9:05 AM**
**Synthesis of Amorphous Al-Co-Ce Alloys via Atomization and Mechanical Milling**: *Zhihui Zhang*[1]; Ying Li[1]; Yizhang Zhou[1]; Enrique Lavernia[1]; [1]University of California Davis

Amorphous Al-Co-Ce coatings have demonstrated unique protection capability when applied to a high-strength Al substrate. These coatings can serve as a protection barrier, sacrificial anode, and reservoir to supply inhibitor ions to protect any defect sites, due to the structural amorphicity and chemical composition. In this study, the glass forming ability of Al86Co10Ce4 and Al89Co7Ce4 alloys, produced via gas atomization, were systematically investigated by varying atomization parameters (superheat 175-340°C, atomization pressure 1.4-6.9 MPa) and using helium as atomization gas. The results showed that it was difficult to suppress the crystallization of the intermetallic compounds Al9Co2 and Al11Ce3 even when the powder particle size was separated down to <10 µm. Alternatively, complete amorphization of the Al9Co2 and Al11Ce3 compounds was achieved by mechanical milling the atomized powder. The effect of initial amorphicity and chemical composition in the starting powder on the amorphization kinetics during mechanical milling is discussed in detail.

**9:25 AM Invited**
**Microstructure and Mechanical Properties of Vanadium Oxide-Based Coatings on Steel Substrates Prepared by Pulsed Laser Deposition**: Andreas Jahja[1]; *Paul Munroe*[2]; [1]Materials Science and Engineering; [2]Materials Science and Engineering, University of New South Wales

A range of vanadium oxide coatings were deposited on a H13 tool steel substrate via pulsed laser deposition as a function of processing conditions. The coatings were subject to detailed microstructural characterization, including cross-sectional TEM studies. It was shown that the pressure of the oxygen reactive gas during deposition was critical to the quality and performance of the coating. That is, more compact and resilient coatings were prepared at lower oxygen concentrations. Further, high oxygen pressures promoted the formation of iron oxide on the substrate prior to deposition. These iron oxide layers promoted delamination of the vanadium oxide. Mechanical behaviour was assessed through nanoindentation using a spherical indenter. The coatings exhibited high hardness values and significant resistance to cracking. Examination of the indented layers revealed intercolumnar cracks within the coatings, together with shear steps at the coating-substrate interface, whilst inclined cracks were observed at the periphery of the indentations.

**9:55 AM Break**

**10:10 AM Invited**
**Method of Characterizing Pore Structure in Porous Coating Layer Using a Simple Voltammetry**: *Nancy Michael*[1]; Woong-Ho Bang[1]; Choong-Un Kim[1]; [1]University of Texas at Arlington

Porous thin films are used in various applications, including the coating for biomaterials and energy storage materials, as they provide unique electrical, structural and chemical properties that are not available at the bulk. Characterization of pore structure is typically done by a direct observation of pores using microscope, but such techniques only visualize limited volume of the film. The need for new characterization techniques is growing, especially because the use of thin films with extremely small pore size, is expanding. A new technique that can characterize the pore structure without limitations of microscopy is developed in our research. This technique is based on a simple voltammetry that uses ions in electrolyte as pore size tracer and found to work even with nanoscale pores. This paper introduces theoretical background and experimental examples of the technique.

**10:40 AM**
**The Effect of Aging Time on the Properties of Sol-Gel Derived Nanostructure Fluorapatite Powder and Coating**: *Ehsan Mohammadi Zahrani*[1]; M. H. Fathi[2]; Akram Alfantazi[1]; [1]The University of British Columbia; [2]Isfahan University of Technology

The effect of aging time on the formation and phase purity of nanostructure fluorapatite (FA:Ca10(PO4)6F2) powder and coating was evaluated in this work. Fluorapatite nanostructure powder and coating were prepaid by sol-gel method using calcium nitrate tetrahydrate, phosphorous pentoxide and hexafluorophosphoric acid as starting materials. Precursor sols were dip coated onto the pure 316L stainless steel substrate. The coated plates were aged at different aging times and dried. Finally samples were calcinated at 600°C. Thermal behaviour, phase formation, size and morphology of nanoparticles, surface morphology and also interfacial coherency of the coating as well as functional groups were investigated by thermogravimetry analysis, x-ray diffraction, transmission electron microscopy, scanning electron microscope and fourier transform infrared spectroscopy, respectively. Results showed that a solution aging time of at least 18-24 hr was required to achieve monophasic nanostructure FA powder and coating. The nanoparticles size was increased as a result of aging time increasing.

**11:00 AM Invited**
**Microstructure and Mechanical Properties of Yttria-stablized Zirconia Coatings Produced By Electrophoretic Deposition and Microwave Sintering**: *Wei Wang*[1]; Shiqiang Qian[1]; [1]Shanghai University of Science Engineering

The coatings of Y2O3-stabilized zirconia have been deposited on superalloy K17 substrates at room temperature by electrophoretic deposition technique followed by two different sintering methods. Scanning electron microscopy (SEM), X-ray diffraction (XRD) and indentation techniques have been employed to characterize morphological, structural and mechanical properties of the coatings. Finer and more uniform microstructures were observed in the microwave sintered coatings when compared to the conventional sintered samples. Nanohardness indentation tests of the coatings exposed (1500 W) to microwave have shown hardness of 4.3 GPa with elastic modulus of 172.7 GPa compared to the conventional sintered coatings of 3.1 GPa with elastic modulus of 83.5 GPa. For the conventional sintered coatings, the monoclinic phase was observed. The microwave sintered coatings of Y2O3-stabilized zirconia contain mainly cubic/tetragonal phases with some metastable phase present. Such coatings have potential in being used as thermal barrier coatings on superalloy substrates.

**11:30 AM Invited**
**Carrier Concentration Tuning and Enhanced Photoelectrochemical Response of Bandgap-Reduced Cu and Ga Co-Doped P-Type ZnO Films**: *Sudhakar Shet*[1]; Kwang-Soon Ahn[2]; Heli Wang[1]; Todd Deutsch[1]; Nuggehalli Ravindra[3]; Yanfa Yan[1]; John Turner[1]; Mowafak Al-Jassim[1]; [1]National Renewable Energy Laboratory; [2]Yeungnam University; [3]New Jersey Institute of Technology

The synthesis of p-type ZnO films with similar bandgaps but with varying carrier concentrations through co-doping of Cu and Ga is studied. The ZnO:(Cu,Ga) films were synthesized by RF magnetron sputtering in O2 gas ambient at room temperature and then annealed at 500C in air for 2 hours. We found that the bandgap reduction and p-type conductivity are caused by Cu incorporation. The tuning of carrier concentration is realized by varying the Ga concentration. It was found that the carrier concentration tuning does not significantly change the bandgap and crystallinity of the ZnO:Cu films. However, it can optimize the carrier concentration and thus dramatically enhance PEC response for the bandgap-reduced p-type ZnO thin films. Our studies suggest that carrier concentration tuning by acceptor-donor co-doping is an important approach to enhance the PEC performance of electrodes

**12:00 PM**
**Research on Nano Fe2O3 Film Coated on Surface of 3D-Meshwork SiC by Sol-Gel Method**: *Yu Liang*[1]; Wu Yanjun[1]; Ru Hongqiang[1]; Yue Xinyan[1]; Li Jingyang[1]; [1]Texture of Materials, Ministry of Education, College of Materials and Metallurgy, Northeastern University,

This paper represented the preparation process of nano ferric oxide thin film coated on three-dimensional meshwork silicon carbide by the sol-gel method, in which Fe(NO3)3•9H2O and CO(NH2)2 were used as raw material, water

as solvent. Concentration of Fe(NO3)3•9H2O and CO(NH2)2, temperature of hydrolyze, mechanism of desiccation, temperature of heat treatment were controlled. X-ray diffraction (XRD), scanning electronic microscope (SEM) were used to study the component, microstructure and phase constitution of the film. Infra-red sepectrometry (IR), differential scanning calorimetry (DSC) and thermogravimetry (TG) were used to study the change of the ferric oxide precursor. The investigation clearly showed that a-FeOOH sol solution in which concentration of Fe3+ was 0.167mol/L and be hydrolyzed in the order under 50°C and 80°C was perfect. With vacuum impregnation and step desiccation methods, and sintered at 480°C, the ideal densification nano ferric oxide thin film coated on three-dimensional meshwork silicon carbide can be prepared.

## Computational Thermodynamics and Kinetics: Diffusion and Defects

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
*Program Organizers:* Jeffrey Hoyt, McMaster University; Dallas Trinkle, Univeristy of Illinois at Urbana-Champaign

Monday AM Room: 308
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**8:30 AM Invited**
**Controlling Diffusion in Semiconductor Nanostructures by Size and Dimensionality**: *James Chelikowsky*[1]; [1]University of Texas

The ability to control the diffusion of dopants or impurities is a controlling factor in the functionalization of materials used in devices both at the macro- and nano-scales. At the nanoscale, manipulating diffusion of dopants is complicated by a number of factors such as the role of quantum confinement and the large surface to volume ratio. Here I will examine Li in Si nanowires as atoms with low atomic mass such as Li can be used as a carrier for energy storage with high specific energy capacity. Specifically, Li-ion batteries with specific energy capacity as high as 4200 mAh/g using Si nanowires as anodes have been achieved. Using ab initio calculations, we determine how the factors of size and dimensionality can be used achieve an optimal diffusion of Li atoms in Si nanowires.

**9:00 AM Invited**
**Compositional Point Defect Evaluation Using Diffusion Multiples**: *Ji-Cheng Zhao*[1]; Xuan Zheng[2]; David Cahill[2]; [1]The Ohio State University; [2]University of Illinois at Urbana-Champaign

Compositional point defects such as vacancies and anti-sites have a significant effect on the properties of alloys and intermetallic compounds. For instance, point defects in NiAl result in orders of magnitude change in diffusion coefficients. Two indirect ways to find the formation of compositional point defects in alloys and intermetallics using diffusion couples and diffusion multiples will be discussed. The first involves micron-scale thermal conductivity mapping over a wide range of compositions. A sharp change in thermal conductivity in a very narrow range of composition is indicative of compositional point defect formation. Thermal conductivity measurements can also be used to study the site preference / elemental substitution in intermetallic compounds. The second method looks at the shape of the diffusion profiles and the dependence of diffusivity on composition. Examples will be used to illustrate these methodologies. Both methods are sufficient, but not necessary tests of the compositional point defects.

**9:30 AM Invited**
**Diffusion in Alloys and Intercalation Compounds from First Principles**: *Anton Van der Ven*[1]; [1]University of Michigan

Diffusion in substitutional alloys and solids containing high concentrations of mobile interstitial elements is a complex kinetic process that depends on the nature of intrinsic defects, the energetically most favorable hop mechanisms and the degree of short and long-range order among the constituents of the solid. In this talk, I will describe how these factors can be rigorously accounted for in a first-principles prediction of diffusion coefficients in non-dilute alloys. The approach relies on the evaluation of Kubo-Green expressions, which provide the link between macroscopic diffusion coefficients and atomic trajectories sampled in kinetic Monte Carlo simulations. A first-principles description of the thermodynamics of short and long-range order in multi-component solids is achieved with the cluster expansion formalism. I will describe recent work on the prediction of diffusion coefficients in intermetallic compounds and Li ion battery electrode materials.

**10:00 AM**
**Ab Initio Modeling of Interstitial Diffusion in bcc Fe**: *Marcel Sluiter*[1]; [1]TU Delft

The role of diffusion of interstitial species such as carbon in bcc iron with minor additions of other common alloying elements such as aluminum, silicon, manganese, niobium and others are studied using ab initio techniques. It is shown that interactions between C and substitutional alloying elements in bcc Fe is not trivially guessed from carbon affinity as derived from carbide formation tendencies.Generally, kinetic Monte Carlo simulations show that substitutional elements reduce the carbon diffusivity.

**10:20 AM Break**

**10:30 AM Invited**
**First-principles Approach to Transition States of Diffusion**: *Zi-Kui Liu*[1]; [1]The Pennsylvania State University

In recent publications, we predicted self- and impurity diffusion coefficients through first-principles calculations based on the transition state theory [1] and a five-frequency model [2]. The free energy of vacancy formation and vacancy impurity binding is obtained through total energy and phonon calculations. Various jump frequencies are evaluated from phonon vibrational frequencies at the equilibrium and transition states when the vacancy is present. More recently, we proposed a new first-principles approach to treat the unstable vibrational mode of transition states [3]. It particularly allows one to determine the entropy of migration and the characteristic vibrational frequency, along with their temperature dependences. In this presentation, above results will be discussed. 1. Mantina et al., "First-principles calculation of self-diffusion coefficients," Phys. Rev. Lett., Vol.100, 2008, 215901. 2. Mantina et al., "First-Principles Impurity Diffusion Coefficients", Acta Mater., (accepted, May 2009)3. Mantina et al. "First-principles Approach to Transition States of Diffusion," submitted

**11:00 AM**
**Quantifying the Strength of Point Defect Based Hydrogen Traps in bcc and fcc Iron**: *William Counts*[1]; Chris Wolverton[1]; Ron Gibala[2]; [1]Northwestern University; [2]University of Michigan

Hydrogen embrittlement of iron and steels is a classic but still unresolved problem in metallurgy. While hydrogen can freely move through the Fe lattice, its diffusion is hindered by lattice imperfections. Experimentally quantifying the binding energy of hydrogen to these defects has proven to be difficult. Fortunately, computational tools are ideally suited to study defect trapping because it is possible to isolate individual traps. Density function theory was used to quantify the binding energy of hydrogen to a number of point defects in both bcc and fcc Fe. In bcc Fe, vacancies are the strongest hydrogen trap with a binding energy of 0.57 eV. The binding energies of common alloying elements are significantly less and range between -0.08 - 0.20 eV. Using a number of different anti-ferromagnetic configurations to model the paramagnetic state in fcc Fe, we found the hydrogen-vacancy binding energy to be between 0.20-0.36 eV.

**11:20 AM**
**Island Shape Controls Magic Size Effect for Heteroepitaxial Diffusion**: *Henry Wu*[1]; Dallas Trinkle[1]; [1]University of Illinois at Urbana-Champaign

Low temperature in situ STM studies of Cu islands on Ag(111) by Weaver at UIUC observed anomalously fast diffusion for specific sizes of small monolayer islands (magic size effect). To computationally study the dynamics of Cu island diffusion, we use ultrasoft pseudo-potential GGA calculations to optimize an embedded atom method (EAM) potential. The optimized EAM calculates diffusion barriers of Cu islands up to 14-atoms. Previous work by Hamilton for 1D and 2D heteroepitaxial island diffusion found a magic size effect associated with collective glide dislocation-mediated mechanism. For Cu/Ag(111) we find---in addition to collective glide---lower diffusion barriers for the reptation dislocation-mediated mechanism. The reptation mechanism is

limited by bond-breaking considerations that result from the 2D island shape. By grouping islands according to the number of [110] rows affected by the diffusion dislocation, the 2D diffusion barriers follow trends seen in the 1D magic size effect.

**11:40 AM**

**Effects of Spin Transition on Diffusion of $Fe^{2+}$ in Ferropericlase in Earth's Lower Mantle**: Saumitra Saha[1]; *Dane Morgan*[1]; Amy Bengtson[2]; [1]University of Wisconsin-Madison; [2]University of Michigan

Diffusion plays a fundamental role for many processes in Earth's mantle involving chemical, isotopic and advective mass transfer, homogenization of mantle materials, grain growth, electrical conductivity and communication between elements in core and mantle. Recent studies have established that iron undergoes an electronic spin transition in ferropericlase ((Mg,Fe)O) in the lower part of the mantle. However, due to the challenges of diffusion experiments at lower mantle pressures and temperatures, the affect of the spin transition of $Fe^{2+}$ on diffusion in ferropericlase is unknown. In the present study we combine first principles methods with the five-frequency statistical model to compute the $Fe^{2+}$ diffusivity. The results are compared with existing experimental diffusion data and changes in the diffusion rate are interpreted in terms of the changes in electronic structure with the spin transition. Finally, possible implications of the spin crossover for the lower mantle properties are discussed.

**12:00 PM**

**Molecular Dynamics Simulation of Self-Diffusion in bcc Metals**: *Mikhail Mendelev*[1]; [1]Ames Laboratory

It is widely accepted that the vacancy mechanism governs the self-diffusion in fcc metals. We used molecular dynamics (MD) simulation to check whether this is the case for bcc metals. We determined the point defect formation and migration energies and found that both interstitial and vacancy formation energies strongly depend on temperature and the sum of the interstitial formation and migration energies can be smaller than the same quantity for vacancies. Next we used MD simulation to determine the equilibrium point defect concentration and found that at high temperatures the interstitial concentration can be comparable with the vacancy concentration. Finally we calculated diffusivities in both vacancy and interstitial mechanisms and found that in bcc Zr, vacancy and interstitial mechanisms give about the same contribution in the self-diffusivity.Work at the Ames Laboratory was supported by the Department of Energy, Office of Basic Energy Sciences, under Contract No. DE-AC02-07CH11358.

## Cost-Affordable Titanium III: Overview and Low Cost Processing

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
*Program Organizers:* M. Ashraf Imam, Naval Research Lab; F. H. (Sam) Froes, University of Idaho; Kevin Dring, Norsk Titanium

Monday AM Room: 618
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Henry Rack, Clemson University; James Sears, Quad City Manufacturing Laboratory

**8:30 AM**

**Cost Affordable Developments in Titanium Technology and Applications**: *F. H. (Sam) Froes*[1]; M. Ashraf Imam[2]; [1]Institute for Materials and Advanced Processes; [2]Naval Research Lab

Titanium is the "wonder" metal, which makes sense as the material of choice for a wide variety of applications. However, because of its relatively high price- a result of extraction and processing costs- it is used basically only when it is the only choice; with the caveat that titanium has a bright "image" which can lead to use even when the economics are unfavorable. The major thrust in the area of titanium development has been aimed at achieving cost reduction rather than the developing alloys with enhanced properties. This paper will overview the potential areas, which are amenable to the cost reduction for developments in titanium technology and applications.

**8:55 AM**

**The FFC-Cambridge Process for Titanium Metal Winning**: *Carsten Schwandt*[1]; Derek Fray[1]; Gregory Doughty[1]; [1]University of Cambridge

The FFC-Cambridge Process is a molten salt electro-deoxidation method that was invented at the University of Cambridge one decade ago. It is a generic technology that allows direct conversion of metal oxides into the corresponding metals through cathodic polarisation of the oxide in molten calcium chloride. The process has wide applicability, and numerous studies on metals, semi-metals, alloys and intermetallics have since been performed at Cambridge and worldwide. The electro-winning of titanium metal is a particularly rewarding target because of the disadvantages of present extraction methods. This presentation summarises the research performed on the FFC-Cambridge Process with a focus on electro-winning titanium metal from titanium dioxide. Topics addressed encompass the investigation of the key process parameters, the identification of the reaction pathway, and the derivation of an optimised set of process parameters. The presentation also discusses some aspects of technology transfer and development activities undertaken to date.

**9:20 AM**

**Direct Electrochemical Reduction of Titanium Dioxide in Molten Salts**: *Kevin Dring*[1]; [1]Norsk Titanium

Electrochemical methods and electrowinning of titanium have been promoted, unsuccessfully, for many years as a replacement for the current Kroll process, which has not achieved the same economies of scale as other light metals (eg. Al, Mg) have. One such method is the direct electrochemical reduction of titanium dioxide, which encompasses a range of processes involving the electrolysis of a molten salt and the subsequent reduction of titanium via the metal of the salt cation. Norsk Titanium AS, in a collaboration with Norsk Hydro and NTNU through a Norwegian Research Council grant, has been actively involved in the development of such a process and the focus of this presentation is to give a summary of the research activities. Particular emphasis will be given to the fundamental reactions underlying the process.

**9:45 AM**

**The Production of Ti Alloy Powder from Chloride Precursors**: *James Withers*[1]; J. Laughlin[1]; Y. Elkadi[1]; J. DeSilva[1]; R. Loutfy[1]; [1]MER Corporation

It has long been a goal to produce Ti Alloy powder directly to eliminate the standard processing of melting sponge, alloying, producing a billet/ingot and then reducing to powder by one of several techniques. The batch Kroll process where reaction occurs at the reactor wall interface from $TiCl_4$ vapor and molten magnesium, limits the potential to directly form alloys. Any batch processing has the limitation of alloy compositional control from batch to batch. A unique continuous processing approach permits the gaseous mixing of chloride precursors with metallothermic reduction that directly produces an alloy powder in a size that is useable for standard powder metallurgy. Discussion will include producing Ti-6Al-4V and other alloy powder.

**10:10 AM Break**

**10:25 AM**

**Quantitative X-Ray Synchrotron Analysis of the FFC Cambridge Process**: *Rohit Bhagat*[1]; David Dye[2]; Ben Jackson[2]; Seema Raghunathan[2]; Douglas Inman[2]; Richard Dashwood[1]; [1]The University of Warwick; [2]Imperial College London

This paper demonstrates how in-situ synchrotron analysis has been used to obtain the first unequivocal description of the reduction pathway for the electrochemical production of titanium via the FFC Cambridge process. In previous studies of this process, phase identification was carried out ex-situ and this has resulted in erroneous and conflicting reduction pathways being proposed. By performing in-situ synchrotron, it was found that the reduction pathway proceeded sequentially through TiO2, sub-stoichiometric TiO2 phases, Ti2O3, cubic TiO and hexagonal titanium. CaTiO3 formed from the sub-stoichiometric TiO2 phases. CaTi2O4 formed by a comproportionation reaction in the inner layers of the pellet. CaO was also observed, indicating that for some interval the electrolyte was locally saturated with oxide ions. TiC formed at the latter stages of reduction on the pellet surface. The occurrence of the individual phases was dependent on location, with the transformations shifting to longer times away from the surface.

**10:50 AM**

**New Methods for Low-Cost Titanium Production**: *Ana Maria Martinez*[1]; Karen Osen[1]; Egil Skybakmoen[1]; Ole Kjos[2]; Geir Martin Haarberg[2]; Kevin Dring[3]; [1]SINTEF; [2]NTNU; [3]Norsk Titanium AS

Metallothermic reduction is, at the moment, the only commercially available process for the industrial production of titanium sponge (Kroll process). Although the apparatus and procedures have improved over the past 50 years, it is still difficult to recover the waste heat from the individual steps and the total efficiency of titanium production is worse than that in continuous steelmaking. Therefore, alternative processes to produce low-cost titanium are desirable. These methods should possess the same requisites as the Kroll process, in terms of metal quality, as well as continuous operation and energy effective.The present work deals with the investigation of a titanium production method that uses TiO2 enriched titania slag as raw material. The process involves two steps: i) carbothermal reduction of the slag to form titanium oxicarbide (TiOxCy) powder; and ii) electrolysis in molten chlorides using a TiOxCy consumable anodes.

**11:15 AM**

**A Continuous Process to Produce Titanium Utilizing Metallothermic Chemistry**: *James Withers*[1]; J. Laughlin[1]; Y. Elkadi[1]; J. DeSilva[1]; R. Loutfy[1]; [1]MER Corporation

In the standard Kroll process reaction between the $TiCl_4$ and Mg is at the reactor wall interface that limits the potential to design a continuous process. Many alternatives have been investigated over the past 70 years to engineer a continuous process utilizing metallothermic reduction of $TiCl_4$. Approaches utilizing burner type architectures for continuous processing result in unacceptable very fine Ti powder. A unique process that operates continuously and produces controlled size powder that can be directly utilized in standard powder metallurgy, rapid manufacturing, or substituted for sponge will be discussed.

**11:40 AM**

**The Electrolytic Production of Ti from a $TiO_2$ Feed (The DARPA Sponsored Program)**: *James Withers*[1]; J. Laughlin[1]; Y. Elkadi[1]; J. DeSilva[1]; R. Loutfy[1]; [1]MER Corporation

DARPA instituted an Initiative in Titanium in 2003 to produce titanium, alternatively to the Kroll process, in a billet form for under $4/lb. This DARPA sponsored program has gone into Phase II consisting of utilizing ore/$TiO_2$ as a feed. The $TiO_2$ is carbothermically reduced to a suboxide-carbide (Ti:O:C) which is used anodically to resupply the titanium content in an electrolysis process that deposits titanium in a powder morphology. The deposited powder is uniquely stripped from the cathodes and harvested in a separate stream that permits continuous electrolytic processing to produce titanium at an estimated cost about ½ the Kroll process. Oxygen contents less than 500 ppm are achievable with particle sizes in the desired range for powder metallurgy applications. The process has been demonstrated on a continuous basis and is in the stage of scaling-up to 500 lbs/day. Approved for Public Release, Distribution Unlimited

**12:05 PM**

**The FFC Cambridge Process for Production of Low Cost Titanium and Titanium Powders**: *Mark Bertolini*[1]; Lee Shaw[1]; Lucy England[1]; Kartik Rao[1]; James Dean[1]; James Collins[1]; [1]Metalysis

The current status and recent advancements in the use of the FFC Cambridge process for the production of low cost titanium and titanium powders is presented. This will include an overview of the process, current and future process equipment and recent results in terms of chemistry, structure and properties of powder and consolidated product. The future direction and activities for the FFC Cambridge process will also be briefly discussed.

## Failure of Small-Scale Structures: Nanowire Behavior

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

*Program Organizers:* Marian Kennedy, Clemson Univeristy; Brad Boyce, Sandia National Laboratory; Reinhold Dauskardt, Stanford; Zhiwei Shan, Hysitron Inc

Monday AM Room: 206
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Brad Boyce, Sandia National Laboratories; Molly Kennedy, Clemson University

**8:30 AM Invited**

**Insights from Nanomechanical Testing: Nanoengineering Surfaces, Thin Films, and Nanowires**: *Christopher Muhlstein*[1]; [1]The Pennsylvania State University

Our ability to engineer nanomaterials for mechanical applications hinges on a fundamental understanding of how they accumulate damage and fail. In this presentation we will review a series of experiments conducted on surfaces, thin films, and nanofilaments that challenge generally accepted nanoengineering strategies. Specifically, nanoindentation and fatigue crack growth experiments conducted on nanograined platinum films show that the films are extremely susceptible to time and cycle-dependent degradation. In contrast, tensile testing results demonstrate that individual nanowires can appear to have anomalous deformation behavior that is an artifact of the testing methodology.

**8:55 AM Invited**

**Mechanical Behavior of Au Nanowires**: *Cynthia Volkert*[1]; B. Roos[1]; B. Kapelle[1]; G. Richter[2]; A. Sedlmayr[3]; D.S. Gianola[4]; R. Mönig[3]; [1]Institute for Materials Physics, University of Göttingen; [2]Max Planck Institute for Metals Research; [3]Institute for Materials Research-II, Forschungszentrum Karlsruhe; [4]Formally at the Institute for Materials Research-II, Forschungszentrum Karlsruhe, currently at the Department of Materials Science and Engineering, University of Pennsylvania

The mechanical behavior of single crystal Au nanowires with diameters between 40 and 100 nm have been investigated by in-situ tensile loading in the TEM and by instrumented testing using a nanoindenter. The nanowires are grown by physical vapour deposition onto W substrates and are initially dislocation-free. Preliminary observations of the nanowires in the TEM reveal that numerous dislocations are nucleated uniformly along the length of the nanowire during tensile loading. These dislocations remain in the wire, possibly influenced by a surface Pt layer formed during transfer in the SEM, before they exit the nanowire on further loading. The wire eventually fails by ductile fracture. Neither the site of fracture nor the dislocation structure in the fracture area show any distinguishing features. Tensile tests with a nanoindenter and instrumented tensile testing in an SEM show that the strengths of the Au nanowires are in good agreement with those measured in nanoporous Au ligaments and in Au micro-compression columns of the same diameters, and support the widely observed trend of increasing strength with decreasing size.

**9:20 AM**

**In-Situ Tensile Deformation of Silver Nanowires**: *Junhang Luo*[1]; Jian Yu Huang[2]; Catherine Murphy[3]; Scott Mao[1]; [1]University of Pittsburgh; [2]Sandia National Laboratories; [3]University of South Carolina

Using a unique transmission electron microscopy (TEM) - scanning tunneling microscopy (STM) platform, in-situ tensile tests combined with high resolution TEM (HRTEM) observations have been performed successfully on silver nanowires. A uniform plastic elongation without necking was achieved with an elongation more than 19% in the bicrystalline Ag nanowires. In-Situ HRTEM observations and fast Fourier transform (FFT) analysis showed that a lot of stacking faults were formed during the tensile test to accommodate the plastic deformation. Interestingly, the stacking faults are not induced by Shockley partial dislocations but by the frank loops formation and expansion.

**9:35 AM**
**In-Situ TEM Studies of Nanomechanics and Fracture in Nanowires and Nanotubes**: *Reza Shahbazian-Yassar*[1]; Hessam Ghasemi[1]; Anahita Pakzad[1]; Kasra Momeni[1]; Anjana Ashtana[1]; Yoke Yap[1]; [1]Michigan Technological University

One-dimensional nanomaterials including nanotubes, nanowires, and nanofibers are building blocks for constructing various complex nanodevices. In this work, deformation of individual nanotubes and nanowires will be performed inside a high-resolution transmission electron microscope (TEM) using a piezo-driven atomic force microscope (AFM) and scanning tunneling microscope (STM)–TEM holder. The electrical and mechanical properties of individual nanotubes/nanofibers are obtained from the experimentally recorded I-V and force-displacement curves. Failure and deformation of various nanostructures including ZnO nanowires, BN nanotubes, carbon nanotubes, and cellulose nanocrystals show distinct behavior.

**9:50 AM Break**

**10:05 AM Invited**
**Fracture and Deformation in Metallic Nanowires**: *Scott Mao*[1]; A. Cao[2]; Y. Wei[2]; [1]University of Pittsburgh; [2]Institute of Mechanics

The mechanical behavior of bulk metals is usually characterized as smooth continuous plastic flow following by yielding. Here we show, by using molecular dynamics simulations and in-situ experiment that the mechanical deformation behaviors of single-crystalline nickel and copper nanowires are quite different from their bulk counterparts. Correlation between the obtained stress-strain curves and the visualized defect evolution during deformation processes clearly demonstrates that a sequence of complex dislocation slip processes results in dislocation starvation, involving dislocation nucleation, propagation and finally escaping from the wire system, so that the wires deformed elastically until new dislocation generated. This alternating starvation of dislocations is unique in small-scale structures. Furthermore, the magnitude of yield stress of these nanowires is strongly dependent of the wire size.

**10:30 AM Invited**
**In-Situ Atomic Scale Nanomechanics Revealed by a TEM-SPM Platform**: *Jianyu Huang*[1]; Junhang Luo[2]; He Zheng[2]; Scott Mao[2]; Nan Li[3]; Jian Wang[3]; Xinghang Zhang[4]; Armit Misra[3]; Yang Lu[5]; Jun Lou[5]; Erik Bitzek[6]; Ju Li[6]; [1]Sandia National Laboratories; [2]University of Pittsburgh; [3]Los Alamos National Laboratories; [4]Texas A&M University; [5]Rice University; [6]University of Pennsylvania

By integrating scanning tunneling microscopy (STM) probes, atomic force microscopy (AFM) probes, and nanoindentors into a transmission electron microscope (TEM), novel deformation and failure mechanisms of individual carbon nanotubes, nanowires, and graphene were revealed at an atomic-scale. In NaCl nanowires, unusual fast diffusion rather than dislocation mediated superplasticity was observed. Silica glass nanowires were ductile at room temperature, with surprisingly large tensile plastic elongations. These ductile glass nanowires also possess high flow strengths. The unexpected ductility is due to the development of a surface affected zone in the nanowires, which enhances ductility. Detwinning, the reverse process of twinning, is an important deformation mechanism in face-centered-cubic metals. In graphene, fractal sublimation patterns and sp2-edge reconstructions were observed, indicating that bilayer rather than monolayer graphene is more realistic candidate for device applications. Future directions of applying the TEM-SPM techniques to studying batteries, thermal and thermoelectric energy-related materials will be discussed.

**10:55 AM**
**An In Situ Scanning Electron Microscopy Study of Size Dependent Mechanical Behaviors of Metallic Nanowires**: *Cheng Peng*[1]; Yang Lu[1]; Yogi Ganesan[1]; Yongjie Zhan[1]; Jun Lou[1]; [1]Rice University

Metallic nanowires are of great technological importance due to their current and potential applications in miniaturized electronic, optical, thermal and electromechanical systems. This talk presents some of our recent efforts to study the size dependent mechanical behavior of metallic nanowires. We have developed a simple micro-device that allows in situ quantitative mechanical characterization of metallic nanowires, in scanning electron microscope chamber equipped with a quantitative nanoindenter. The unique design of this device makes it possible to convert compression from nanoindentation to uni-axial tension at the sample stages. Finite element analysis is employed to model the device behavior under mechanical loading and compared with experiments. Ni, Cu and Au nanowires with different diameters ranging from tens of nanometers to hundreds of nanometers were fabricated by template-assisted electro-chemical deposition and hydrothermal synthesis. Finally, main results on size effects in deformation and fracture behavior of Ni, Cu and Au nanowires will be discussed.

**11:10 AM Invited**
**Deformation Mechanisms in Quasi-1D Nanostructures: *In Situ* Observations and Measurements during Tensile Testing in Electron Microscopes**: *Daniel Gianola*[1]; [1]University of Pennsylvania

The mechanical behavior of single crystalline metals has been known to be size dependent for decades, but the details of the underlying deformation mechanisms have seen only recent attention. Escape to free surfaces and nucleation of new dislocations are expected to trump interactions between dislocations, precluding the operation of bulk hardening mechanisms. However, the real strength and rate controlling mechanisms at sub-micrometer length scales are still elusive.In the present work, we describe quantitative *in situ* tensile experiments on quasi-1D nanostructures in a dual-beam SEM and FIB. Complementary *in situ* TEM studies of dislocation multiplication and shearing processes in sub-micrometer Al fibers were also conducted. The detailed operation and nature of the sources have been systematically analyzed and compared to their crystal dimensions. The influence of pre-existing defects and flaws in these nanostructures, which is critically linked to the materials synthesis route, is discussed.

**11:35 AM Invited**
**Statistical Effects on Material Strength at Small Length Scales**: Shyam Keralavarma[1]; *Ahmed Benzerga*[1]; [1]Texas A&M University

One of the characteristic features of available experimental data for the mechanical strength of materials at small length scales is the increased scatter resulting from the statistical nature of discrete dislocation events. From a design point of view, it is important to characterize this observed feature of material behavior in order to improve the reliability of the mechanical components at the nano scale. In this work, we employ two-dimensional discrete dislocation dynamics (DD) simulations of the homogeneous deformation of single crystals under compressive loads to study the statistical effects on material response as a function of size. Some examples of non-homogeneous deformation such as discrete dislocation effects on micro-void growth under plane strain conditions will also be considered. The results are compared to available experimental results, such as the micro-pillar compression experiments.

## General Abstracts: Materials Processing and Manufacturing Division: Welding

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Global Innovations Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Phase Transformations Committee, TMS: Powder Materials Committee, TMS: Process Technology and Modeling Committee, TMS: Shaping and Forming Committee, TMS: Solidification Committee, TMS: Surface Engineering Committee
*Program Organizers:* Thomas Bieler, Michigan State University; Corbett Battaile, Sandia National Laboratories

Monday AM Room: 611
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**8:30 AM**
**High Brightness Nd:YAG Laser Welding of Aluminum 5754**: *Jyotirmoy Mazumder*[1]; Leslie Pipe[1]; Yi Liu[1]; David Roessler[1]; [1]University of Michigan

Bead-on-plate keyhole welds were made in aluminum alloy Al-5754, (a popular alloy in auto industry for weight reduction) using a pumped solid-state diode-laser with 500W average power and high-brightness, operating at a 20% duty cycle and argon as the shielding gas. The welding parameters included laser beam pulse width, repetition rate, focusing lens optics and traverse speed. Besides penetration depth data, spectral lines from Al I transitions were examined with a spectrometer and an Intensity Charge Coupled Device. At laser

power of 400 W, full penetration of 3-mm sheets was possible with a traverse speed-focal length combination of 0.85 cm/s and 150-mm or 0.21 cm/s and 200-mm. Emission spectroscopy revealed the temperature of the vapor plume between 8,000-10,000 K at the workpiece surface and increasing over the first millimeter of height. Plume temperatures seem to be higher with longer laser pulses, shorter focal lengths and higher welding traverse speeds.

**8:50 AM**

**Robotic Welding of Large Floor Panels Made of Light Aluminum Extrusions**: *Michel Guillot*[1]; [1]Laval University

Gaz metal arc welding is commonly used for joining large assemblies and is increasingly preferred for welding thin wall components despite some limitations. In this paper, several parameters of the GMAW process and robot motion are investigated for joining AL6061-T6 and AL6063-T6 extrusions of floor panels. Based on welding tests carried out on flat stock samples, the effect of several parameters like GMAW and robot settings, joint preparation, wire diameters, alloys and feed are studied and optimized for weld quality as well as for higher welding speed. In the second and main part of the paper the best settings are implemented for joining complex extrusions of a floor panel used typically in a 53ft semi-trailer. The geometric and positioning errors of the components mounted in the fixture and the distortion after welding several 2.4m long extrusions on both sides are studied. The weld quality is tested at different locations in the floor panel. Several improvements are experimented to optimize even further the welding performances.

**9:10 AM**

**Effects of Novel Processing Techniques on the Fatigue Crack Growth Behavior of 6061 Alloys**: *Brendan Chenelle*[1]; Christopher Lammi[1]; Diana Lados[1]; [1]Worcester Polytechnic Institute

Friction stir welding and coldspray are solid-state processes that could be beneficially used for joining and repair in transportation and defense applications. Understanding fatigue crack propagation is necessary to use these processes in critical structural applications. In this study, tensile properties and fatigue crack growth mechanisms in friction stir welded and coldspray 6061 alloys were investigated. Fatigue crack growth testing of the base and processed materials was conducted on compact tension specimens in ambient conditions using a constant stress ratio, R=0.1. Tests parallel and normal to the friction stir weld traverse direction and to the coldspray consolidation direction were conducted. The microstructures after processing were characterized and the resulting residual stresses were examined using fracture mechanics techniques. Individual and combined effects of residual stresses, microstructural changes, and reinforcements of the processed regions were related to the fatigue crack growth response of the materials and will be presented and discussed.

**9:30 AM**

**Numerical and Experimental Analyses for Effect of Welding Speeds on Cooling Rates in Manual Metal Arc Welding(MMAW)**: Muna Abbass[1]; *Jalal M. Jalil*[1]; Abbas Sh. Alwan[1]; [1]University of Technology, Baghdad

Abstract Manual Metal Arc Welding (MMAW) is carried out for low carbon steel (AISI 1015) for plate of thickness 8mm. Experimentally, cooling rates are determined for the fusion zone at different welding speeds(1.7, 2.3 and 3.2) mm/s with constant welding current at (100 Amp). Numerical analysis by using the Control Volume Method (CVM), applied to three-dimensional heat transfer model to determine the cooling rate in fusion zone. Cooling rates models are helping in prediction the microstructure and microhardness distribution in weld metal and heat affected zone. The comparison of cooling curves between numerical and experimental work have a good agreement, so that deviation was in range (6□-21□ (which is confirming the capability and reliability of the proposed numerical heat transfer model in manual metal arc welding. The best result for cooling rates when applying mathematical model is at welding speed 1.7mm/s.

**9:50 AM**

**The Effect of Welding Speed on the Crystallographic Texture Observed in Friction Stir Welding of Near-Alpha Titanium**: *Richard Fonda*[1]; Keith Knipling[1]; [1]Naval Research Laboratory

In a series of experiments performed at different welding speeds, friction stir welds in a near-a Ti-5111 titanium alloy exhibit different crystallographic textures. The crystallographic orientations produced at slower weld speeds, which allow more heat to accumulate in the weld, indicate that the weld-induced deformation occurs primarily while the material is in the low-temperature a phase. The faster, cooler welds exhibit a texture indicative of transformation in the high-temperature ß phase. This apparent contradiction indicates that friction stir welds of this titanium alloy are influenced less by the heat generated during welding than by the speed at which the rotating tool is retreating from the deposited weld. The accelerated cooling at faster weld speeds quenches-in the initial high-temperature deformation structure, while slower weld speeds allow for a more efficient transfer of the stirring deformation into the cooler material behind the tool.

**10:10 AM Break**

**10:30 AM**

**Spot Welding of Automotive Steels and Light Metals by Friction Bit Joining**: *Michael Miles*[1]; K. Kohkonen[1]; Zhili Feng[2]; [1]BYU; [2]Oak Ridge National Lab

Resistance spot welding (RSW) of steels and light metals in automotive manufacturing is difficult, because of incompatibility of these alloys during fusion. Self piercing riveting (SPR) can be done to join dissimilar alloys, but if the strength of the steel is very high this is difficult. It can also be difficult to form magnesium sheets around the rivet using SPR. A new process, called friction bit joining (FBR), was used to weld several high strength steels and light metals, where very good joint strength was obtained, even when joining a very hard alloy like DP 980 to a very soft alloy like AA 5754. Joint properties and microstructures will be presented and compared to current technologies like RSW and SPR.

**10:50 AM**

**Ultrasonic Welding a Novel Approach to Friction Spot Welding Dissimilar Aluminum to Steel Automotive Sheets**: *Farid Haddadi*[1]; Philip Prangnell[1]; Dimitrios Bakavos[1]; [1]The University of Manchester

High power Ultrasonic Spot Welding (USW) has recently been developed as an alternative technique for friction joining aluminum automotive panels. USW can produce high quality joints in gauges up to 3 mm, with a lower energy requirement than resistance spot welding and a shorter weld cycle than FSSW. However, to date USW has not been studied for applications in joining dissimilar automotive materials, where the rapid weld cycle should be beneficial in preventing bond line embrittlment.In the present research, the optimization of the USW technique has been studied for joining aluminum to steel. Effects of different welding parameters, such as pressure and weld energy, on weld quality was assessed for two types of galvanized and un-galvanized steel. The Mechanical properties of the welds were examined and correlated to the weld parameters and interface microstructures. Results are also presented on the defects present and stages of bond formation.

**11:10 AM**

**Computational Welding Mechanics: Hardening Models in Welding Simulation**: *Amir Masoud Akbari Pazooki*[1]; [1]TU Delft

Dual phase steel is used in automotive industries because of good strength and formability. Major problems in the welding process for this type of steel are residual stress and distortion. FEM is a very straightforward strategy for prediction of residual stress and distortion in welding engineering nowadays. Many models have been developed for prediction of residual stress and distortion in an accurate way. Most of these models use an isotropic hardening model. This paper presents isotropic, chaboche and kinematic hardening models in simulation of welding process for DP600 steel. The predicted results have been compared with the experimental measurements. It is concluded that different hardening models predict longitudinal residual stresses dissimilarly while the maximum distortion predictions are very close.

**11:30 AM**

**Thermo-Mechanical-Metallurgical Simulation of DP600 Steel during Welding**: *Amir Masoud Akbari Pazooki*[1]; [1]TU Delft

In this paper a 2D thermo-mechanical-metallurgical simulation of DP600 steel plate during GTAW process is presented. The first part of the paper deals with the thermal analysis and then in the second part the microstructural evaluation during heating and cooling cycles (in welding process) is shown. Finally the thermal and microstructural results are used as input files in mechanical analysis. The predicted results of residual stress and distortion during welding of this type of steel are compared with experiments.

**11:50 AM**

**Correlation between Shoulder Flow Zone Quality and Material Flow Quantity during Friction Stir Welding of Thick Aluminum Section Using Scroll Shoulder Tool**: *David Yan*[1]; Zhan Chen[1]; Guy Littlefair[1]; [1]AUT University

Scroll tool offers advantages of eliminating the tilted tool axis and performing non-linear thick section FSW with a simple machine. However, the correlation between its weld quality and material flow quantity remains unrevealed. A novel scroll tool was used to FSW thick aluminium plates at a range of welding parameters. These were followed by quantifying the mass of pick up material (PUM), and then evaluating the effect of welding parameters and PUM on the shoulder flow zone formation and weld quality. It was found that there is a positive linear relationship between the mass of PUM and shoulder flow zone weld quality. In order to obtain a defect-free weld produced by scroll tool, the scroll groove not only needs to be fully filled up with PUM before tool starts to travel, but also needs to be filled in with a sufficient amount of PUM per tool revolution during tool travelling.

**12:10 PM**

**Distortion Assessment of a Direct Cast Uranium - 6 wt. % Niobium Cylinder**: *Hunter Swenson*[1]; Rob Aikin[1]; [1]Los Alamos National Laboratory

A homogenization and quenching cycle generates complex thermal histories where components experience thermal and phase transformation-induced volume changes. The residual stress profile and resulting geometric changes are established by the interaction and timing of these competing factors. This work details the process history, material characterization, and distortion analysis of vacuum induction melted and direct cast U-6 wt. % Nb component. The parts evaluated are a 13 kg hollow cylinder (tube) with a 120 mm diameter, an 8.5 mm wall thickness and a length of 250 mm. The cylinders were homogenized in a vacuum furnace 1000°C, cooled and stabilized to 850°C, and then quenched in cold oil bath. The high temperature gamma structure is converted to an alpha" martensite during the quench. Dimensional data obtained from a coordinate measuring machine allowed for direct geometric comparison before and after heat treatment. Microstructure characterization was performed at various locations on the cylinder.

## General Abstracts: Structural Materials Division: Materials Characterization and Shock Loading

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Composite Materials Committee, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee, TMS: Refractory Metals Committee, TMS: Titanium Committee
*Program Organizers:* Eric Ott, GE Aviation; Robert Hanrahan, National Nuclear Security Administration; Judith Schneider, Mississippi State University

Monday AM Room: 608
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**8:30 AM**

**Characteristics of High Interstitially Alloyed 18Cr-10Mn Stainless Steels**: *Chang-Seok Oh*[1]; Tae-Ho Lee[1]; Heon-Young Ha[1]; Sung-Joon Kim[1]; [1]Korea Institute of Materials Science

Some results of characterizations on Cr-Mn stainless steels with combined addition of carbon and nitrogen are presented, where high nitrogen alloyed steels having same compositions of Cr and Mn were chosen for reference. High nitrogen alloyed steels (HNS) of 18Cr-10Mn-(0.4-0.7)N and high interstitially alloyed steels (HIA) of 18Cr-10Mn-0.38C-0.38N and 18Cr-10Mn-0.42N-0.15C were manufactured by a VIM process under the nitrogen atmosphere. Regardless of kind of interstitial elements, tensile and yield strengths of annealed specimens were increased with increasing amount of interstitial elements . The deformed microstructures of HNS alloys were changed from planar dislocation glide, strain induced martensitic transformation and to deformation twinning at higher strain, whereas no deformation induced transformation was not observed in HIA alloys. The electrochemical polarization response in chloride solution revealed that simultaneous alloying of carbon and nitrogen enhanced pitting corrosion resistance, which was confirmed by galvanostatic reduction test, Mott-Schottky analysis and XPS measurement.

**8:50 AM**

**Research on the Thermal Plasticity of the 18Mn-18Cr-0.77N-2Mo High Nitrogen Austenitic Stainless Steel**: *Li Wanming*[1]; Li huabing[1]; Jiang Zhouhua[1]; [1]Northeastern University

Hot compression tests of the 18Mn-18Cr-0.73N-2Mo high nitrogen austenitic stainless steel were performed on a GLEEBLE-2000 thermomechanical simulator under different deformation temperatures and strain rates. The microstructure of the compression samples were also observed by optical microscope. The model of the resistance to deformation was built, and the recrystallization activation energy, stress exponent and other parameters were obtained through analyzing the experimental data. The dynamic recrystallization model was established, and the distribution map of the dynamic recrystallization critical condition was given. The microstructure of the compression samples show that there are equiaxed grains on the edge of the samples and recrystallization grains in the core. Under same strain rate, the dynamic recrystallization becomes more sufficient under higher deformation temperature. But the grain coarsens and the plastic plasticity decreases when the temperature is above 1150°C. The recrystallization grain size is bigger and the amount becomes more under lower strain rate.

**9:10 AM**

**An Overview of the Microstructures of High-Strength Two-Phase near-Equi-Atomic FeNiMnAl Alloys**: *I. Baker*[1]; Y. Liao[1]; X. Wu[1]; H. Wu[1]; M.K. Miller[2]; K.F. Russell[2]; P.R. Munroe[3]; [1]Dartmouth College; [2]Oak Ridge National Laboratory; [3]University of New South Wales

This presentation will discuss the microstructures of several high strength alloys that have been discovered in the Fe-Ni-Mn-Al alloy system at near equi-atomic compositions. The microstructures of the alloys have been characterized using a combination of transmission electron microscopy, including X-ray mapping and convergent beam electron diffraction, and atom probe tomography. The highest strength alloys appear to have formed by spinodal decomposition and have very fine (10-90 nm wavelength) microstructures consisting of b.c.c. and B2 or L21 phases. Several alloys in this system that formed by eutectic decomposition still have very fine microstructures (0.1-1 μm) but consist of f.c.c. and B2 phases. The latter alloys can show significant tensile ductility. Research supported by NSF grants DMR-0505774 and DMR-0905229, DOE Award #DE-FG02-07ER46392 and the ORNL SHaRE User Facility which is sponsored by the Scientific User Facilities Division, Office of Basic Energy Science, U.S. Department of Energy.

**9:30 AM**

**Quantitative Measurement of Crack Initiation and Propagation in High Explosives and Mocks**: *Carl Cady*[1]; Cheng Liu[1]; Philip Rae[1]; Manuel Lovato[1]; [1]Los Alamos National Laboratory

Cracking is the most dominating mechanical failure mechanism in high explosives and cracks could affect both safety and performance of high explosive materials. However, one of the major challenge in studying fracture and failure of high explosives and their mock materials, is that physical cracks can exist that are not distinguishable by eye before they have grown large enough. A technique was developed to quantitatively describe macroscopic cracks, both their location and extent, in heterogeneous high explosive and mock material. By combining such a technique with the displacement field measurement using digital image correlation (DIC), we can describe, in detail, quantities that characterize material fracture process, like the crack-opening displacement and crack-opening angle. Results of this investigation will provide insights and validation data for the development of material failure models. In this presentation, properties like damage nucleation, crack initiation and extension, and crack-opening displacement will be quantitatively described.

**9:50 AM**

**Was There a Bomb on Mattei's Aircraft?**: *Donato Firrao*[1]; Graziano Ubertalli[1]; [1]Politecnico di Torino

Enrico Mattei, the President of the Italian oil conglomerate, was about to land in Milan Linate Airport, when his airplane crashed on the ground due to

a then unexplained accident. The investigation, reopened more than 30 years later, implied complete re-examining of the theories on macroscopic and lattice deformations under high velocity waves emanating from a small charge explosion. At the microstructural level multiple slip bands or mechanical twins, induced from the pressure wave caused by an explosion, can be observed, depending on the type of metal, the pressure and the strain rate. Different situations regarding stainless steels, aluminium, copper and gold alloys are analysed. Calculations and results of field explosion experiments are incorporated into the evaluation of microstructural signs possibly induced on metal targets by an unknown explosive event. Revisited theories were applied to this forensic case, reaching the conclusion that the aircraft had fallen following an …

**10:10 AM Break**

**10:20 AM**

**The Response of Aluminium Alloys to Shock Loading**: *Jeremy Millett*[1]; Neil Bourne[1]; Ming Chu[2]; Ian Jones[2]; George Gray III[3]; Gareth Appleby-Thomas[4]; [1]AWE; [2]University of Birmingham; [3]Los Alamos National Laboratory; [4]Cranfield University

Like most materials, the response of aluminium alloys to shock loading is strongly influenced by a number of factors such as chemistry, microstructure and processing. In this work, we examine the response of the aluminium alloys, 6061 and 5083, in particular, the evolution of lateral stress and shear strength behind the shock front. In 6061, the alloy is investigated in the solution treated (T0) and fully aged conditions (T6). Stress rise times and equilibration behind the shock front are observed to be quicker in T0 than T6, and has been attributed to fine Mg2Si particles inhibiting dislocation generation and motion in T6. In 5083, strength has also been observed to be relatively stable behind the shock front. In this case, the alloy was received in the H32 state, with the material previously worked by ca. 20%. Full one-dimensional recovery experiments have also been performed, confirming the above observations.

**10:40 AM**

**Deformation Behavior of U-6wt%Nb Following Shock Loading**: *Adam Farrow*[1]; Heather Volz[1]; George Gray III[1]; Ellen Cerreta[1]; Donald Brown[1]; Carl Cady[1]; Mike Lopez[1]; Ann Kelly[1]; Pallas Papin[1]; [1]Los Alamos National Laboratory

Uranium-6wt%Niobium samples in two different temper conditions were shocked with a high-explosive plane-wave lens and tested in compression. The suppression of the Shape-Memory effect typical of artificially aged Uranium-Niobium alloys appears to have been removed following shock loading. Metallography, scanning electron and ion microscopy, X-ray diffraction, and neutron diffraction techniques were applied to characterize the deformation mechanisms. Texture evolution during compressive deformation evaluated via neutron diffraction in both temper conditions is discussed in comparison to the texture evolution normally observed in non-shocked Uranium-Niobium Shape-Memory Alloys, as is the role of retained high-pressure phases on the subsequent deformation behavior. Additionally, the shear-banding behavior is investigated via the "top-hat" forced-shear specimen geometry. Metallography is presented to reveal the twinning local to the shear-banding.

**11:00 AM**

**Mechanical and Computational Investigation of Ni-Al Laminates of Laser-Shock Compression and Spalling**: *Chung-Ting Wei*[1]; Vitali Efrem[1]; David Benson[1]; Brian Maddox[1]; Timothy Weihs[1]; Adam Stover[1]; Marc Meyers[1]; [1]University of California, San Diego

Ni-Al laminates with two different bilayer thicknesses, 5 and 30 µm, were subjected to high intensity laser-shock. The initial pressures of the laser-shock vary from 25 to 333 GPa. The cross-sections of recovered samples, analyzed by SEM and the 2-dimentional simulation RAVEN, show the consistencies between the SEM observations and the computational simulations. The morphologies of fragments, which were expelled by laser-shock wave compression and reflection from the spall surface, reveal that fragments from thinner bilayer sample have non-layered morphology, which is distinct from the fragments of thicker bilayer samples. The as-prepared Ni-Al laminates were subjected to quasi-static extension test and Vikers hardness test. The fracture toughness of thinner and thicker bilayer samples are 75 and 130 MPa, respectively. The measurements of the Vikers hardness are 1.51 and 1.92 GPa as well as the yield strength are 502 and 641 MPa for 5 and 30 µm bilayer samples, respectively.

**11:20 AM**

**Improvement of Charpy Impact Properties in Heat Affected Zones of API X80 Linepipe Steels Containing Complex Oxides**: *Hyo Kyung Sung*[1]; Sang Yong Shin[1]; Woo-Yeol Cha[2]; Kyungshik Oh[2]; Sunghak Lee[1]; [1]POSTECH; [2]POSCO

This study is concerned with the improvement of Charpy impact properties in heat affected zones (HAZs) of API X80 linepipe steels containing complex oxides. The steels were fabricated by adding Mg and O2 to form complex oxides, and their microstructures and Charpy impact properties were investigated. The number of complex oxides increased as the excess amount of Mg and O2 was included in the steels. After the HAZ simulation test, the steel having many oxides contained a considerable amount of acicular ferrite, together with bainitic ferrite and granular bainite. The volume fraction of acicular ferrite increased as the amount of complex oxides increased and the heat input decreased, and thus Charpy impact properties of the oxide-containing steel HAZs were greatly improved. These findings suggested that the active nucleation of acicular ferrite in the oxide-containing steel HAZs was associated with the great improvement of Charpy impact properties of the HAZs.

**11:40 AM**

**Microstructural Analysis of Separations Occurring during Charpy Impact Test of Linepipe Steels**: *Seokmin Hong*[1]; Sang Yong Shin[1]; Jin-ho Bae[2]; Kisoo Kim[2]; Sunghak Lee[1]; Nack J. KIM[3]; [1]POSTECH; [2]POSCO; [3]POSTECH GIFT

The microstructural investigation was conducted on separations occurring during Charpy impact tests of linepipe steels. Particular emphasis was placed on roles of microstructural phases such as acicular ferrite, bainite, and hard secondary phases. The microstructural analysis of fractured impact specimens revealed that the band structure of bainite elongated along the rolling direction worked as prior initiation sites for separations, and that the number and length of separations increased with increasing bainite. Tearing-shaped separations were found in the steels having high work hardenability because the hammer-impacted region was seriously hardened during the impact test, which led to the reduction in the impact toughness. According to the analysis on the effect of separations on Charpy toughness, the energy transition temperature increased as the length of separations increased. This was because brittle fracture occurring at low temperatures reduced the ratio of ductile fracture, while the increased separations further decreased ratio of ductile fracture.

## Global Innovations in Manufacturing of Aerospace Materials: The 11th MPMD Global Innovations Symposium: Perspectives from Government and Industry

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Shaping and Forming Committee, TMS: High Temperature Alloys Committee

*Program Organizers:* Deborah Whitis, General Electric Company; Thomas Bieler, Michigan State University; Michael Miles, BYU

Monday AM Room: 306
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Deb Whitis, GE Aviation; Rollie Dutton, AFRL-RX

**8:30 AM Invited**

**Future Materials and Process Needs for Commercial Jet Transports: The 21st Century Challenge**: *Alan Miller*[1]; [1]Boeing

Despite the recent economic turndown and its effects on the commercial aviation industry, the long term prognosis for future aircraft demand is still robust with demand for approximately 29,000 new airplanes through 2028. Recent twin aisle airplanes have dramatically increased the amount of carbon fiber reinforced materials used in the airframe which changes the proportions of different materials used in the airplanes. Looking to future airplane design opportunities, we can visualize multiple development paths which drive varying combinations of materials for future airplanes. One scenario would address more polymer composite applications while other scenarios could envision significant alloy improvements which drive more metallic airplane structures. Looking beyond structures we can also see the demand for novel

materials for systems and propulsion applications. The fusion of design properties into integrated material systems (e.g., structural, electrical, acoustic, thermal conductivity, thermal stability, etc.) offers unique design solutions to be developed and matured. The international market economics will continue to drive more performance for less cost so that integrated manufacturing processes, production systems, and fleet support technologies will need to be designed commensurate with design enhancements. Overarching all of this development work are progressive environmental requirements that will drive substantial reductions in emissions, hazardous materials, waste streams, and overall environmental effects. There has never been a time when the combined technical challenges have been higher for performance, cost, and environmental factors – these challenges will take all of us working together across industry, government and academia to find solutions that work.

**9:00 AM Invited**
**Realizing Advances in Aerospace Materials: ONR Perspective**: *Julie Christodoulou*[1]; [1]Office of Naval Research

The Office of Naval Research supports basic and applied research in advanced materials and processing for numerous applications that support the needs of naval aviation and sea-launched defensive and strike systems.Our role is to ensure that essential and unique requirements imposed by operations in the marine environment can be met,reliably and affordably.Examples of current programs include, environmental/thermal barrier coating systems and environmentally resistant monolithic materials for turbine engines; novel approaches to materials systems for hypersonic vehicles that must operate at high Mach number near sea level; environmentally insensitive high strength steels for landing gear; and concepts for integrating thermal management capability into load bearing structures.Transitioning these advancing into competitive systems and platforms is never trivial.However,recent efforts to capture understanding into tools for design and life prediction provide a forum for the transfer of knowledge that has proven to be agile and efficient.

**9:30 AM Invited**
**Modeling in Aerospace Materials and Manufacturing in AFRL**: *Mary Kinsella*[1]; Howard W. Sizek[1]; [1]United States Air Force, Air Force Research Laboratory

The development of computational tools to model materials processing and manufacturing has been long supported by the Air Force Research Laboratory and its predecessor. This rich tradition continues today with the Materials and Manufacturing Directorate's support of projects to develop computational tools to advance the state of modeling to support the Department of Defense's needs. This presentation will provide a brief background on past developments supported by the Air Force as well as ongoing efforts in computational materials and manufacturing science. The manufacturing and process modeling needs of the Air force as they modeling of manufacturing processes and needs of the Air Force will also be discussed.

**10:00 AM Break**

**10:20 AM Invited**
**Innovations in Aerospace Materials and Manufacturing Process Development**: *Robert Schafrik*[1]; [1]GE Aviation

High temperature structural materials, such as nickel-based superalloys and titanium alloys, have contributed immensely to societal benefit. These materials provide the backbone for many applications in the aerospace industry. Many challenges, technical and otherwise, were overcome to achieve successful applications. This presentation will highlight some of the key developments in nickel and titanium alloy technology, from the perspective of aeronautical applications. In the past, it was not unusual for development programs to stretch out 10 to 20 years as the materials and processing technology was developed, followed by the development of engineering practice, and lengthy production scale-up. Today, new classes of materials, such as intermetallics and ceramics are challenging traditional superalloys and titanium alloys for key applications. A new development paradigm is described that emphasizes creativity, development speed, and customer value that will enable continued innovations in concurrent material and manufacturing process development.

**10:50 AM Invited**
**Global Innovations in Manufacturing Aerospace Materials: A Rolls-Royce Perspective**: *Malcolm Thomas*[1]; [1]Rolls-Royce

The aerospace industry has provided a number of materials challenges and opportunities relative to the unique temperature, strength, weight and safety requirements that apply to aero applications. Global design and materials communities have responded to these challenges with many innovations to enable continued enhancement of capabilities. Unprecedented improvements in fuel in fuel efficiency coupled with efforts to reduce the environmental impact of aero engines are being achieved. To support the theme of this symposium, this presentation will review a number of materials technology innovations and initiatives that have occurred globally, and their impact on future technology directions. As an example, global aerospace collaboration efforts have become commonplace with new materials technologies and manufacturing efforts supported by organizations throughout the world. Additionally, universities and government research laboratories are also linking with industry to jointly work on opportunities that emerging composite, hybrid and smart materials can offer the aerospace industry, and the challenges that increased performance and environmental issues present.

**11:20 AM Invited**
**The DARPA/DSO Perspective on Materials Science**: *Leontios Christodoulou*[1]; [1]Defense Advanced Research Agency

DARPA's Defense Sciences Office (DSO) pursues and exploits fundamental science and innovation for national security. Materials programs range from developing physics- and chemistry-based models that allow for the design of novel material systems and possessing for radically improved or new properties, to innovative technologies that dramatically reduce the cost of producing titanium metal and its alloys. Mathematical and characterization tools are being generated to enable rapid design and development of new armor systems. Biologically inspired approaches to material synthesis and design are pervasive in many of the DARPA DSO initiatives. Future investments will continue to explore the frontiers of material science, which include new science-based tools for the development of new materials, novel materials for energy and water harvesting, new mechanical designs that exploit or challenge new materials and material systems, and innovative electromagnetic materials that will revolutionize the field of electronics. This aggressive vision to pursue the development of radically new materials and material systems is producing the critical technologies that will allow for the next generation of high-performance military platforms.

## Heterogeneous Nucleation and Initial Microstructure Evolution in Alloys and Colloids: Simulation I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS/ASM: Phase Transformations Committee
*Program Organizers:* Rainer Schmid-Fetzer, Clausthal University of Technology; Heike Emmerich, RWTH Aachen University; Frans Spaepen, Harvard University; Martin Glicksman, University of Florida; John Perepezko, University of Wisconsin, Madison

Monday AM Room: 614
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Yunzhi Wang, Ohio State University; Frans Spaepen, Harvard School of Engineering and Applied Sciences

**8:30 AM Invited**
**Molecular Dynamics Simulation of Two-Dimensional Nucleation in the Context of Crystal Growth**: Dorel Buta[1]; *Mark Asta*[1]; Jeff Hoyt[2]; [1]University of California, Davis; [2]McMaster University

We present results of molecular dynamics (MD) simulations of crystal growth from the melt, mediated by two-dimensional terrace nucleation at a faceted crystal-melt interface. The work focuses on the well-characterized Stillinger-Weber model of Si, where the (111) oriented crystal-melt interface is faceted. An analysis of equilibrium crystalline fluctuations is used to extract step free energies. The steps are diffuse in nature and have a free energy considerably

smaller in magnitude than that measured at crystal-vacuum interfaces in the same system. We will also present results of non-equilibrium MD growth simulations as a function of system size. The results are consistent with a two-dimensional nucleation and growth mechanism for undercoolings up to approximately 30 K. An analysis of the kinetics at 20 K undercooling is used to extract the lag time and steady-state island nucleation rates. The results will be compared to available classical theories of two-dimensional terrace nucleation kinetics.

**8:55 AM**
**Molecular Dynamics Simulation of Nucleation Process**: *Ramanarayan Hariharaputran*[1]; David Wu[1]; [1]Institute of High Performance Computing, Singapore

Phase transfomation is exploited in designing and processing materials for various applications. In most phase transformations, nucleation process plays a significant role in controlling the final microstructure. In the study of nucleation, insights from experimental results are limited due to finite resolution of the observation tools, while theories such as classical nucleation theory (CNT) are handicapped by its assumptions. In this talk, we present our studies of nucleation using molecular dynamics (MD) simulation.Due to length and time scale limitations in equilibrium MD, traditionally the nucleation process is studied at very high driving forces which induces artifacts into the simulation. We propose non-equilibrium corrections to the MD methodology in order to cure these artifacts arising from high driving forces. In this talk, we present our results from simulation of nucleation in unary systems using this modified MD. These results are also compared with the predictions of CNT.

**9:15 AM**
**Prefreezing and Heterogeneous Nucleation at the Cu/Pb Solid-Liquid Interface: A Molecular-Dynamics Simulation Study**: *Jesus-Pablo Palafox-Hernandez*[1]; Mark Asta[2]; Brian Laird[1]; [1]University of Kansas; [2]University of California at Davis

We report results of molecular-dynamics simulations of the Cu/Pb solid-liquid interface. We employ Embedded Atom Method (EAM) potentials to study this interface at temperatures between the melting points of Pb (618K for this model) and Cu (1279K). The two interfacial orientations studied (Cu [111] and [100]) are characterized through the calculation of the density, energy, orientational order, stress and diffusion constant as functions of distance normal to the interface. Our results at 625K (slightly above the melting temperature of Pb) for the Cu [111] interface show the complete "wetting" of the Cu/Pb interface by a layer of (metastable) crystalline Pb, leading to rapid heterogeneous nucleation of crystalline Pb when this system is quenched below the the Pb melting temperature. For the Cu [100] orientation at the same temperature, no such wetting layer is observed, and heterogeneous nucleation at this interface is not observed when undercooled.

**9:35 AM**
**Computer Simulation of Solid-Liquid Interfaces in Metals**: *Roberto E. Rozas*[1]; Juergen Horbach[1]; [1]German Aerospace Center

Key parameters for the description of nucleation phenomena are solid-liquid interfacial free energies. Using molecular dynamics (MD) computer simulation, interfacial properties of crystal-liquid interfaces are investigated for Ni and Ti. Potentials of the embedded atom type are employed. We show that these models lead to a realistic description in terms of melt properties and the crystallization process. Inhomogeneous systems are simulated where the crystal phase in the middle of an elongated simulation box is surrounded by the liquid phase and separated by two interfaces (due to periodic boundary conditions in all Cartesian directions). The melting temperature and crystal growth coefficients are determined. At coexistence, we demonstrate how one can accurately obtain interfacial free energies from a detailed analysis in the framework of capillary wave theory, using both the broadening of the solid-liquid interface with increasing lateral system size and the capillary wave spectrum as input.

**9:55 AM**
**Dissolutive and Reactive Wetting**: *James Warren*[1]; Daniel Wheeler[1]; William Boettinger[1]; [1]NIST

The analysis a solid nuclei wetting and/or reacting with an impurity in a melt provides the basis for classical models of nucleation. Conversely, models of a liquid melt dissolving into/reacting with a solid substrate provide insight into phenomena as diverse a VLS growth and soldering. In this talk I will explore current efforts to model dissolutive and reactive wetting using phase field models of wetting and spreading and explore some of the new metrics that might provide better predictive power in understanding these systems. Particular attention will be paid to the computational and theoretical challenges in achieving realistic experimental simulations, as well as the outstanding issues towards complete models of a liquid-solid system.

**10:15 AM Break**

**10:35 AM Invited**
**Entropy in Crystal Nucleation of Hard Spheres**: *Eli Sloutskin*[1]; Peter Lu[1]; David Weitz[1]; [1]Harvard University

Crystal nucleation is among the most important and fundamental processes in materials science. Yet this process is poorly understood, even in a system as simple as hard spheres. We characterize crystal nucleation in a model system of colloidal hard spheres, using confocal microscopy to individually resolve each sphere. The hard-sphere crystal nuclei are neither compact solids nor tenuous fractals, but instead adopt a wide range of morphologies. We account for these morphologies with a simple empirical model that describes our experimental size distributions of the nuclei and exactly reproduces our measured nucleation rates. We use the same model to account for size distribution of nuclei in copper and find quantitative agreement with computer simulations; this indicates that ourapproach may be applicable to other systems.

**11:00 AM**
**Analysis of Cluster Statistics in Homogeneous and Heterogeneous Nucleation**: *David Wu*[1]; Ramanarayan Hariharaputran[1]; [1]Institute of High Performance Computing

In nucleation studies of conventional materials, precritical clusters of the stable phase are normally too small to observe directly; thus the nucleation rate is inferred from counting large clusters that are formed via a subsequent growth stage. In colloid systems and in computer models, however, very small clusters are routinely observed in situ. We survey existing methods for interpreting the statistics of small clusters and conclude that they are either inaccurate or are impractical due to the amount of data required. We propose a new method to analyze cluster statistics and show that parameters such as the critical size and the Zeldovich factor can be obtained. These parameters can give insight to the nucleation process that is not obtainable from measured nucleation rates alone.

**11:20 AM**
**Phase Behavior and Microstructure of Binary Colloidal Mixtures**: *Nina Lorenz*[1]; Hans-Joachim Schöpe[1]; Holger Reiber[1]; Thomas Palberg[1]; Patrick Wette[2]; Ina Klassen[2]; Dirk Holland-Moritz[2]; Dieter Herlach[2]; Tsuneo Okubo[3]; [1]University of Mainz; [2]German Aerospace Center; [3]Institute for Colloidal Organization

We report a systematic study of the phase behavior of charged colloidal binary mixtures of different size and charge ratio. We find that with decreasing size ratio $\Gamma=a_S/a_L$ (where a refers to the particle radius and S and L refer to the small and large particles, respectively) the miscibility decreases substantially. Accordingly, phase diagrams of spindle-, lower aceotrope- and eutectic-type are observed as well as fluid-fluid phase separation for low $\Gamma$. The influence of the charge ratio $\Lambda=Z_S/Z_L$ shows no systematic behavior. The observed trends bear great resemblance to the behavior of binary metal systems and colloidal hard sphere systems. As compared to the latter, the phase boundary is located at much lower freezing densities and compound formation is only rarely seen. For selected samples we discuss interesting morphologies observed after heterogeneous nucleation.

**11:40 AM**
**Wall-Induced Structures in Heterogeneous Nucleation**: *Fathollah Varnik*[1]; Markus Gross[1]; Suvendu Mandal[1]; [1]ICAMS, Ruhr University Bochum

Via event driven molecular dynamic simulations, we study the effect of atomic structure of the wall on heterogeneous nucleation. A particular focus is the effect of mismatch between the crystalline structure emerging from the undercooled melt and the wall structure. We observe a complex time evolution through structures with a relatively long but finite life time as intermediate steps towards the fnal crystalline order.

## Hume-Rothery Symposium: Configurational Thermodynamics of Materials: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee
*Program Organizers:* Chris Wolverton, Northwestern University; Mark Asta, University of California, Davis; Gerbrand Ceder, Massachusetts Institute of Technology (MIT)

Monday AM Room: 212
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**8:30 AM Plenary**
***William Hume-Rothery Award Winner*: How Hume-Rothery's Work Led to Computational Thermodynamics**: *Didier De Fontaine*[1]; [1]University of California

As an undergraduate student of metallurgy in Belgium, I became acquainted with Hume-Rothery's textbooks, and I was particularly intrigued by the question: why could the Cu-Au phase diagram not be reproduced by the Bragg-Williams approximation? The answer came much later with the Cluster Variation Method of Ryo Kikuchi, which was able to handle the problem of frustration on the fcc lattice. Clusters of lattice points, rather than mere pairs, led Juan Sanchez to formulate a cluster expansion (CE) whose cluster functions formed a complete orthonormal set with which solid solutions could be analyzed rigorously. The resulting cluster algebra could then be used in conjunction with first-principles LDA electronic structure calculations to provide a veritable "computational thermodynamics of alloys", a very active field today, as will be apparent from the many applications of it to be presented at this symposium.

**9:15 AM Invited**
**Deconstructing the Cluster Expansion**: *Juan Sanchez*[1]; Alejandro Diaz-Ortiz[2]; [1]University of Texas at Austin; [2]Max Planck Institute for Metals Research

The cluster expansion (CE), initially introduced in the late seventies to describe the configuration of finite clusters will be reviewed. In particular, the presentation will focus on distinguishing between the ability of the CE to accurately parametrize a set of configurationally dependent quantities, e.g. the energy of a binary alloy, and the physical meaning, or lack thereof, of the fitting parameters, e.g. the effective cluster interactions (ECI). Several examples will be presented, such as expansions giving concentration-dependent ECIs and the application of the method to the electronic density of states of binary alloys. Finally, the structure of the CE that emerges from density functional theory will be explored and discussed in the context of numerous efforts over the last 20 years to map the energy of alloys into a relatively small set of ECIs.

**9:45 AM Invited**
**The Existence of a Multi-Phase Critical Point in Metastable fcc Ordering Phase Diagrams and Its Influence on Phase Diagram Features, Interfacial Energies and Alloy Properties**: *John Cahn*[1]; [1]University of Washington

Analysis of the puzzling four-phase critical point computed for the fcc (CuAu) ordering phase diagram by the Bragg-Williams approximation suggests that it should persist in an altered form as a metastable feature of real phase diagrams at the composition along the ordering spinodal where the cubic term in the free energy expansion changes sign. Properties that are still affected when the point is metastable are heats of ordering, some curves on the phase diagrams, and some interfacial energies. These in turn should affect the concentration dependences of gamma prime nucleation, coarsening rates, and mechanical properties.

**10:15 AM Break**

**10:45 AM Invited**
**What's New in Cluster Expansion?**: *Gus Hart*[1]; Rodney Forcade[1]; Tobias Kerscher[2]; Richard Taylor[1]; Lance Nelson[1]; Alejandro Diaz-Ortiz[3]; [1]Brigham Young University; [2]University of Erlangen; [3]Max-Planck Institute for Metals

The cluster expansion has evolved significantly in the last several decades and has become an extraordinarily accurate method. What started out as conceptually simple, qualitative approach has become a complex method where practioners achieve accuracies approaching that of first-principles methods. Despite the maturity of the method, there are still some fundamental questions that remain elusive. In this talk I will discuss some of the unanswered questions and some recent developments: representation of derivative structures, fitting approaches, parallelization of CE-based MonteCarlo simulations, understanding the effects of noisy data, etc.

**11:15 AM Invited**
**Cluster Expansions from Bond-Order Potentials**: *Ralf Drautz*[1]; David Pettifor[2]; [1]Ruhr-Universität Bochum; [2]University of Oxford

Cluster expansion coefficients are calculated routinely by interpolating density functional total energy data. The cluster expansions obtained in this way may be used to predict accurately thermodynamic and kinetic properties of the system at hand. For complex lattices or multi-component systems it may be useful to estimate the cluster expansion coefficients from coarse grained models of the electronic structure and to predict the systematics of the expansion coefficients as a function of crystal structure and alloy constituents. In our contribution to Didier de Fontaine's Hume-Rothery Symposium, we show how cluster expansion coefficients may be obtained for arbitrary lattices from the analytic Bond-Order Potentials by decomposing the local moments of the atomic density of states into occupation dependent clusters and occupation independent reference contributions.

**11:45 AM Invited**
**Application of Continuous Displacement Cluster Variation Method to Phase Equilibria Calculations**: *Tetsuo Mohri*[1]; [1]Hokkaido University

Cluster Variation Method (CVM) has been recognized as one of the most reliable theoretical tools to incorporate wide range of atomic correlations. By combining CVM with electronic structure total energy calculations, first-principles phase equilibria calculations have been extensively attempted. One of the deficiencies of the conventional CVM, however, is the fact that the local lattice distortion is not efficiently introduced. Hence, various inconveniences such as the overestimation of an order-disorder transition temperature or an underestimation of a single phase field have reulted in the calculated phase diagram. In order to circumvent such inconveniences, Kikuchi and his coworkers developed Continuous Displacement Cluster Variation Method. It is demonstrated that the additional freedom introduced by the local atomic displacement further decreases the free energy of a system and a resultant phase diagram is significantly improved.

## Hydrometallurgy - General Session: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Aqueous Processing Committee
*Program Organizer:* Michael Free, University of Utah

Monday AM Room: 310
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Michael Free, University of Utah

**8:30 AM**
**Highly Selective Oxygen Evolution Anodes for Electrowinning of Metal**: *Masatsugu Morimitsu*[1]; Kana Uno[1]; Naoyuki Oshiumi[1]; [1]Doshisha University

This paper presents our recent development on the oxide coated titanium electrode, which is highly active for oxygen evolution and has an excellent selectivity to oxygen evolution against unwanted side reactions such as manganese or cobalt oxides deposition on the anode used in zinc or cobalt electrowinning. The electrode consists of a mixture of amorphous $IrO_2$ and $Ta_2O_5$ on a titanium substrate by thermal decomposition method. The results demonstrate that the amorphous oxide coating is effective to reduce the electric power consumption of electrowinning process and can suppress the deactivation of the anode's surface during continuous operation.

**8:50 AM**
**Characteristics of Cathodic Reduction of Oxygen on Gold Electrode**: *Yong-bin Yang*[1]; Tao Jiang[1]; Qian Li[1]; Yu-feng Guo[1]; [1]CSU

Cathodic reduction of oxygen is a process involved in many fields. As is well known, the mechanism of oxygen reduction usually varies considerably

with electrode substrates. In this paper, cathodic reduction of oxygen on gold electrode was studied by linear sweep voltammetry with regards to cyanide leaching of gold. As the results showed, the electron transfer number increased as potential shifted negatively and decreased as pH value increased. Besides, the increment of electron transfer number as a function of potential decreased when pH value increased. When pH increased to 13.0, the volt-ampere curve exhibited an approximate plateau with currents approximate to the limiting current of an 2e process. By varying dissolved oxygen concentration, the electron transfer number was measured for pH11.0 at different potentials, the results being 1.894e, 2.387e, 2.732V, 2.982V, and 3.227V at -0.30V, -0.35V, -0.40V, -0.45V, and 0.50V, respectively.

**9:10 AM**

**Dissolution of Precious Metal Alloys Containing Zinc in Acid Solution**: *Hideaki Sasaki*[1]; Takashi Nagai[1]; Masafumi Maeda[1]; [1]Institute of Industrial Science, The University of Tokyo

Dissolution of precious metals in aqueous solutions can be enhanced by alloying with other metals. Taking the advantage of the phenomena, a new recovery process for precious metals from scraps was proposed. The process incorporates an exposure of precious metals to the vapor of Zn prior to acid leaching. Precious metals form alloys with zinc and become easy to dissolve. In this study, dissolution of precious metal alloys containing zinc (Pt-Zn, Au-Zn, Rh-Zn) was examined quantitatively by electrochemical measurement. Anodic dissolution rates of precious metals and zinc from the alloys were measured separately by channel flow double electrode (CFDE). Potential dependencies and time variations of the dissolution were observed.

**9:30 AM**

**Extraction of Copper from Sulfate Leach Solution Containing Minor Metallic Constituents in Mixer Settler Unit**: *Vinay Kumar*[1]; Manis Kumar Jha[1]; Manoj Kumar[1]; Jinki Jeong[2]; Jae-chun Lee[2]; [1]National Metallurgical Laboratory; [2]Korea Institute of Geosciences and Mineral Resources (KIGAM)

The present investigation describes R & D studies carried out for recovery of copper from the sulfate solution expected from the leaching of e-wastes in presence of minor constituents such as cadmium, zinc and nickel using solvent extraction process. In order to extract copper in continuous mode in mixer settler unit (MSU), basic studies have been made to optimize the optimum condition using 5%LIX84 in kerosene. Subsequently, the solvent extraction of copper was made in MSU maintaining leach solution flow rate 4.0 L/h and A/O ratio 1. The results showed complete extraction (~97%) of copper in three stages at A/O ratio 1 from the aqueous solution containing 1.98g/L Cu at pH 1.91 in presence of impurities. Loaded metal was completely stripped after scrubbing with 10% sulfuric acid in two stages. The stripped solution could be used for metal/ salt recovery by electrolysis/ crystallization.

**9:50 AM**

**Gold and Silver Recovery by Electrocoagulation**: *Jose Parga*[1]; Jesus L. Valenzuela[2]; [1]Technology Institute of Saltillo; [2]University of Sonora

In Mining operations, cyanidation is the predominant method by which gold and silver are recovered from their ores and it is recognized that the Merrill-Crowe, Carbon in Pulp process are used to recovery gold and silver. Among several options available for recovery precious metals from cyanide solutions, Electrocoagulation (EC) is a very promising electrochemical treatment technique that does not require high concentrations of gold and silver in solutions. First, this study will provide an introduction to the fundamental concepts of the EC method for recovery precious metals from cyanide solutions. In this study, Powder X-ray Diffraction, Scanning Electron Microscopy and Transmission Mössbauer Spectroscopy were used to characterize the solid products formed at iron electrodes during the EC process. The results suggest that magnetite particles and amorphous iron oxyhydroxides present in the EC products remove gold and silver in 5 minutes with an efficiency of more than 99% from cyanide solutions.

**10:10 AM Break**

**10:20 AM**

**Pressure Leaching of Enargite-Pyrite Concentrates**: *Maria Ruiz*[1]; Maria Vera[1]; Rafael Padilla[1]; [1]University of Concepcion

Enargite rich copper concentrates cannot be treated by conventional smelting/converting technology and thus nonconventional methods such as leaching must be used. Since enargite ($Cu_3AsS_4$) is a hard to dissolve mineral in acidic solutions, pyrite, a common impurity in copper concentrates, could be used to increase the enargite dissolution rate. On this matter, we present in this work experimental data on the pressure leaching of a mixed enargite-pyrite concentrate in $H_2SO_4$-$O_2$. The results show that the dissolution of enargite from the mixed concentrate is considerably faster than the dissolution of pure enargite mineral. The leaching rate increases significantly with temperature. Over 95% of enargite was dissolved in leaching 64 micron size particles at 200 °C and 100 psi of partial pressure of oxygen in just 30 minutes. A change in the partial pressure of oxygen from 50 to 150 psi showed also a large influence in the leaching rate.

**10:40 AM**

**The Rate-Enhancing Role Provided to Oxygen by Nitrite (N[III]) in Acidic Aqueous Oxidation-Processes**: *Gerard Martins*[1]; O. Solak-Gok[1]; [1]Colorado School of Mines

During the past three decades since the discovery of the "catalytic" role of nitrite in these processes (e.g. leaching of refractory gold ores), the mechanism by which nitrogen species (N(IV) and N(II) ) provide a cyclic pathway for the enhancement of oxidation by oxygen gas has been tackled, for the most part, from a qualitative or semi-quantitative, euphemistic, perspective. The work conducted recently in the Metallurgical and Materials Engineering Department at the Colorado School of Mines has now provided an insight (as best as we are able to determine) hitherto not published in the open literature. A Rate Model that incorporates the simultaneous homogeneous-reactions participating in the oxidation of ferrous to ferric in a sulfuric-acid electrolyte has been developed. The Model includes the prominent nitrogen species that are constituents in the system: $HNO_2$, $N_2O_3$, NO, $NO_2NO_{-2}$, $NO^{-3}$ and $[Fe(NO)]^{2+}$ and provides time trajectories of their concentrations during the conversion of $Fe^{2+}$ to $Fe^{3+}$. Laboratory-scale experiments have also been performed to address "calibration" characteristics of the Model simulations relative to the data acquired from the experiments.

**11:00 AM**

**Halide Chlorine Leaching for Malachite and Chrysocolla Mineral Copper from Western Utah Copper Concentrate Company**: *Edgar Blanco*[1]; Mark Dotson[1]; [1]Western Utah Copper Company

The production of oxide copper flotation concentrates found a lack of sulfur content necessary to get exothermic reaction in smelter operations. A Malachite and Chrysocolla dissolution is achieved by sulfuric acid leachant (pH: 1.5) and the oxide and precious metals remained in the ore are attacked by halide reaction oxidants. The results show the dissolution of malachite and chrysocolla is greater than 93%. The behavior of precious metals dissolution during the process was investigated. The PLS generated were 22 g/L. The copper can be displaced from PLS by a lesser noble iron element. The main factor to achieve that extraction was a fast reaction by the halide oxidant in acidic media.

## International Symposium on High-Temperature Metallurgical Processing: Innovations in Ironmaking

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
*Program Organizers:* Jaroslaw Drelich, Michigan Technological University; Jiann-Yang Hwang, Michigan Technological University; Tao Jiang, Central South University; Jerome Downey, Montana Tech

Monday AM Room: 619
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Jaroslaw Drelich, Michigan Technological University

**8:30 AM Introductory Comments**

**8:35 AM Keynote**

**Development of a Novel Gas-Suspension Ironmaking Technology with Greatly Reduced Energy Consumption and $CO_2$ Emission**: *Hong Yong Sohn*[1]; Moo Eob Choi[1]; [1]University of Utah

A novel alternative ironmaking technology based on direct gaseous reduction of iron ore concentrate is under development. The process is based on the flash reduction of concentrate particles in hydrogen or syngas with drastically reduced $CO_2$ emission. The direct use of the concentrates, bypassing agglomeration

and cokemaking, greatly decreases the energy requirement for ironmaking, by about 40% of a typical blast furnace operation. The process is expected to be sufficiently intensive to give a production rate comparable to a modern blast furnace with a much smaller furnace volume.The kinetic feasibility tests in lab- and bench-scale reactors demonstrated sufficiently rapid flash reduction of the concentrates in pure hydrogen or syngas at 1200 – 1500°C within 2 – 10 seconds residence time. Process design and simulation has been performed using the METSIM software with several process flowsheets composed of a flash reactor, heat recovery system, water removal and recycle of hydrogen.

**9:15 AM**

**Thermal Equilibrium Calculation and Application of Microwave Heated Ignition (MHI) for Iron Ore Sintering**: *Yuanbo Zhang*[1]; Xiaoming Mao[1]; Zhucheng Huang[1]; Guanghui Li[1]; Tao Jiang[1]; [1]Central South University

As well-known, conventional sintering ignition has many disadvantages, such as low energy utilization rate, much waste gas, etc. To develop a clean and efficient sintering ignition technology is significant to the Iron & Steel industry. Central South University and Bao-steel have put forward a novel process of microwave heated ignition (MHI) for iron ore sintering. In this investigation, the thermal equilibrium of MHI is firstly calculated. Providing that ignition time is 90s, the ignition temperature required is no less than 639°C, and the lowest heat quality for ignition is 25625 kJ/m2. The pilot-scale sintering experiments using MHI have been done in a 1260mm×320mm×300mm sintering pot, and the optimal conditions are obtained. Compared with the former findings, the energy consumption of MHI is only 25.65 MJ/m2, far less than that of coal gas ignition (CGI) of 185.13MJ/m2.

**9:35 AM**

**Coal-Based Direct Reduction of Iron Concentrate Pellets by Microwave Heating**: Wang Xia[1]; *Huang Zhucheng*[1]; [1]Central South University

In this paper, direct reduction of iron concentrate pellets in continuous shaft furnace by microwave heating is studied. Fe2O3 and fixed carbon in coke has a strong capacity to absorb microwave, the material can quickly reach the required temperature. However, gangue minerals remain lower temperature because of their weak microwave absorbing capacity, so that selective heating of pellets can be realized. In this investigation, the oxidized pellet with TFe of 64 .24% is studied. The results show that under the condition of carbon to iron ratio of 0.32 heated by microwave, the temperature of materials can rapidly rise up with the increasing of microwave output power. The metallization rate of pellets is up to 90% when direct reduction time more than 50 minutes and reduction temperature higher than 950°.

**9:55 AM**

**Effects of Composite Binder (CB) on Oxidation Behavior of Iron Ore Pellets**: *Tao Jiang*[1]; Youming Hu[1]; Yanfang Huang[1]; Guanghui Li[1]; Guihong Han[1]; Yuanbo Zhang[1]; [1]School of Minerals Processing and Bioengineering, Central South University

Organic binders are found superior to bentonite in improvement of iron grade of pellet. As an organic binder, Funa, has been developed and used in the reduced pellets production in China. In this work, a comparative investigation on the oxidation behaviors of various binder pellets is carried out in order that Funa is applied in production of oxidative pellets. Results show FeO content of bentonite pellets decreases obviously with the increase of roasting temperature from 550° to 1050°, but increases slightly from 1050° to 1250° due to formation of double-layer structure. Meanwhile, the minimal FeO content of Funa pellets appears at 950°, and then the FeO content increases sharply from 950° to 1250°. Polarizing microscope and XRD analysis indicate that reduction reaction occurs inside of Funa pellets, leading to the rise of FeO content of pellets.

**10:15 AM**

**Study on Direct Reduction-Separation of Limonite by Microwave Heating**: *Zhu-cheng Huang*[1]; Lili Lv[1]; [1]Central South University

As well-known, there are a large number of limonite ores all over the world and they are still not completely utilized. It can be separated by physical separation and magnetic roasting technology but the effect is not ideal, iron concentrate grade can't meet the production requirements. In this paper the direct reduction-separation of carbon-containing limonite pellets by microwave heating is studied. The results show that the metallization rate of reduced pellets is up to 91.03% when under the condition of microwave output power at 1.0kw, roasting temperature reaches to 1150° from room temperature in 90 minutes. The concentrate of iron content 75.95% and iron recovery 91.45% can be obtained under the condition of grinding time 20min, magnetic field intensity 0.15T. The new process possesses a promising prospect.

**10:35 AM Break**

**10:50 AM**

**Preparation of Metallized Pellets and Recovery of Tin and Zinc from Tin, Zinc-Bearing Complex Iron Concentrates**: Dan Huang[1]; Yuanbo Zhang[1]; Guihong Han[1]; Guanghui Li[1]; *Tao Jiang*[1]; [1]School of Minerals Processing and Bioengineering, Central South University

The tin, zinc-containing iron ores are typical complex and intractable ones, and great reserves of them are found in China. At present, they have not been efficiently utilized. Based on the thermodynamic analysis of iron, tin and zinc oxides reduction by CO, technological conditions of metallized pellets preparation from the complex iron concentrates (containing 0.30% Sn, 0.21% Zn and 65.42% Fe) by one-step direct reduction process are studied and a new comprehensive utilization process is developed in this investigation. Volatilization of 90% tin and 95% zinc are obtained under the optimum conditions, and metallization of iron is above 94%. The compression strength of roasted pellet is more than 1800 N/P. The residual tin and zinc in the finished pellets is below 0.03%. The metallized pellets can be used as steelmaking burdens.

**11:10 AM**

**Researches on Magnetic Roasting-Separation of Coal-Containing Limonite Pellets by Microwave Heating**: Hu Bing[1]; *Huang Zhucheng*[1]; [1]Central South University

Limonite ores is difficult to obtain ideal indexes by traditional physical processes. In this investigation, the limonite with TFe of 48.92% is studied. The experimental results show the conventional heating takes a long reduction time, easily leads to cold center and over reduction, and even forms fayalite. Magnetic roasting-separation of limonite by microwave heating under the conditions of output power 1.0kw at 35min achieves the concentrate of iron content 61.15% and recovery of 88.35% while the concentrate of iron content 60.55% and recovery of 74.06% is obtained under the optimal conditions for 60min at 800° following conventional heating. The whole pellet is heated simultaneously, which gives priority to heat coal particles and iron minerals, and accelerate the transformation of limonite into magnetite. Meanwhile, gangue minerals remain lower temperature due to their weak microwave absorbing capacity, and thus it greatly restrains the formation of fayalite.

**11:30 AM**

**Mathematical Modeling for Side-Blow Combustion Region in Iron Bath Reactor with H2-C Mixture Reduction**: *Zhang Bo*[1]; Hong Xin[1]; [1]Shanghai University

The basic idea of H2-C mixture reduction reflexes using hydrogen as main reductor and carbon as main heat generator in iron bath smelt reduction reactors on purpose to cut down total energy consumption and CO2 emission. The author applied the methods of modeling for separating regions and complex integration to research the kinetics behavior of this new metallurgical reactor. Important one among separating regions was side-blow region where the changes of temperature and substance concentration field in multiple-phase mixture of solid, liquid and gas were coupled integrating models including shrinking core of granulated carbon, carbon-oxygen combustion and heat absorption of slag drops etc. Besides inlet flux of injecting oxygen and carbon, the boundary and initial conditions included substance and energy exchanges in boundaries between the region and other reaction regions. After dispersion treatment with Control-Volume-Method, the model was programmed as software for digital simulation of combustion procedure in side-blow region.

**11:50 AM**

**Al-Fe Separation from High Aluminium Content Limonite Ores by Salt-Added Reduction Roasting Process**: *Tao Jiang*[1]; Mudan Liu[1]; Na Sun[1]; Guanghui Li[1]; [1]School of Minerals Processing and Bioengineering, Central South University

Large reserves of high aluminium content limonite ores are widely found all over the world. Traditional processes are found invalid to remove aluminium from the ores due to the close combination of aluminium with iron minerals. Sodium salt-added reduction roasting followed by magnetic separation is developed in this study. A metallic iron concentrate with the total iron grade of 91.00% and aluminium content of 1.36% is achieved in laboratory for a sample

of 48.92% iron and 8.16% Al2O3, and the recovery of iron is greater than 90%. SEM and XRD investigations on microstructures of reduced pellets indicate that, aluminium and iron minerals combine closely without additives, and added sodium salts destroy the complex substitution relationship between iron and aluminium and accelerate the growth of metallic iron granules remarkably.

**12:10 PM**

**A Study on Beneficiation of Low Grade High-Phosphorus Iron Ore**: Zhu Deqing[1]; Chun Tiejun[1]; Pan Jian[1]; Wei Xuemei[1]; [1]Central South University

The reduction roasting-magnetic separation-acid leaching of low grade high-phosphorus iron ores was studied. The process parameters of acid leaching were optimized with rougher iron ore concentrate which was obtained by reduction roasting-magnetic separation of ROM ores. Effects of various factors on upgrading iron and dephosphorization were discussed in acid leaching. The result showed that the iron ore concentrate, assaying 62.35% iron and 0.20% phosphorus content were achieved at the overall iron recovery rate of 90.54% and dephosphorization rate of 87.42% under the conditions of leaching for 2 hours, at 2.5 liquid-to-solid ratio, 50kg/t sulphuric acid and 500 r/min agitating speed after the ROM ore with 47.28% iron and 1.59% phosphorus content was pretreated by reduction roasting-magnetic separation. It is important to decrease the feedstock of acid leaching by the preconcentration technology of reduction roasting-magnetic separation for saving investment and reducing operation cost.

## Jim Evans Honorary Symposium: Flow Phenomena in Steel Continuous Casting

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division
*Program Organizers:* Ben Li, University of Michigan; Brian G. Thomas, University of Illinois at Urbana-Champaign; Lifeng Zhang, Missouri University of Science and Technology; Fiona Doyle, University of California, Berkeley; Andrew Campbell, WorleyParsons

Monday AM Room: 620
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Brian Thomas, University of Illinois at Urbana-Champaign

**8:30 AM**

**Jim Evans: A Reflection on his Impact**: *Fiona Doyle*[1]; [1]University of California, Berkeley

The Jim Evans Honorary Symposium was organized to pay tribute to a true giant in the field of extractive and process metallurgy. This opening talk briefly discusses how Jim's approach to technical problems has paved the way for new developments in the production of materials, and inspired fruitful work in a much broader arena. But Jim's impact goes far beyond his technical influence; many of Jim's colleagues, collaborators and students (former and present) are here because of the friendship and mentorship that Jim has warmly offered over the years. As discussed here, Jim's love of life and inimitable sense of humor are inextricably interwoven with his lasting technical legacy.

**8:55 AM**

**Liquid Metal Modelling of Continuous Steel Casting**: *Gunter Gerbeth*[1]; Sven Eckert[1]; Klaus Timmel[1]; Xincheng Miao[1]; [1]Forschungszentrum Dresden-Rossendorf

Model experiments with low melting point liquid metals are an important tool to investigate the flow structure and related transport processes in melt flows relevant for metallurgical applications. We present the new experimental facility CONCAST for modelling the continuous casting process of steel using the alloy SnBi at temperatures of 200-400C. The possibilities for flow investigations in tundish, submerged entry nozzle and mould will be discussed. In addition, experimental results will be presented on the impact of a steady magnetic field on the outlet flow from the nozzle, obtained at a smaller-scale set-up working with the room-temperature alloy GaInSn. Local velocities in both facilities are measured by Ultrasound Doppler Velocimetry and contactless inductive flow tomography. The latter is attractive also for real-scale steel casting.

**9:20 AM**

**Experimental and Numerical Simulation of the Mold Region of a Steel Continuous Caster**: *Koulis Pericleous*[1]; Zacharias Kountouriotis[1]; Georgi Djambazov[1]; Francois Domgin[2]; Pascal Gardin[2]; [1]University of Greenwich; [2]ArcelorMittal

We present the development and validation (using an oil/water system) of a FV computer model of the steel continuous casting process. The emphasis is on hydrodynamics and particularly the dynamic behavior of the metal/slag interface. Instability and wave action encourage inclusion entrainment into the melt affecting product quality. To track the interface between oil and water a new implicit algorithm was developed, the Counter Diffusion Method. To prevent excessive numerical damping, a time-filtered version of the k-e turbulence model was used. Air sparged through the Submerged Entry Nozzle simulates experimentally argon in a real process. Air bubbles disturb the oil water layer and affect its dynamics. This effect is modeled using a Lagrangian particle tracking scheme. Integral gas concentration in each computational cell alters the fluid density, as a feedback mechanism between gas and liquid. The simulations are compared against LDA velocity measurements for mean value, turbulence and frequency content.

**9:45 AM**

**Effect of Stopper-Rod Misalignment on Asymmetric Flow and Vortex Formation in Steel Slab Casting**: *Seong-Mook Cho*[1]; Go-Gi Lee[2]; Seon-Hyo Kim[1]; Rajneesh Chaudhary[3]; Oh-Duck Kwon[4]; Brian G Thomas[3]; [1]Pohang University of Science and Technology; [2]Research Institute of Industrial Science and Technology; [3]University of Illinois at Urbana-Champaign; [4]POSCO

Vortices forming near the slag-steel interface in the mold can entrap inclusions and cause defects in continuous casting of steel slabs. Lab experiments employing a 1/3 scale water model were performed to quantify the effects of stopper rod asymmetry on vortex formation. Three stopper-rod placements (aligned, front-misaligned and left-misaligned) were considered. Vortex formation was visualized with a high speed camera by placing sesame-seed tracer on the surface, which enables counting the number and detecting the location of vortices with time. Impeller flow probes were adopted to measure velocity profiles near the surface. Misaligning the stopper-rod placement induces asymmetric flow, resulting in asymmetric surface velocity, velocity variations, and turbulent kinetic energy. These factors influence vortex frequency and location among four zones near the SEN. Most vortices form at the left regions beside the SEN with a left-misaligned stopper-rod. Vortices form more preferentially at outside regions with a front-misaligned stopper-rod.

**10:10 AM Break**

**10:30 AM**

**Slag Infiltration and Initial Solidification Mechanisms during Continuous Casting**: *Pavel Ramirez Lopez*[1]; Peter Lee[1]; Kenneth Mills[1]; [1]Imperial College London

An integrated model able to simulate the metal flow, heat transfer and solidification inside the continuous casting mould is presented. The model calculates direct slag infiltration and its effects on shell formation during mould oscillation. Extremely fine meshes in the meniscus corner (~100\956m) were required to characterise the pressure, velocity and temperature through the slag layers in the shell-mould gap, allowing the prediction of the heat transfer between shell and mould. Results show excellent agreement with physical models for features such as slag film thicknesses and heat flux evolution during an oscillation cycle whereas, predicted shell thicknesses and powder consumption are also in good agreement with plant measurements. Results also describe how variations in shell and slag layer thicknesses are decisive on the formation of typical defects such as oscillations marks and transverse cracks, which are a major source of defects in the casting practice.

**10:55 AM**

**Control of Fluid Flow, Heat Transfer and Inclusions in Continuous Casting: CFD and Neural Network Studies**: *Petri Väyrynen*[1]; Shenqiang Wang[1]; Jukka Laine[1]; Seppo Louhenkilpi[1]; [1]Helsinki University of Technology

Fluid flow and heat transfer calculations have been carried out in tundish and mould including different kind of submerged entry nozzles. The effect of different kind of tundish dams have been studied in a bloom tundish in steady state and transient conditions. Many different CFD parameters, like turbulence models and mesh density, were tested. CFD calculations were also carried out to study the effects of swirling flow inside the SEN as well as different kind on

SEN nozzles on mould flow phenomena. Different kind of criteria for the ideal mould flow were derived. Neural network model was developed to predict and control the tundish temperature from process parameters and casting time.

**11:20 AM**

**Turbulent Instabilities in a Thin Slab Mold**: *Rodolfo Morales*[1]; Saul Hernandez-Garcia[1]; [1]IPN

Mathematical simulations are employed to describe short and large scale flow distortions observed in a water model of a thin slab casting mould using a two-port SEN located at deep and shallow positions for two casting speeds of 5 and 7 m/min. Two types of oscillations are identified high frequency and low frequency; the first have short length scales and long life while the length scale of the second involves the whole mould length and have short life. Through turbulent flow principles it was found that the high frequency of the discharging jets is the same as those of the oscillating meniscus. Therefore the discharging jets transfer vibrating momentum, at frequencies of 1.1-5 Hz, to the two upper roll flows during long periods of time inducing meniscus oscillations. Large-scale, short-life oscillations induce a dynamic distortion of the flow which forms deep meniscus depressions.

**11:45 AM**

**Numerical Investigation of the Flow and Steel/Slag Interface Behaviors in Slab Continuous Casting Mold with Electromagnetic Brake and Argon Gas Injection**: *Miaoyong Zhu*[1]; Haiqi Yu[1]; [1]Northeastern University

A new 3-D code for modelling the flow and interface fluctuation behaviours was developed in this paper, with fully taking into account the effects of flow-control-mold (FC-mold) EMBr, argon gas injection and the double actions of two means. The finite element code ANSYS was used to calculate the magnetic induction intensity distribution that produced by the EMBr device. The finite volume code FLUENT was used to calculate the coupled solution for the magnetic field, turbulence model, VOF model and discrete phase model on the basis of imposing the magnetic field data files of the mold region calculated by the ANSYS in a certain data format on the corresponding nodes of the mold geometry model by its own magneto-hydrodynamics (MHD) module. So the simulation results of the magnetic field distribution and the coupled calculation of multi-physics fields are more in line with the actual production situation and more accurate.

## Magnesium Technology 2010: Plenary Session

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
*Program Organizers:* Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Monday AM Room: 612
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Sean Agnew, University of Virginia; Wim Sillekens, TNO Science and Industry

**8:30 AM Introductory Comments**

**8:35 AM**

**Magnesium Alloys in Aerospace Applications - Flammability Testing and Results**: *Bruce Gwynne*[1]; [1]Magnesium Elektron

Current Federal Aviation Administration regulations and other restrictions prohibit magnesium alloy for use in internal aircraft applications that would benefit greatly from its light weighting advantages. This paper reviews magnesium alloy uses in past and potentially new aerospace applications, and then focuses on the regulatory environment and magnesium fire testing evaluations in an effort to have these restrictions modified or eliminated. Flammability experiments conducted with the FAA using laboratory oil burner type bench testing have found that rare earth containing magnesium alloys had a tendency to either not ignite or self extinguish. This is in contrast to traditional aluminum containing magnesium alloys that do not perform as well due to the lack of a protective oxide film. Actual in-aircraft full-scale flammability testing of traditional aluminum alloy and magnesium alloy aircraft seat structure components will be shown and discussed.

**9:00 AM Keynote**

**A Possible Route to Making Magnesium Fit for Hydrogen Storage in Automotive Applications**: Vladimir Skripnyuk[1]; Eugene Rabkin[1]; *Yuri Estrin*[2]; Rimma Lapovok[2]; [1]Technion; [2]Monash University

The high hydrogen storage capacity of magnesium, coupled with the reversibility of hydrogen absorption/desorption, give it an advantage over other metallic systems. However, a high hydrogen release temperature is an obstacle to its mobile hydrogen storage applications. Attempts to reduce this temperature below the desired level of 200 ºC have been unsuccessful. While nanosructuring does improve the storage kinetics, the temperature dependence of hydrogen pressure cannot be controlled in this way. We proposed a novel approach to Mg processing using equal channel angular pressing, which changes both the kinetics and the thermodynamics of hydrogen storage. ECAP produces a long-lived non-equilibrium microstructure characterized by ultrafine grains, enhanced vacancy concentration and excess volume. These factors induce thermodynamic shifts in a desired way. Future efforts will be directed at ECAP optimization in order to reach the temperature and pressure levels required for use of magnesium as a hydrogen storage material for automotive applications.

**9:30 AM Keynote**

**Precipitation Strengthening in Magnesium Alloys Containing Rare Earth Elements**: *Xiaoqin Zeng*[1]; Wenjiang Ding[1]; Yujuan Wu[1]; Liming Peng[1]; Alan Luo[2]; [1]National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University; [2]Materials and Processes Laboratory, General Motors Research and Development Center

Rare earth (RE) elements, gadolinium (Gd), yttrium (Y), and neodymium (Nd), have been studied for their effects on the room- and elevated-temperature properties of magnesium alloys. Phase identification and microstructure analysis of RE-containing magnesium alloys have been conducted using optical microscopy (OM), scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM) techniques. Tensile and creep properties of the alloys have also been tested at room and elevated temperatures. The results have shown significant aging hardening responses in these alloys and revealed the precipitation sequences and the crystal orientation relationships of the RE-containing strengthening phases. It has been demonstrated that the primary strengthening mechanism in these RE-containing magnesium alloys is the formation of meta-stable phases during aging treatment. Long period stacking order (LPSO) has been observed when zinc (Zn) is added to the RE-containing magnesium alloys, which provides further strengthening to the alloys.

**10:00 AM Keynote**

**Thermodynamics and Constitution of Mg-Zn-Ce Alloys**: Chen-nan Chiu[1]; Artem Kozlov[2]; Joachim Groebner[2]; *Rainer Schmid-Fetzer*[2]; [1]National Tsing Hua University; [2]Clausthal University of Technology

This is a first report in our series of ongoing studies of ternary Mg-Zn-RE systems. Cerium is a major component of misch-metal and thus considered as the most important part in these industrially used rare earth (RE) additions. Therefore, the Mg-Zn-Ce ternary system plays a prominent role among the Mg-Zn-RE systems and precise knowledge of the phase diagram and thermodynamic properties of the Mg-Zn-Ce system is necessary for a better understanding of alloy design. It is also a key system for extending our multicomponent Mg-alloy database. The Mg-rich part of the ternary Mg-Zn-Ce system was experimentally investigated (DTA, DSC, SEM/EDS) by key samples to determine the primary crystallization, invariant reactions and solid solubilities. A consistent thermodynamic model of the ternary Mg-Zn-Ce system is developed by using the Calphad method.This work is supported by the German Research Foundation (DFG) in the Priority Programme “DFG-SPP 1168: InnoMagTec”

**10:30 AM Break**

**10:50 AM Keynote**

**Modeling Temperature and Strain Rate Dependent Inelastic Deformation and Recrystallization in Mg Alloys**: *Douglas Bammann*[1]; Esteban Marin[1]; Kiran Solanki[1]; [1]Mississippi State University

The thermodynamics of Coleman and Gurtin are employed with respect to the natural to ensure satisfaction of the second law of thermodynamics and to derive a consistent thermal balance equation including both internal and external work as source terms. Evolution equations for the state variables are determined from the physics of the defects they represent. For example, Kocks-Mecking dislocation storage and recovery for SSDs and the Cocks-Ashby

evolution for void growth in a steady state creeping material. In addition, kinematic expressions (plastic stretching, plastic spin…) are required, consistent with the degrees of freedom introduced by the multiplicative decomposition of the deformation gradient. The volume fraction of recrystalized grains is also introduced to describe both static and dynamic recrystallization. Grain size dependence of the kinetics of plastic flow, mean free path of the SSDs and GNDs, and grain evolution are also considered in the model.

**11:20 AM**

**Magnesium Alloys in Army Applications: Past, Current and Future Solutions**: *Suveen Mathaudhu*[1]; Eric Nyberg[2]; [1]U.S. Army Research Laboratory; [2]Pacific Northwest National Laboratory

As early as the 1940's Magnesium-alloys have been used for military applications, from aircraft components to ground vehicles. The drive for usage was largely availability and lightweighting of military systems. But the promise of widespread usage has not been met. With recent fuel costs and logistical burdens has come increased recognition of the need to develop and implement lightweighting strategies for the full range of weapons platforms (land, sea, and air) without a loss in platform functionality. The potential for significant improvements in the mechanical and physical properties of lightweight metals will drive design of ultralightweight metal structures and components, thus reducing large logistical burdens, minimizing operational constraints and liabilities, and reducing vulnerabilities. This lecture will cover past, present and potential future Mg-alloy applications with a focus on scientific barriers relevant to integration of Mg-alloy, and solutions which are currently being developed to address these issues.

**11:45 AM**

**MagForming - Development of New Magnesium Forming Technologies for the Aeronautics Industry**: *Bruce Davis*[1]; Amir Fein[2]; Wolfgang Entelmann[3]; Elke Hombergsmeier[4]; [1]Magnesium Elektron North America; [2]Palbam-AMTS; [3]Airbus-Deutschland; [4]EADS Deutschland GmbH

MagForming was a European Union Framework Six Priority 4 Aeronautics and Aerospace funded program. Its purpose was to advance the state of the art in forming methods for a range of magnesium alloys in extruded, forged, sheet and plate forms using aerospace prototypes as demonstrators. The program was a collaboration between 12 partners including material manufacturers, tier 1 aerospace suppliers, OEMs, and academic institutes.The program targeted specific areas of forming, including forging, superplastic forming, roll bending, pad forming, deep drawing, and creep forming. The aim was to develop best practices for all of these methods via the production of aerospace prototype parts. This not only acted to demonstrate the forming process and its application to magnesium alloys, but also resulted in the production of useful parts for testing, potentially leading to the use of magnesium in the system and structure areas from which the parts had been taken.

## Materials in Clean Power Systems V: Clean Coal-, Hydrogen Based-Technologies, Fuel Cells, and Materials for Energy Storage: Materials for Clean Coal Power and CCS Systems I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Conversion and Storage Committee

*Program Organizers:* Xingbo Liu, West Virginia University; Zhenguo Yang, Pacific Northwest National Lab ; K. Weil, Pacific Northwest National Lab ; Mike Brady, Oak Ridge National Lab; Jay Whitacre, Carnegie Mellon University; Ayyakkannu Manivannan, National Energy Technology Laboratory; Zi-Kui Liu, Penn State University

Monday AM Room: 211
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Xingbo Liu, West Virginia University

**8:30 AM Keynote**

**Development and Implementation of Materials to Enable Clean Coal Technologies**: *Cynthia Powell*[1]; [1]Office of Research and Development, National Energy Technology Laboratory, US Department of Energy

A growing realization that the environmental impact of energy production must be reduced on a global scale, combined with an increased desire in this country to reduce dependence on foreign energy sources, is driving significant change in the way the United States will derive and produce power in the future. While renewable energy resources will continue to grow in importance, environmentally responsible fossil energy production will be necessary to provide a bridge to the next energy revolution. The practical result is a requirement for affordable, high-performance materials and materials systems capable of withstanding increasingly severe operating environments to enable these next-generation fossil energy systems. This talk will focus on the research being performed at the National Energy Technology Laboratory to identify and meet the requirements for high performance structural materials for advanced combustion and gasification power systems.

**9:10 AM Invited**

**Ageing and Corrosion in CO2 Rich Flue Gases and Their Influence on the Creep and Fatigue Properties of Superheater Materials**: *Axel Kranzmann*[1]; Jürgen Olbricht[1]; Daniela Hünert[1]; Diana Marcano[1]; Wencke Schulz[1]; Werner Österle[1]; Romeo Saliwan-Neumann[1]; Hellmut Klingelhöffer[1]; Gabriele Oder[1]; Ingrid Urban[1]; Birgit Skrotzki[1]; [1]Federal Institute for Materials Research and Testing

Oxyfuel Power Plants combined with Carbon Capture and Sequestration produce flue gases with high CO2 and H2O partial pressures. Commercial steel tubes for steam heaters and super heaters were annealed for up to 1000 h in atmospheres of 70 mol% CO2, 30 mol% H2O and a variation with 29 mol% H2O and 1 mol% O2. Temperatures between 500°C and 650°C and surface stress were applied. The reaction with CO2 during annealing caused significant material loss due to oxidation and, carburization of the base material. Oxidation and carburization may affect the lifetime of plant components by embrittlement, mechanical interaction of thick oxide scales and base material, or rapid local attacks when cracks are induced by mechanical loads. Mechanical testing in oxyfuel atmospheres, involving static creep and cyclic low cycle fatigue loading, was carried out to demonstrate the environmental effect on the mechanical performance of super heater materials.

**9:50 AM**

**Designing Amine-Based CO2 Sorbents - A Computational and Experimental Study**: *John Kitchin*[1]; [1]Carnegie Mellon University

Atmospheric CO2 concentrations are increasing by 1-2 ppmv/year due to CO2 emissions from the combustion of fossil fuels for power generation and transportation. Efficient methods for separating CO2 from flue gas are needed to make the capture cost-effective and technologically viable. We are developing a sorbent-based approach to CO2 capture in which molecular amines are supported on porous substrates. We have used experimental methods to probe the role of moisture in the capture mechanism of CO2, the role of the support in parasitic moisture sorption and the capture capacity of two amidines, DBU and

DBN. We have also used quantum mechanical calculations to explore the range of CO2 capacities that might be possible from functionalized amidines. These functional groups modify the electronic and geometric environment around the CO2 binding site through steric hindrance, hydrogen bonding and electron withdrawing/donating effects.

**10:10 AM Break**

**10:40 AM Invited**

**Deployment of New High Temperature Alloys for Power Generation Systems**: *Bruce Pint*[1]; [1]Oak Ridge National Laboratory

Two Fe-base alloys developed at ORNL, alumina-forming austenitic (AFA) steel and cast austenitic CF8C-Plus, are in the process of moving from invention to application. A considerable effort is required to move an alloy from laboratory heats to commercial products. AFA steels were developed to bridge the gap between high strength austenitics and oxidation-resistant alumina-forming alloys. They are being considered for applications such as high-temperature, thin-walled heat exchangers, boiler and petrochemical tubing, and non-stack fuel cell applications where they are expected to produce minimal Cr poisoning. Decades of alloy strengthening knowledge developed for fission and fusion applications was used to rapidly develop cast CF8C-Plus, which is already used in heavy-duty commercial diesel engines. New applications being considered are in additional automotive applications, turbine casings and boilers. Examples will be given about the deployment process for these alloys and the program required to support these efforts.

**11:20 AM**

**Density Functional Theory Study of Grain Boundary Properties in Ni-Base Superalloys**: *Kuiying Chen*[1]; Wei-Di Cao; [1]ATI Allvac

Traditionally, impurities phosphorus was treated as a detrimental element in the development of Ni-base superalloys. However, the experimental works conducted at ATI Allvac demonstrated that the effect of phosphorus in Ni-base superalloys is quite complex. Within a certain range of concentrations, phosphorus has a positive effect on stress rupture / creep resistance of all wrought Ni-base alloys. The rupture life was increased, not decreased, by the addition of phosphorus. This positive effect of phosphorus is alloy-dependent with the largest effect observed in Fe-containing Ni-base alloys and much weaker in alloys without significant Fe addition such as Waspaloy and Rene 220. The mechanisms responsible for the above phenomena are not clear. Therefore it is proposed to investigate the effect of phosphorus on Σ5 (012) grain boundary (GB) properties with Fe-containing and Fe-free Ni-base superalloys using ab initio density functional theory calculations. The study will (1) reveal why and how phosphorus at the GB of Fe-containing and Fe-free Ni-base superalloys affect the GB strength as a function of phosphorus content; (2) to examine how boron at the GB of Fe-containing and Fe-free Ni-base superalloys affect the GB strength as a function of boron content; (3) to study the interaction between phosphorus and boron in Fe-containing and Fe-free Ni-base superalloys in terms of effect on GB strength with an aim of providing reasonable explanations for experimental results; (3) on the basis of (1) and (2), find the critical phosphorus composition $C_0$, where phosphorus or boron starts to weaken GB strength. The research will provide a comprehensive understanding on impurity such as phosphorus or boron complex effect on GB strength.

**11:40 AM**

**Materials Selection for Steam Turbine Components in Advanced Ultra Supercritical Power Plants**: *Jeffrey Hawk*[1]; [1]U.S. Department of Energy

In order to reduce greenhouse gas emissions to combat global warming, the efficiency of pulverized coal steam power plants must be increased. Raising the temperature is the primary way to do this, but to significantly increase efficiency, temperatures in excess of 700C are needed. Above 700C, wrought nickel alloys are needed for steam turbine component parts such as the rotor, blades and valve internal parts. Most wrought nickel alloys were designed primarily for aerospace applications or where excellent resistance to corrosion is needed. In many instances high temperatures are experiences but the life of the component is considerably less than the 100,000 hours minimum needed for steam turbine parts. This paper will explore candidate selection criteria for the European AD700 initiative and the DOE 1400F steam turbine program. Phase stability, mechanical behavior, and microstructural stability will be discussed with respect to steam turbine components.

## Materials Processing Fundamentals: Solidification and Casting

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
*Program Organizer:* Prince Anyalebechi, Grand Valley State University

Monday AM Room: 601
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Prince Anyalebechi, Grand Valley State University

**8:30 AM**

**Fe-Mn-Al-C Austenitic Steels Treated by Ca and Ce**: *Simon Lekakh*[1]; Von Richards[1]; Angella Schulte; David Van Aken[1]; [1]MST

Phosphorous segregated at grain boundaries during solidification and heat treatment dramatically decreases low temperature toughness of precipitation hardened Fe-Mn-Al austenitic steel. Thermo-dynamical analysis was performed for evaluation of the possibility of decreasing Pgrain boundary segregation by Ce- and Ca-additives in the melt. It was shown that the predicted probability of P containing compound formation depends on melt composition and treatment sequence. Experimental heats were performed and the several micro-and macro-structural effects of Ce- and Ca-additives were recognized by using automated inclusion analyzer technique. Increasing low temperature toughness in austenitic steel by complex Ca-wire injection followed by Cemishmetal additions was achieved.

**8:50 AM**

**Strong Magnetic Field Induced Phase Alignment during Solidification**: *Zhi Sun*[1]; Muxing Guo[1]; Jef Vleugels[1]; Omer Van der Biest[1]; Bart Blanpain[1]; [1]Katholieke Universiteit Leuven

Imposing a strong magnetic field is considered as an attractive method to prepare materials with aligned or textured structures due to its intrinsic properties, e.g. non-contact and easily adjustable. However, the nature of strong magnetic field effect still remains unclear and further research is needed. In the present paper, the effect of a strong magnetic field on the solidification of a hypereutectic Al-Cu alloy was investigated. After magnetic field treatment, the primary theta phase was aligned parallel to the magnetic field direction. Meanwhile, the eutectic micro-structural constituent became elongated and thinner. A theoretical analysis was conducted to have a better understanding of the phenomena. During solidification, the magnetic Faraday force and dipole-dipole interaction force are considered to decrease the solute pile-up in front of the solid-liquid interface and increase the theta phase formation. The alignment of the theta phase is due to the shape magnetic anisotropy and crystal magnetic anisotropy.

**9:10 AM**

**Experimental Analysis of Thermal, Tensile and Microhardness Properties in Directional Solidified ZA, Zn-Ag and ZINAG Alloys**: Alicia Ares[1]; Sergio Gueijman[2]; *Carlos Schvezov*[1]; [1]CONICET/FCEQyN-UNaM; [2]FCEQyN-UNaM

The family of Zn-Al (ZA) and Zn-Al-Ag (ZINAG) alloys offers high mechanical properties, ease of finishing, the advantage of low energy and pollution-free melting. Also, present alternatives to the well developed alloy systems, such as brasses, bronzes, cast irons and aluminum alloys. The main objective of this paper is to measure thermal parameters in ZA and ZINAG hypoeutectic and hypereutectic alloys directionally solidified which present columnar, equiaxed and columnar to equiaxed transition structures (CET). Also, estimate the mechanical properties, such as microhardness and tensile parameters and correlate these with the thermal and structural parameters (grain size and dendritic spacing). The different types of structures were analyzed with optical and scanning electron microscopy. The results show that bulk alloys have greater microhardness values than those directionally solidified. Also, alloys with equiaxed structures presented a better tensile resistance than the columnar and CET zones. The results of the present investigation are compared with previous experiments. Keywords: Thermal parameter, tensile parameters, microhardness, ZA and ZINAG alloys, directional solidification.

**9:30 AM**

**Effects of Solidification Rate and Alloy Composition on the Cast Microstructure of Aluminum Alloy 5182**: *Prince Anyalebechi*[1]; [1]Grand Valley State University

The effects of solidification rate (0.4-26.1 K/s) and alloying elements on the cast microstructure of aluminum alloy (AA) 5182 have been investigated with directionally cooled laboratory-size ingots. The alloying elements examined included Mg (3.8-4.66 wt.%), Mn (0.35-0.75 wt.%), Fe (0.11-0.42 wt.%), Cu (0.03-0.25 wt.%), and Si (0.11-0.26 wt.%). These reflect the chemical compositions of AA 5182 end stocks with high recycled used beverage container (UBC) content. For a given chemical composition, the average dendrite cell and second phase particle size decreased with increase in average solidification rate. In general, within the levels investigated, Mg did not have a significant effect on the types, volume fractions, and average sizes of the second phase particles. However, increase in Mn, Si, Fe, and Cu increased average sizes and volume fractions of the second phase particles. These effects appear to depend on the maximum solid solubility of the element in aluminum.

**9:50 AM Break**

**10:10 AM**

**Elaboration and Nanoscale Characterization of a Fe-$Y_2O_3$ Nanocomposite Prepared by Reactive Ball-Milling and Annealing**: *Mathilde Brocq*[1]; Fabrice Legendre[1]; Bertrand Radiguet[2]; Mathieu Couvrat[1]; Fabien Cuvilly[2]; Philippe Pareige[2]; Jean-Marie Lebreton[2]; [1]SRMP - CEA; [2]GPM-Université de Rouen

Reactive high energy ball-milling has known an increasing interest from both fundamental and applied point of view. We studied the properties of this technique through the study of a metal-oxide nanocomposite. It was synthesized by ball-milling $YFe_3$ and $Fe_2O_3$ in the stoichiometric proportions of the following chemical reaction: $2YFe_3 + Fe_2O_3 \rightarrow 8Fe + Y_2O_3$. Then it was briefly annealed. The material was characterized at different steps of the process by X-Ray Diffraction, Mössbauer Spectroscopy and Atom Probe Tomography.We will present the different stages of the process – mixing of reactants, propagation of chemical reaction and refinement of the microstructure during ball-milling followed by composition change and phase coarsening during annealing. It will be explained with the concept of competition between thermal and forced processes. We will also identify the processing parameters which control it.

**10:30 AM**

**The Effect of Boron Addition on the Wear Resistance of High Chromium White Cast Iron**: *Cenk Saglam*[1]; Selim Ozavar[2]; Onuralp Yucel[1]; [1]Istanbul Technical University; [2]UMIT Casting

In this study, mechanical properties and wear resistance of high chromium white cast irons were investigated with varying boron concentration. Fe-Cr-C-Si-Mo-B alloys with varying boron concentration from 0.0 to 0.56 wt.% were prepared by pilot scale induction melting furnace. Effects of alloying elements and heat treatment methods on wear resistance were studied. For every chemical composition, a specimen for metallographic research was prepared. Microstructure of the specimens was studied by optical microscope. Quantitative elemental analysis was performed by using XRD, XRF, EPMA and AAS. Moreover, micro hardness measurements were made on different cross-section areas and wear resistance of the specimens was measured by using ball-cratering methods. It has been concluded that with the increasing of boron content, the hardness increases and the tensile strength decreases. The wear resistance is improved up to 0.1 wt.% B concentration. Above 0.1 wt.% B, there is a decrease in wear resistance.

**10:50 AM**

**Novel Current Activated Tip-Based Sintering (CATS) of Advanced Materials**: *K. Morsi*[1]; K. Moon[1]; S. Kassegne[1]; R. Ugle[1]; M. Patel[1]; [1]San Diego State University

This paper discusses the Current-activated Tip-based Sintering (CATS) of intermetallic and metallic powder-based materials. In CATS electric current is applied to a powder bed/compact using a small stationary or moving conductive tip to selectively sinter macro- or micro-scale features. The effect of processing variables on the microstructure and properties of metallic and shape memory alloy (TiNi) are discussed.

**11:10 AM**

**Comparison of Microstructural Evolution of Nickel during Conventional and Spark Plasma Sintering**: Matthew Luke[1]; Darryl Butt[1]; *Megan Frary*[1]; [1]Boise State University

Spark plasma sintering (SPS) is a rapid powder consolidation technique that uses pulsed electric current to directly heat the powder. A comparison was made between the microstructural evolution of pure polycrystalline nickel powder processed by conventional (press and sinter) and SPS routes. The sintering temperature, dwell time, applied pressure and ramp rate were varied to affect the densification and microstructural evolution of the powders. Characterization of grain size, morphology, grain boundary character, and crystallographic texture was performed on SPS and traditionally-processed specimens using electron backscatter diffraction. The microstructure of SPS nickel was observed to vary between one that resembles a green body with some grain coarsening to one that is indistinguishable from a wrought nickel microstructure except for some porosity. Grain size distributions in press and sintered nickel were much wider than those for SPS nickel. SPS nickel has increased porosity at the edges, evidence of processing temperature variations.

**11:30 AM**

**Secondary Cooling Technology for Casting of Hypo-Peritectic Steels**: Jian Zhang[1]; Chen Dengfu[1]; *Long Mujun*[1]; Wang Shuigen[1]; Bi Yanyan[1]; [1]Chongqing University

From the standpoint of quality, surface cracks normally pose more of a problem than internal cracks. Hypo-peritectic steel grades are well known for their proneness to surface longitudinal cracking during continuous casting. This study analyzed the influences of chemical composition, technique parameter and secondary cooling on the surface longitudinal cracks. Based on the optimized the first and secondary cooling zone water distribution in the process of continuous casting, low cooling water distribution at solidification initial stages and uniform cooling at secondary cooling zone were suggested, which can be use to effectively control the surface longitudinal cracks.

**11:50 AM**

**Investigation of a "Swirling" Phenomenon in Tungsten Carbide-Cobalt during Laser Deposition Using In-Situ Thermal Imaging**: *Yuhong Xiong*[1]; William Hofmeister[2]; John Smugeresky[3]; Jonathan Nguyen[1]; Jean-Pierre Delplanque[1]; Julie Schoenung[1]; [1]University of California; [2]University of Tennessee Space Institute; [3]Sandia National Laboratories

Laser deposition is used for the fabrication of net shapes from a broad range of materials, including tungsten carbide-cobalt (WC-Co) cermets (composites composed of a metallic phase and a hard refractory phase). The thermal behavior of WC-Co cermets during the Laser Engineered Net Shaping (LENS®) process was studied using an in-situ high-speed thermal imaging system. An interesting "swirling" phenomenon was observed in the molten pool, and was not present when the bottom of the sample was being built. To provide fundamental insight into this phenomenon for cermets, the thermal behavior of pure Co during the LENS® process was investigated for comparison. Temperature gradients and cooling rates in the vicinity of the molten pool for both material systems were analyzed. Their unique physical properties, such as wettability, emissivity, and viscosity at high temperature were considered to determine the source of the observed differences.

## Mechanical Performance for Current and Next-Generation Nuclear Reactors: Advances in Mechanical Testing

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Nuclear Materials Committee
*Program Organizers:* Dylan Morris, NIST; Greg Oberson, Nuclear Regulatory Commission; Nicholas Barbosa, National Institute of Standards & Tech; Wolfgang Hoffelner, Paul Scherrer Institute

Monday AM Room: 201
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Nicholas Barbosa, National Institute of Standards and Technology; Peter Hosemann, LANL

**8:30 AM Invited**
**Small Specimen Test Techniques for Evaluating Properties of Irradiated Materials**: *Mikhail Sokolov*[1]; [1]ornl

Small specimens are playing the key role in evaluating properties of irradiated materials. The use of small specimens provides several advantages for researchers. Typically, only small volume of material can be irradiated in a reactor at desirable conditions in terms of temperature, neutron flux, and dose. Small volume of irradiated material may also allow for easier handling of specimens. However use of small specimens imposes variety of challenges as well. These challenges are associated with proper accounting for size effects and transferability of small specimen data to the real structures of interest. In this presentation variety of small specimens techniques will be reviewed. Main interest will be focused but not limited on tensile, fracture toughness, creep, and Charpy impact tests.

**9:00 AM**
**Micro and Macro Scale Mechanical Testing and Characterization on Irradiated Structural Materials for Nuclear Application**: *Peter Hosemann*[1]; Manuel Pouchon[2]; Yong Dai[2]; Stuart Maloy[1]; [1]LANL; [2]Paul Scherrer Institute

Testing of reactor irradiated materials for nuclear applications (fission and fusion) are cost and labor intensive tasks. Large scale materials testing means that large amounts of highly radioactive materials must be handled. Using small scale materials testing can reduce the amount of active material and allow for relatively inexpensive lower energy irradiations.Here we present micro compression testing and nanoindentation results performed on spallation source (neutron) and ion beam irradiated engineering materials such as HT-9 (12Cr ferritic/martensitic) and F82H (8Cr 2W ferritic/martensitic). It was found that the trend of radiation induced hardening measured with small scale techniques is the same as observed with large scale tensile testing. Measurements performed on nanostructured ODS alloys such as MA957 (14Cr ODS alloy) and PM2000 (20Cr 5Al ODS alloy) show that the yield strength measured using small scale techniques is equal to data obtained with large scale testing techniques.

**9:20 AM**
**Microscale Methods for Evaluating Mechanical Behavior of Ion Irradiated Metals at High Damage Levels**: *Luke Brewer*[1]; Khalid Hattar[1]; Brad Boyce[1]; Joseph Michael[1]; [1]Sandia National Laboratories

Next generation nuclear power reactors will push materials to radiation damage levels beyond 100 displacements per atom. It is vital to understand how the mechanical behavior of structural metals changes at these damage levels. High energy, heavy ion accelerators are able to produce these damage levels in metals, but only in small volumes. In this talk we will extend current and introduce new microscale methods for assessing the change in micromechanical behavior of metals irradiated by high energy, heavy ions. We will present data that extends the recent work using micropillar compression of ion irradiated metals, including work on copper single crystals which is allowing an examination of the accuracy and precision of these methods. These results will be compared to spherical indentation experiments on the same materials. In addition, we will present novel in situ experiments to monitor swelling by using an SEM attached to a tandem accelerator.

**9:40 AM Invited**
**Small Specimen and in situ Mechanical Test Methods in the US Fusion Reactor Materials Program**: *Roger Stoller*[1]; G. Robert Odette[2]; Richard Kurtz[3]; Mikhail Sokolov[1]; Yutai Katoh[1]; Thak Sang Byun[1]; Anton Moeslang[4]; [1]Oak Ridge National Laboratory; [2]University of California; [3]Pacific Northwest National Laboratory; [4]FZK Karlsruhe

The development of small specimens for mechanical testing has been a priority for the US Fusion Materials Program for several practical reasons. High-flux volume available for materials irradiation is relatively limited, and a large number of specimens is required to map a material's response to irradiation. Smaller specimens reduce the amount of radioactive material, minimizing personnel exposures and waste disposal. Notably, design-relevant data for fusion must ultimately be obtained in a prototypical 14 MeV neutron source such as the International Fusion Materials Irradiation Facility. The high-flux volume of IFMIF will be limited, leading the international community to devote considerable effort to refining experimental matrices using small specimens. The history and status of this and related work will be discussed, including a comparison of small and conventional specimens to demonstrate their validity and value to the development of advanced reactors.

**10:10 AM Break**

**10:25 AM Invited**
**Mechanical Testing of Core Fast Reactor Materials for the Advanced Fuel Cycle Initiative**: *Stuart Maloy*[1]; Tobias Romero[1]; Mychailo Toloczko[2]; Thak-sun Byun[3]; [1]Los Alamos National Laboratory; [2]PNNL; [3]ORNL

The Advanced Fuel Cycle Initiative is investigating methods of burning minor actinides in a transmutation fuel. To achieve this goal, the core fast reactor materials (cladding and duct) must be able to withstand significant radiation damage (greater than 200 dpa). Developing these radiation tolerant materials requires high dose irradiation effects data on relevant materials. These data should include tensile, fracture toughness and creep data after irradiation to doses greater than 200 dpa at irradiation temperatures of 350-600°C. To obtain this data in the near term, tensile, fracture toughness, Charpy specimens and in-reactor creep specimens of traditional ferritic/martensitic alloys (HT-9 and T91) previously irradiated in the Fast Flux Test Facility (FFTF) to doses up to 210 dpa at irradiation temperatures from 350-700°C are being tested and analyzed. This includes analysis of a duct made of HT-9 after irradiation to a total dose of 155 dpa at temperatures from 370 to 510°C.

**10:55 AM**
**Damage Related Information Contained in Small Material Volumes of Advanced Nuclear Plants**: *Wolfgang Hoffelner*[1]; Manuel Pouchon[1]; Jiachao Chen[1]; Maria Samaras[1]; [1]Paul Scherrer Institute

Condition based residual life assessments are important for plants and operating conditions where not enough service experience exists. This will be also the case for advanced nuclear power plants. Investigations on service exposed material (taken either directly from a component or from surveillance coupons) are considered as one possible option particularly for damage types where current NDE methods are not accurate enough (creep, cyclic softening, microstructural changes etc.). Advanced micro-sample testing (indentation, micro-pillars, indentation creep, punch test etc.) together with microstructural analyses and materials modeling can help to increase the accuracy of damage and residual life assessments. The paper will discuss possibilities of getting damage related information from small sample volumes for advanced nuclear plants. Test results from micro-samples and it's interpretation will be shown.

**11:15 AM**
**Studying the Effect of Carbon on DU-Mo Foil Fabrication Using Small-Scale Specimen Testing**: *Ramprashad Prabhakaran*[1]; Douglas Burkes[1]; Amy DeMint[2]; Jack Gooch[2]; Dennis Keiser[1]; Daniel Wachs[1]; Indrajit Charit[3]; [1]Idaho National Laboratory; [2]Y-12 National Security Complex; [3]University of Idaho

In support of the Reduced Enrichment for Research and Test Reactors (RERTR) program, efforts are ongoing to develop and validate a monolithic depleted uranium molybdenum (DU-Mo) foil fabrication process adaptable for a potential implementation in a manufacturing environment. Efforts are ongoing to study the effect of carbon (source material impurity) on DU-Mo foil fabrication. Carbon is of particular interest due to its potential effects on the metalworking process. Hence, DU-10Mo test foils were fabricated with different carbon levels (150-400 ppm). Small-scale specimen testing (Sub-size tensile, shear punch and microhardness testing) along with optical metallography, SEM and XRD are

being conducted to understand the effect of carbon on the fabrication reliability and process yields. Thus, this study would help in finding out a tolerable limit of carbon content in the alloy.

**11:35 AM**

**Mechanical Properties of Fresh and Irradiated Monolithic U-Mo Fuels**: *Ramprashad Prabhakaran*[1]; Douglas Burkes[1]; Dennis Keiser[1]; Daniel Wachs[1]; Adam Robinson[1]; Jan-Fong Jue[1]; Indrajit Charit[2]; [1]Idaho National Laboratory; [2]University of Idaho

The Reduced Enrichment for Research and Test Reactors (RERTR) program was initiated to develop new nuclear fuels to enable research and test reactors to use low-enriched uranium instead of high-enriched uranium fuels without significant loss in performance. This has led to a pursuit of developing high uranium density monolithic fuel that possesses the greatest possible uranium density in the fuel region. Hot-isostatic pressing and friction bonding are being investigated to encapsulate fuel foils in Al alloy cladding. The fabrication technique significantly affects the mechanical properties of the foil and it also dominates the overall plate mechanical behavior. Sub-size tensile and microhardness testing are being performed to evaluate the mechanical properties of the fresh fuel and fuel irradiated at the Advanced Test Reactor. Efforts are ongoing to standardize the shear punch testing procedure so that the tensile behavior can be predicted using small volumes of material and at reduced irradiation costs.

**11:55 AM**

**Structural Modifications and Mechanical Degradation of Ion Irradiated Glassy Polymer Carbon**: *Malek Abunaemeh*[1]; Mohammad Seif[1]; Lumin Wang[2]; Ibidapo Ojo[1]; Young Yang[3]; Claudiu Muntele[1]; Abdulla Elsamadicy[4]; Daryush ILA[1]; [1]Alabama A&M University; [2]University of Michigan; [3]University of Wisconsin; [4]University of Alabama in Huntsville

The TRISO fuel consists of UOx coated in several layers of materials with different functions. Here we are looking at ion irradiation induced structural modifications of glassy polymeric carbon (GPC) and the effect on mechanical and physical properties. GPC is one material considered as a potential replacement for pyrolytic carbon coatings, with a function of diffusion barrier for the fission products. Here we irradiated GPC with 5 MeV Ag and Au. The fission fragment mass distribution has two maxima around mass 98 and 137 that would best fit Rb and Cs, respectively. However, both ions are hard to produce from our SNICS source, therefore we chose Ag and Au as best replacements. We used scanning electron microscopy, transmission electron spectroscopy, and nano-indentation for characterization. We were able to correlate the imaging results with SRIM simulations of ion range and distribution, energy loss, and damage to the GPC material.

## Modeling, Simulation, and Theory of Nanomechanical Materials Behavior: Plasticity and Strength of Nanostructured and Nanoscale Materials I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Nanomechanical Materials Behavior Committee

*Program Organizers:* Thomas Buchheit, Sandia National Laboratories; Sergey Medyanik, Washington State Univ.; Douglas Spearot, University of Arkansas; Lawerence Friedman, Penn State University; Edmund Webb, Sandia National Laboratories

Monday AM Room: 304
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Douglas Spearot, University of Arkansas; Stephen Foiles, Sandia National Laboratories

**8:30 AM Invited**

**Molecular Dynamics Simulation of the Deformation of an Equilibrated Nanograined Metal**: *Stephen Foiles*[1]; [1]Sandia National Laboratories

Molecular dynamics (MD) simulations of grain growth of nanograined metals indicate the presence of a high density of twin boundaries in the resulting microstructure. This is consistent with experimental observations of the post-annealing structure of pulsed laser deposited Ni. In this work, an initial structure is obtained via the annealing of an initial nano-scale grain structure. The tensile deformation of this system is then computed via MD simulations. The resulting deformation mechanisms are examined to elucidate the influence of the twin boundaries on the deformation mechanism. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC0494AL85000.

**9:00 AM**

**Analysis of Dislocation Twin-Interaction in Deformed Nanocrystalline fcc Metals**: *Karsten Albe*[1]; Alexander Stukowski[1]; Diana Farkas[2]; [1]TU Darmstadt; [2]VirginiaTech

The strengthening effect of twins in nanocrystalline metals has been reported both in experiment and simulation. While twins are mostly considered as effective barriers to slip transfer of dislocations, they can also provide nucleation sites for dislocations or migrate during the deformation process, contributing to plasticity. Here we present a comparative atomistic study of the effect of twins on the deformation behavior of nanocrystalline Cu, Al and Pd. A new analysis method based on an automated Burgers circuit is applied, which allows to analyze the dislocation interaction with twin planes and grain boundaries. While Cu shows hardening from the twins, Pd and Al show the opposite effect. The results provide evidence how the dissociation widths affectsthe slip transfer across the twin boundary and why twin planes provide additional dislocation sources, which is the main reason for softening of these samples as compared to identical nanocrystals without twins.

**9:20 AM**

**Dislocation Nucleation and Starvation in Metallic Nanowires**: *Scott Mao*[1]; A. Cao[2]; Y Wei[2]; [1]University of Pittsburgh; [2]Institute of Mechanics

The mechanical behavior of bulk metals is usually characterized as smooth continuous plastic flow following by yielding. Here we show, by using molecular dynamics simulations that the mechanical deformation behaviors of single-crystalline nickel and copper nanowires are quite different from their bulk counterparts. Correlation between the obtained stress-strain curves and the visualized defect evolution during deformation processes clearly demonstrates that a sequence of complex dislocation slip processes results in dislocation starvation, involving dislocation nucleation, propagation and finally escaping from the wire system, so that the wires deformed elastically until new dislocation generated. This alternating starvation of dislocations is unique in small-scale structures. Furthermore, the magnitude of yield stress of these nanowires is strongly dependent of the wire size.

**9:40 AM**

**Dislocation Nucleation from Grain Boundaries in Nanocrystalline Pd and Pd-Au Studied by Molecular Dynamics Simulations**: *Jonathan Schaefer*[1]; Alexander Stukowski[1]; Karsten Albe[1]; [1]TU Darmstadt

Molecular dynamics simulations on the nucleation and absorption of dislocations at grain boundaries are presented for nanocrystalline Pd and Pd-Au with grain sizes exceeding 15nm. Initial structures are generated with the Voronoi tessellation method and alloyed systems are created via a Monte-Carlo simulation in the semi-gandcanonical ensemble. A novel approach that allows to identify dislocation lines within the grains and grain boundaries from atomistic data is introduced and applied.Nucleation processes are analyzed with respect to the local stresses, grain boundary structure, alloy composition and relaxation state of the initial structure.

**10:00 AM**

**Orientation Dependent Plasticity in Metal Nanowires under Torsion**: *Christopher Weinberger*[1]; Wei Cai[1]; [1]Stanford University

Plasticity at small scales is more heterogeneous and intermittent than in the bulk which may lead to the limitation of plastic metal forming at the micro- and nanoscales. In order to better understand the plastic response of metals at these scales and the effects of strain gradients, we investigate the orientation dependent plasticity in pristine metal nanowires under torsion using molecular dynamics and dislocation dynamics simulations. When the wires are oriented along the <110> direction, coaxial dislocations nucleate, creating a homogeneous plastic deformation which is similar to the Eshelby twist problem. However, in the <111> and <100> direction, plasticity occurs through the formation of twist boundaries, which localizes the plastic deformation. These results illustrate that plastic deformation in the presence of strain gradients can be strongly influenced by crystal orientation.

**10:20 AM Break**

**10:40 AM Invited**

**Slip Transmission Mechanisms for Glide Dislocations across Dissimilar Metallic Interfaces**: *Jian Wang*[1]; Richard Hoagland[1]; John Hirth[1]; Amit Misra[1]; [1]LANL

Using atomistic modeling and anisotropic elastic theory, we have studied slip transmission for glide dislocations across dissimilar metallic interfaces in a model Cu-Nb system. The Cu-Nb interfaces have relatively low shear strength and are referred to as 'weak' interfaces. Our work shows that such interfaces are very strong traps for glide dislocations and thus, effective barriers for slip transmission, because the weak interface is readily sheared under the stress field of an impinging glide dislocation, resulting in an attractive force on the glide dislocation and leading to the absorption of dislocation in the interface. Besides the trapping of dislocations in weak interfaces, we also discuss geometric factors such as the crystallographic discontinuity of slip systems across the Cu/Nb interfaces, which contribute to the difficulty of dislocation transmission across an interface. The implications of these findings to the unusually high strengths experimentally measured in Cu/Nb nanolayered composites are discussed.

**11:10 AM**

**Molecular Mechanics Simulation of Plastic Deformation in Nanoscale FCC-BCC Multilayered Metallic Composites**: *Shuai Shao*[1]; Sergey Medyanik[1]; [1]Washington State University

A significant increase in strength can be often observed in multilayered metallic composites when the individual layer thickness is decreased from microscale to nanoscale. Recently, a number of atomistic simulation studies have been conducted to investigate nanoscale metallic bilayers with different types of interfaces. However, the case with incoherent interfaces focused mainly on studying the interaction of single artificially introduced dislocations with the interface under two-dimensional loading conditions. In this work, molecular mechanics simulations are applied to study dislocation nucleation and propagation in a Cu-Nb bilayer with an incoherent FCC-BCC interface under three-dimensional loading conditions due to indentation. Indenter is applied to generate dislocations at and near the surface from each side of the bilayer (in separate simulations). The role which incoherent interfaces play in the strengthening of multilayered metallic composites is explored. Mechanisms of interactions between gliding dislocations and the incoherent interface are identified and studied in detail.

**11:30 AM**

**Shock Response of Cu-Nb Nanolayer Composites**: *Timothy Germann*[1]; Shengnian Luo[1]; Nathan Mara[1]; [1]Los Alamos National Laboratory

Large-scale classical molecular dynamics (MD) simulations and laser-driven shock wave experiments (both direct laser drive and laser-launched miniflyer plates) have been used to study the response of Cu-Nb nanolayered composites to shock compression. At a layer thickness of 5 nm, the hardness of such metallic multilayers (as measured by quasistatic indentation or compression tests) reaches a maximum due to the difficulty of dislocation transmission across the interfaces. We observe a similar strengthening effect under dynamic shock loading, both in the MD simulations and in post mortem examinations of shock-recovered samples subjected to ~20 GPa shock loading. The MD simulations provide insight into the dislocation nucleation and transmission processes that occur under compression, as well as the subsequent annihilation upon release.

**11:50 AM**

**Yield Strength in Nanocrystalline Cu during High Strain Rate Deformation**: *Nhon Vo*[1]; Robert Averback[1]; Pascal Bellon[1]; Alfredo Caro[2]; [1]University of Illinois, Urbana-Champaign; [2]Lawrence Livermore National Laboratory

We observe in molecular dynamics a nearly linear relationship between the yield strength in nanocrystalline Cu during high strain rate deformation and the fractional grain boundary (GB) volume. Specimens were prepared with grain sizes between 2.5 to 20 nm and with different grain configurations. They were subsequently annealed to reduce the fraction of grain boundary volume, but without significantly changing their grain sizes. The yield strengths of the samples were then calculated for uniaxial compression at strain rates of 1x10^10 1/s and 1x10^9 1/s. For each strain rate, the observed strength was found to scale with the fractional number of GB atoms in the sample. This observation suggests a new scaling behavior for the onset of plasticity in nanocrystalline materials, controlled not by the grain size alone, but by a combination of both grain size and degree of GB relaxation, as measured by the grain boundary volume.

**12:10 PM**

**A Quantized Crystal Plasticity Model for Nanocrystalline Metals: Dislocation Source Strengths and Internal Stress**: Lin Li[1]; Myoung-Gyu Lee[2]; *Peter Anderson*[1]; Steven Van Petegem[3]; Helena Van Swygenhoven[3]; [1]The Ohio State University; [2]Korea Institute of Materials Science; [3]Paul Scherrer Institute

A quantized crystal plasticity (QCP) model is used to study several distinctive deformation features of nanocrystalline (NC) metals [1]. A key model feature is that the average plastic strain evolves in a stepwise fashion, as seen in MD simulations involving dislocation slip across grains. The model reproduces several experimental features of NC metals, including an enhanced flow stress, an extended elasto-plastic transition, large recoverable strain and reversible peak broadening upon unloading, and even the evolution of lattice strains with deformation. These successful predictions occur when the grain-to-grain distribution of source strengths for plasticity has certain features, and only for certain pre-deformation histories. The experiment-model comparison therefore provides an approach to determine some fundamental deformation characteristics of NC metals.[1] Li L, et. al.. Acta Materialia 2009;57:812.

## Neutron and X-Ray Studies of Advanced Materials III: Strain and Dislocation Gradients from Microdiffraction I

*Sponsored by:* The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Titanium Committee
*Program Organizers:* Rozaliya Barabash, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Erica Lilleodden, GKSS Research Center; Peter Liaw, University of Tennessee; Yandong Wang, Northeastern University

Monday AM Room: 303
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Rozaliya Barabash, ORNL; Hongbin Bei, Oak Ridge National Laboratory

**8:30 AM Keynote**

**Imaging Methods for Mapping Orientations, Plastic Strain and Stresses in Grains**: *Henning Poulsen*[1]; [1]Risoe DTU

3DXRD microscopy is an emerging technique for characterizing individual grains in 3D. In favorable cases, the position, shape, orientation and stress state of thousands of grains can be determined in situ during processing. The status of the technique is outlined and four new functionalities are highlighted: 1) mapping of type-II stresses 2) orientation imaging of deformed materials 3) combined tomography-3DXRD techniques and 4) 3D maps of the local slip amplitudes. Examples of use are presented from the areas of polycrystal deformation, nucleation, recrystallisation, and grain growth.

**9:00 AM Invited**

**Using High Energy X-Ray Experiments and Crystal-Based Simulations to Understand Micromechanical Deformation Behavior of Metallic Polycrystals**: *Matthew Miller*[1]; Paul Dawson[1]; Christos Efstathiou[1]; Donald Boyce[1]; Ulrich Lienert[2]; [1]Cornell University; [2]Advanced Photon Source

The mechanical behavior of a crystal within a loaded aggregate is complicated by anisotropic properties and complex microscale loading conditions. Our approach for understanding this behavior consists of high fidelity micromechanics-based simulations together with a new class of synchrotron x-ray diffraction experiments to determine mechanical properties and performance measures for polycrystalline structural alloys. The stress determination experiments are part of the High Energy Diffraction Microscopy (HEDM) suite of capabilities at the Advanced Photon Source (APS) beamline 1-IDC. The polycrystal model begins with anisotropic elasticity and restricted slip plasticity. Each crystal is discretized with finite elements. The initial orientations are specified by matching the measured crystallographic texture. This talk describes experiments and simulations on beta 21s, a bcc titanium alloy. Measured lattice strains are compared to simulation results. Since no

results exist in the literature, the first step in the analysis was the determination of the single crystal elastic moduli.

**9:20 AM Invited**

**Three Dimensional X-Ray Scanning Micro/Nano-Diffraction Probe for High-Pressure Research**: *Wenjun Liu*[1]; Ho-kwang Mao[2]; Wenge Yang[2]; Yang Ding[2]; Lin Wang[2]; Przemyslaw Dera[2]; Gene Ice[3]; Paul Zschack[1]; [1]Argonne National Laboratory; [2]Carnegie Institution of Washington; [3]Oak Ridge National Laboratory

During the past decade, substantial progress has been made in the research and development of x-ray focusing optics at synchrotron facilities. Submicron focused beams are now in routine operation and sub-hundred nanometer spots have been achieved. However, these advances have not yet been well applied to high-pressure research. We now have the opportunity for major breakthroughs that will fundamentally change the diamond anvil cell (DAC) technology using this more than order-of-magnitude improvement of beam size. 3D x-ray diffraction microscopy developed at 34-ID beamline at the Advanced Photon source has been proved to be a powerful tool for providing detailed local crystal structural information of crystallographic orientation, strain tensor, and lattice structure in materials, as well as materials at high-pressure. Preliminary experiments on adopting DAC at submicron focused beamline for structure determination by single-crystal diffraction are presented. Related instrumentation issues to high-pressure research problems will be also discussed.

**9:40 AM**

**A Facility for μXRD, μXRF and μXAS at the Canadian Light Source**: *Renfei Feng*[1]; Morgan Bradford[1]; Stewart McIntyre[2]; [1]Canadian Light Source; [2]University of Western Ontario

VESPERS is a newly commissioned beamline at the Canadian Light Source. It is a hard X-ray microprobe beamline dedicated to X-ray micro-diffraction (μXRD), X-ray micro-fluorescence (μXRF), and micro X-ray absorption spectroscopy (μXAS) studies. The beamline offers four widely differing bandwidths, ~0.01%, ~1.6%, ~10%, and fully polychromatic beam, to simplify the Laue diffraction analysis, to optimize XRF excitation, and to enable X-ray absorption spectroscopy measurement. The energy range provided by the beamline covers 6-30 keV. The beamline and its current status will be described and some applications will be presented.

**9:55 AM Invited**

**Size Effects in Plasticity Investigated by In Situ Laue Diffraction**: *Steven Van Petegem*[1]; Helena Van Swygenhoven[1]; [1]Paul Scherrer Institut

Microcompression of micron-sized pillars is a promising tool which allows evaluating mechanical properties of materials via specimens with sizes in the range of micrometers. In order to investigate the microstructural evolution during deformation a new in-situ testing technique was developed at the Swiss Light Source. Here, micron-sized single crystal pillars are illuminated by a micron-sized pink x-ray beam during continuous deformation. The evolution of the position and shape of the diffraction spots is directly linked to changes in local crystallographic orientation and strain gradients. Remarkable differences in the initial microstructure of single crystal micropillars prepared from the same bulk material were found. Furthermore we show that initial plastic deformation is controlled by the boundary constraints of the test. It is anticipated that this new in situ technique has great potential to gain insight into the deformation mechanisms in more complex structures, providing essential input for predictive plasticity models.

**10:15 AM Invited**

**Residual Stress Effects on the Phase-Specific Strains in Directionally-Solidified Nial-Mo Composite under Thermal and Mechanical Loading**: *Hongbin Bei*[1]; R Barabash[2]; Easo George[2]; Gene Ice[1]; [1]Oak Ridge National Laboratory; [2]Oak Ridge National Laboratory and University and Tennessee

Neutron diffraction measurements and model calculations suggest that, in directionally-solidified NiAl-Mo composites, stresses develop between the NiAl and Mo phases because of thermal expansion mismatch. It is known that such residual stresses play important roles in the thermal and mechanical responses of individual phases in a composite material. In this study, three-dimensional (3D) spatially resolved strain distributions in the NiAl matrix and the ~550–1000 nm Mo fibers of a NiAl–Mo eutectic were investigated by microbeam x-ray diffraction. For embedded Mo fibers, the measured elastic strain is consistent with the predicted thermal mismatch strain between the NiAl and Mo phases. However, when the NiAl matrix is etched the d-spacing of the exposed Mo micropillars, increases to that of unconstrained Mo, indicating release of the residual strain in the Mo fibers. We will discuss how the residual stress influences the phase-specific strain response in a NiAl–Mo composite under thermal and mechanical loading.

**10:35 AM Break**

**10:45 AM**

**X-Ray Micro Beam Probing Pre-Strain Effects on Dislocation and Strain Gradients in the NiAl-Mo Composite**: *Rozaliya Barabash*[1]; H Bei[2]; Y. Gao[1]; G. Ice[2]; E. George[1]; [1]Oak Ridge National Laboratory and the University of Tennessee, Knoxville; [2]Oak Ridge National Laboratory

3D spatially-resolved polychromatic microdiffraction was applied to probe the depth-dependent dislocation density and strain gradients in separate phases of a NiAl –Mo composite. Dislocation density and strain gradients are investigated as a function of pre-strains. It is found that pre-strain changes the stress state as compared to the as grown composite. In as grown composites, due to the thermal expansion mismatch, the Mo phase is under compression and the NiAl phase is in tension. After pre-straining, the Mo phase is in tension and NiAl matrix is under compression, which can be understood from their mismatch in yield strain and the elastic constraints during unloading. The dislocation density in both phases is found to grow with increased pre-straining. These measurements provide dislocation information to recent directional solidified Mo micro-pillar testing.

**11:00 AM Invited**

**Synchrotron X-Ray Microdiffraction for the Study of Micromechanics of Materials at Nanoscale**: *Nobumichi Tamura*[1]; Martin Kunz[1]; Kai Chen[1]; [1]Lawrence Berkeley National Lab.

The dedicated x-ray microdiffraction beamline BL12.3.2 at the Advanced Light Source is opened to the user community since 2007 while additional commissioning has been completed in 2008. Providing both monochromatic and polychromatic focused x-ray beam from a superbend source in the energy range of 5-22 keV, the beamline is especially well suited for spatially resolved measurements of strain in micro-devices and phase identification in heterogeneous samples. The sample can be scanned under the focused beam and 2D x-ray diffraction patterns as well as fluorescence data are collected at each step in an automated fashion to provide for grain orientation, phase distribution and strain/stress distribution map of the sample with micron to submicron resolution. We will review some of the applications from this beamline including measurements of stress in copper interconnects and lead-free solder joints, study of tin whisker growth, compression test in nanopillars and domain switching in ferroelectric materials.

**11:20 AM Invited**

**Spatially Resolved Elastic Strains within Bulk Dislocation Cell Structures: What Next?**: *Lyle Levine*[1]; Bennett Larson[2]; Peter Geantil[3]; Jon Tischler[2]; Michael Kassner[3]; Wenjun Liu[4]; [1]National Institute of Standards and Technology; [2]Oak Ridge National Laboratory; [3]University of Southern California; [4]Advanced Photon Source

Over the past several years, we have used depth-resolved, submicrometer X-ray beams to directly measure the axial elastic strains within dislocation cell interiors and cell walls in plastically deformed copper single crystals. Although these studies have settled long-standing questions about the long-range stresses in heavily deformed metals, other critical issues remain both unresolved and largely unaddressed in the literature. For example, the usual picture of high dislocation density cell walls surrounding dislocation-free cell interiors comes primarily from TEM measurements of thin foils, even though it is well established that a substantial fraction of the dislocation content is lost during sample thinning. Also, most work on stresses within dislocation cell structures, including our own, looks only at axial stresses and doesn't address the full tensorial nature of the elastic strain. We will discuss our progress in tackling both of these problems using depth resolved submicrometer X-ray diffraction.

**11:40 AM Invited**

**In-situ X-Ray Diffraction of Brittle and Ductile Nanostructured Materials**: *Ralph Spolenak*[1]; [1]ETH Zurich

The combination of several in-situ methods can shed additional light on deformation and failure mechanisms in nanostructured materials. In the current study in-situ X-ray diffraction studies at synchrotron light sources are combined with in-situ SEM and Raman spectroscopical observations. In

this context the scaling behavior in fracture and delamination of Ta thin films on polyimide substrates, the fracture of natural zirconia on Zr alloys and the fracture of ductile Au and Al thin film alloys is studied. Both brittle and ductile crack patterns can be correlated to the corresponding stress transfer between substrate and thin film. A further extension of this concept is presented when Raman spectroscopy is utilized to locally measure the stress evolution in nacre, a natural nanocomposite, and correlating it to the macroscopic deformation behavior as obtain by diffraction. Fracture toughness and strength is correlated to the microstructure of different sea shells.

**12:00 PM Invited**

**Real, Orientation and Reciprocal Space Coverage of 3DXRD at the APS 1-ID Beamline**: *Ulrich Lienert*[1]; [1]ANL

The three-dimensional x-ray diffraction (3DXRD) technique employs high energy synchrotron radiation and area detectors for the structural characterization of polycrystalline bulk materials. The 'tomographic' data acquisition provides the temporal resolution for in situ investigations. Within the high energy diffraction microscopy (HEDM) program at the APS 1-ID beamline near-, farfield, and high reciprocal space resolution variants of the technique are developed. Unique capabilities of these variants are grain boundary mapping, high strain sensitivity, and dislocation structure sensitive peak profile measurements, respectively. By combining these techniques the real, orientation and reciprocal space coverage can be significantly extended. The status, future plans, and limitations will be presented and illustrated by selected case studies.

**12:20 PM**

**Inverse Analysis of Engineering Neutron Diffraction Data**: Seung-Yub Lee[1]; Youngshin Kim[1]; Hyuntae Na[1]; *Ersan Ustundag*[1]; [1]Iowa State University

Integration of engineering neutron diffraction (ND) data analysis and solid mechanics modeling offers a powerful approach to deduce in-situ constitutive behavior of materials. The basic approach is to perform an inverse analysis of ND data so that experimental strains fit predictions of the model by optimizing the model parameters that define the constitutive law. This presentation will outline recent results from the use of finite element analysis (FEA) and self-consistent modeling (SCM) in the interpretation of ND data. Various optimization algorithms (including least-square and artificial neural network analyses) were employed. The latter also allowed the study of the relative influence of various model parameters and yielded suggestions on optimum experiment design.

**12:35 PM**

**Compositional Effects on the Superelasticity of Gum Metal**: Russell Talling[1]; *David Dye*[1]; [1]Imperial College

The deformation mechanisms of the bcc Ti alloy, Gum metal (Ti-36Nb-2Ta-3Zr-0.3O, wt.%) have recently been the focus of much attention. This alloy possesses high strength, high elastic strain, low Young's modulus and high ductility. We have identified that Gum metal deforms via a reversible stress-induced phase transformation to the a" phase. This work has focuses on the effect of O and Sn on the micromechanics. In-situ synchrotron X-ray diffraction has been used to infer the shear modulus C', which gives an indication of the susceptibility of the bcc phase to transform. The texture of the martensite and bcc parent phase has been reconstructed at various points during tensile tests. We have also witnessed shape memory type loading behaviour in some Gum metal compositions, with evidence of martensite self-accommodation within the plateau region of the stress strain curve.

**12:50 PM**

**First ex-situ/in-situ Measurements of Strains/Stresses at Engineering Diffractometer VULCAN at SNS**: *Ke An*[1]; H.D. Skorpenske[1]; A.D. Stoica[1]; Dong Ma[1]; Ercan Cakmak[2]; Hahn Choo[2]; X.L. Wang[1]; [1]Oak Ridge National Laboratory; [2]The University of Tennessee

The engineering diffractometer VULCAN at the SNS started commissioning since June 26th in 2009. This instrument is designed for materials science and engineering studies. With VULCAN, users can not only conduct mapping of residual stresses, chemistry, microstructure and texture but also measure dynamically mechanical deformation response, material chemical properties change and microstructure evolution under dynamic loading (e.g. fatigue) or severe material processing (e.g., welding). With special sample environments, such as the vacuum furnace and unique multi-axial tension-torsion load frame, etc., VULCAN extends neutron scattering studies to broader areas of material science and engineering mechanics.

## Neutron and X-Ray Studies of Advanced Materials III: Structure from Diffraction

*Sponsored by:* The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Titanium Committee
*Program Organizers:* Rozaliya Barabash, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Erica Lilleodden, GKSS Research Center; Peter Liaw, University of Tennessee; Yandong Wang, Northeastern University

Monday AM Room: 613
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Emil Zolotoyabko, Technion

**8:30 AM Invited**

**Atomic Structure and Nano-Structure of Natural Bio-Composites**: *Emil Zolotoyabko*[1]; [1]Technion

Biomineralization process within living organisms results in producing the organic/inorganic composite materials with superior characteristics. This field continues to attract a lot of attention of many research groups worldwide. Organic macromolecules, supplied by organisms according to genetic program, play a crucial role in this sophisticated "processing". Our studies using high-resolution x-ray and neutron powder diffraction show a complexity of bio-minerals on the atomic and nano-scale due to interaction of organic macromolecules with ceramic crystallites. Based on systematic studies of natural calcium carbonate structures, we indicate three main structural features of biogenic crystals (as compared to their geological counterparts): anisotropic distortions of unit cells; bond lengths differences; unusual diminishing under annealing of the sizes of crystallite blocks which coherently scatter X-rays. All this is the result of local forces imposed on mineral crystallites by intra-crystalline organic macromolecules, the forces being strongly affected by mild annealing at 200-300 C.

**8:50 AM Invited**

**In-Situ Study of Time and Thickness Dependence of Crystallization of Amorphous $TiO_2$ Thin Films and Powders**: *Radomir Kuzel*[1]; Lea Nichtova[1]; Zdenek Matej[1]; Jindrich Musil[2]; [1]Charles University in Prague, Faculty of Mathematics and Physics; [2]University of West Bohemia in Pilsen

Numerous interesting properties of titanium dioxide, in particular photocatalytic activity, strongly depend on crystallization and phase composition. Annealing temperatures were selected below 220ºC where fast crystallization appears. Then the slow process allowed detailed time XRD in-situ investigations in laboratory conditions. Strong dependence of crystallization kinetics on the film thickness was found. The process could be well described by the Avrami equation modified by initial time of crystallization. The crystallization starts later and it is slow for very thin films (below 300 nm). Evolution of preferred orientation was observed, the (001) crystallites grow first but after complete crystallization the texture was usually weak. Line broadening was constant and small from the beginning of observable crystallization which was due to the rapid growth of a few crystallites. Tensile residual stresses were developed slowly during the crystallization and confirmed by detailed measurements at room temperature. They increase rapidly with decreasing film thickness.

**9:10 AM**

**Lattice Distortion Formations by Low Energy Ar+ Bombardment of Epitaxial Thin Films Grown on Silicone (100)**: *Paul Rozenak*[1]; [1]Hydrogen Energy Batteries LTD

Transmission electron microscopy (TEM) and X-ray characterization of lattice distortion forms, caused by low energy Ar+ bombardment of grown thin silicon films on silicon (001) substrate were studied. The isotropic case (of spherical distortions) takes place in epitaxial silicon "as grown" processes. The intensity distribution consists of two maxima, one from the distorted layer and the other from original un-effected silicon lattice. The significant changes in the 2θ location, peak broadening and integrated intensity from the (004)* reflections were obtained as the function of aging temperatures. First, aging

heat treatment, affects the distribution of distortions obtained from local regions at the "as grown" layer, which changes to a special topography of continued distortions at higher aging temperatures. At aging temperatures above 650ºC, this extra diffraction peak disappears. The TEM observations reveal the appearance of dislocation lines, with dark and bright contrasts around the lines and inter-dislocation strain contrasts.

**9:25 AM Invited**

**Voyaging around Nacre with the X-Ray Shuttle: From Bio-Mineralisation to Prosthetics via Mollusc Phylogeny**: *Daniel Chateigner*[1]; M. Morales[1]; L. Lutterotti[1]; [1]Ecole Nationale Supérieure d'Ingénieurs de Caen (ENSICAEN)

Bobbio (1970) analysed Maya cranes exhibiting nacre teeth with ante-mortem character, demonstrating the oldest signature of human implantology, using natural mollusc shells! The possibility of their osteointegration in human bone was born, in dentistry and prosthetics in general. Lopez (1980) promoted this idea further, demonstrating that natural nacres are not only biocompatible but osteoinductive. These two fascinating discoveries gave rise to tremendous research developments at the interfaces of several scientific disciplines opening the following questionings: - are all natural shells implantable? can we use shell spares from mollusc farming? - can we detect which shell microstructures, structures ... are the best appropriated to a given medical application? can we detect some differences between natural nacres? - can we synthetically reproduce biogenic-like nacre? - can we produce nacre-like bulks or cover prostheses? can we implant them? We illustrate these questionings using x-ray investigations.

**9:45 AM Invited**

**Materials Characterization Using the Hard X-Ray Nanoprobe Beamline at Argonne National Laboratory**: *Jörg Maser*[1]; Martin V. Holt[1]; Robert P. Winarski[1]; Volker Rose[1]; Peter Fuesz[1]; Gregory Brian Stephenson[1]; [1]Argonne National Laboratory

The Center for Nanoscale Materials' Hard X-ray Nanoprobe (HXN) at the Advanced Photon Source is designed to characterize composition and structure of nanoscale materials and devices with high spatial resolution [1]. The HXN uses hard x-rays with a photon energy between 3 and 30 keV, taking advantage of the good penetration of hard X-rays to study buried structures and interfaces, and perform trace element analysis of elemental constituents of the sample with high sensitivity. X-ray diffraction is used to identify crystallographic phase, strain and structure in novel materials and devices. In addition, the HXN integrates a tomographic full-field transmission mode that is used to characterize the 3-dimensional structure of complex systems and devices. All operation modes can be changed in-situ. The HXN takes advantage of the good penetration of hard X-rays to study buried structures and interfaces. The HXN uses diffractive optics to allow specimen characterization at highe spatial resolution. In scanning probe mode, an incident, spatially coherent wave front is focused by a Fresnel zone plate to a diffraction limited probe with a size of 50 nm and below. X-ray excited x-ray fluorescence photons and scattered photons are collected by energy dispersive and area detectors. In full-field transmission mode, the sample is illuminated using a capillary condenser system; a magnified image of the sample with a spatial resolution of 30 nm is created by an imaging zone plate downstream of the sample. 3-D images are obtained by rotating the sample for tomographic data acquisition. The HXN is optimized for the study of novel materials, such as ferroelectrics, complex oxides, thin films with novel structural and electronic/magnetic properties, and devices that utilize these properties. Typical applications include Si-based materials and devices, as well as novel materials and materials systems for potential use in energy applications, actuation, switching. The high trace element sensitivity is also useful for characterizing metal distributions in biological systems such as cells and tissues. We will give an overview of the HXN system and its characterization capabilities, and will present initial scientific applications. References: [1] J. Maser, G.B. Stephenson, D. Shu, B. Lai, S. Vogt, A. Khounsary, Y. Li, C. Benson, G. Schneider, "Conceptual design for a hard x-ray nanoprobe beamline with 30 nm resolution," AIP Conf. Proc., 705, AIP (2004), 470 - 473. [2] H. Kang, H. Yan, R. P. Winarski, M. Holt, J. Maser, C. Liu, R. Conley, S. Vogt, A. T. Macrander, G. B. Stephenson. "Focusing of hard x-rays to 16 nanometers with a multilayer Laue lens". Appl. Phys. Lett. 92, 221114-1-221114-3 (2008).

**10:05 AM Invited**

**In-Situ Characterization of Creep-Damage by X-Ray Microtomography**: Krzysztof Dzieciol[1]; Federico Sket[1]; Thomas Buslaps[2]; Marco di Michiel[2]; *Andras Borbely*[1]; Anke Pyzalla[3]; [1]Max-Planck Institut für Eisenforschung; [2]European Synchrotron Radiation Facility; [3]Helmholtz Zemtrum für Materialien und Energie

In-situ tomography based on its nondestructive nature has opened new possibilities for studying the kinetics of damage during high temperature creep. Application of accurate image processing algorithms to three-dimensional reconstructions series characterizing consecutive stages of damage offers an excellent opportunity to characterize the deterministic nature of the process. In contrast to metallographic techniques, where damage evolution results from statistical considerations by averaging over cavities of different sizes, in situ tomography allows studying the more appropriate evolution of single cavities, which is in accordance with the theoretically predicted volume dependence of cavites growth-rate. The new approach delivered for the first time accurate experimental data that can be compared with theoretical predictions. Experimental results for copper confirm the theoretically predicted proportionality between cavity growth-rate and cavity volume as well as between growth-rate and strain-rate. The real growth-rates, however, are larger by a factor of about 10 compared to theoretical predictions.

**10:25 AM Break**

**10:35 AM**

**In Situ X-Ray Synchrotron Observations of Steel Phase Transformation under Non-Equilibrium Conditions**: *Wanchuck Woo*[1]; Eliot Specht[2]; Zhili Feng[2]; Wei Zhang[2]; Xunli Wang[2]; [1]Korea Atomic Research Institute; [2]Oak Ridge National Laboratory

Non-equilibrium solid-state phase transformation in cold-rolled carbon steels was investigated using an X-ray synchrotron beam line of the Advanced Photon Source. A programmable electric resistance heating device was used to heat the steel specimens up to 1300 ºC (fully austenitized) under vacuum environment. Different heating rates (1, 3, 10, 30 ºC/s) were used to study the non-equilibrium phase transformation kinetics. A fast CCD detector with time resolution of 1 second was used to collect the diffraction patterns from the surface of the specimen during the experiment. The phase transformation starting and finishing temperatures ($T_{A1}$, $T_{A3}$) and the relative volume fractions of austenite and ferrite phases were obtained from the diffraction peak analysis. The experiments revealed unexpected transformation behavior including reduced $T_{A1}$ temperature and increased volume fraction of the austenite phase at increased heating rate. The experimental details and the observed phase transformation behavior will be presented and discussed.

**10:50 AM**

**Characterization of Hydride Phase Stability in Zirconium Alloys as a Function of Yield Stress with Synchrotron X-Ray Diffraction**: *Eric Tulk*[1]; Matthew Kerr[2]; Mark Daymond[1]; [1]Queen's University; [2]US Nuclear Regulatory Commission

Zirconium alloys find application world wide as a structural material in nuclear reactors. The mechanical properties of zirconium components degrade in-service as a result of hydride embrittlement. While some aspects of the mechanics of embrittlement are well characterized, there is significant disagreement in the literature regarding hydride phase stability during thermo-mechanical processing. It has been suggested that zirconium yield stress is the dominate factor associated with hydride phase stability, but no comprehensive database was available to test this hypothesis (previous studies examined only a narrow range of alloy compositions and processing conditions). This work uses synchrotron x-ray diffraction to characterize the hydride phase formed in zirconium alloys under a wide range of zirconium alloy compositions and processing conditions. The surprising result is that zirconium yield stress is only one parameter effecting hydride phase stability, a result that may explain the wide variation reported for hydride phase stability in the literature.

**11:05 AM**

**Growth of Anti-Phase Domains in Ternary Feco Alloys under Various Annealing Treatments and Cooling Processes**: *Ralph Gilles*[1]; Michael Hofmann[1]; Yan Gao[2]; Frank Johnson[2]; Debashis Mukherji[3]; Christoph Hugenschmidt[1]; Philip Pikart[1]; [1]TU Muenchen; [2]GE Global Research; [3]TU Braunschweig

Recent years have seen a reawakened interest in Fe-Co alloys due to the increased demands of modern electrical power generation and distribution equipment. For industrial application the challenge involves increasing the tensile strength and ductility of FeCo alloys. The effects of alloying FeCo with Pt, Pd, Mn, Ir, and Re have been investigated as part of this work by neutron diffraction. In the composition range of about 30%-70% Co, FeCo alloys undergo a continuous order-disorder phase transformation in the temperature range of 615±30°C [1]. Whereas the Tc temperature is independent of the thermal history, the microstructure of ordered domains is sensitively dependent on thermal history and on the kinetics of the order/disorder transition. As a complementary method positron measurements have been performed to describe the nucleation of domains with the correlation to the defect density. Reference:[1] R. Gilles et al., Metall. Mat. Trans. A, 2009, in print.

**11:20 AM**

**Effects of Shot Peening Aluminum Alloy A356.2 Cast Plate with Steel and Glass Shot on the through-Thickness Residual Stresses**: A. Ritter[1]; B. Kuhr[1]; C. Hubbard[2]; T. R. Watkins[2]; *Carl Boehlert*[1]; X. Niu[3]; [1]Michigan State University; [2]Oak Ridge National Laboratory; [3]Magna Cosma International

Shot peening forms a compressive residual stress on the surface which can favorably increase fatigue life. The goal of this study was to gain an understanding of the residual stresses formed after shot peening a cast automotive aluminum alloy A356.2. The residual stresses were measured using X-ray diffraction for near surface and neutron diffraction for through thickness residual stresses. Using the Neutron Residual Stresses Mapping Facility in the High Flux Isotope Reactor at Oak Ridge National Laboratory the residual stresses were measured in the longitudinal, transverse and normal direction as a function of depth from the surface. The d-spacings and strain free d-spacing, obtained by careful measurement of a stress free coupon, were used to calculate the residual strains and derive the residual stresses using Young's modulus and Poisson's ratio. The results indicated that the greatest compressive residual stress existed within 20 µm from the surface and the compressive residual

**11:35 AM**

**Residual Stress Analysis of Resistance Spot Welding, FE Modeling and Neutron Diffraction Measurement**: *Liang Wang*[1]; Sergio Felicelli[2]; Camden Hubbard[3]; Douglas Bammann[1]; [1]Center For Advanced Vehicular Systems, Mississippi State University; [2]Mechanical Engineering Department, Mississippi State University; [3]Oak Ridge National Laboratory

A finite element model was developed based on the welding software SYSWELD in order to calculate and analyze the residual stress distribution in the resistance spot welding process. The model takes into account the electrical-thermal, thermal-metallurgical, and thermo-metallurgical-mechanical aspects of the process. The model was employed to calculate the temperature history, nugget size growth, and the residual stress field during different stages of resistance spot welding. The effects of welding parameters such as current, pressure, and weld cycles on residual stress distribution were investigated. The predicted nugget shape and size agree well with experimental data. To validate the predicted stresses, neutron diffraction residual stress mapping was used to characterize the through-thickness stresses and surface stresses in a spot welded steel joint. The origin of the residual stress distribution is discussed based on the thermal histories of the samples, and the calculated stresses are compared with measurements obtained by neutron diffraction mapping.

**11:50 AM**

**Deformation Behavior of Nanocrystalline Co Measured by High-Energy X-Ray Diffraction**: *Ryan Ott*[1]; Morris Wang[2]; Matthew Besser[1]; Jon Almer[3]; Matthew Kramer[1]; [1]Ames Laboratory (USDOE); [2]Lawrence Livermore National Laboratory; [3]Argonne National Laboratory

Unlike most nanocrystalline (nc) metals, nc-Co exhibits large tensile plasticity prior to failure. Previous molecular dynamics simulations have shown that plastic deformation in nc-Co is characterized by an hcp-fcc phase transformation while bulk mechanical tests have suggested that mechanical twinning is responsible for the enhanced ductility. We have examined the deformation behavior of nc-Co subject to cycled loading at different strain rates using in situ X-ray diffraction. From these experiments we do not see any evidence of a strain-induced phase transformation, but instead we find distinct changes in the crystal orientations during plastic flow. The relationship between the texture changes and mechanical twinning and slip are discussed.

## Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Electromigration

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* Kwang-Lung Lin, National Cheng Kung University; Sung Kang, IBM; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Iver Anderson, Iowa State University; Fu Guo, Beijing University of Technology; Thomas Bieler, Michigan State University; Andre Lee, Michigan State University; Rajen Sidhu, Intel Corporation

Monday AM Room: 204
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Sung Kang, IBM Corp.; K. N. Subramanian, Michigan State University

**8:30 AM Introductory Comments by K. L. Lin**

**8:35 AM Invited**

**Thermomigration and Creep in Pb-Free Flip Chip Solder Joints**: *King-Ning Tu*[1]; [1]University of California, Los Angeles

Due to joule heating powered by AC, thermomigration was observed in Pb-free flip chip solder joints. Motion of indented markers indicated that Sn had moved from the cold end to the hot end, and hillocks of Sn were found at the hot end. Hillock formation means that creep has occurred. We have used irreversible processes to analyze the interaction between thermal force and chemical force. To do so, we have included temperature as a variable in the chemical force. Comparing to the interaction between electromigration and creep at constant temperature, we have not found a critical length in thermomigration. Both experimental and theoretical analyses will be presented.

**9:00 AM**

**Effect of Initial Microstructure on Electromigration Behavior of Eutectic Sn-Pb Solder Joints**: *Andre Lee*[1]; Yi-Chih Lee[1]; K.N. Subramanian[1]; [1]Michigan State University

Atom/ion migration from current stressing a multi-phase alloy should depend on the size, shape, and distribution of phases present since they will affect the number and orientation of phase boundaries encountered by migrating species. In order to gain a better understanding of the role of microstructure on material movement resulting from current stressing, electromigration studies were carried out on eutectic Sn-Pb solder joints subjected to various isothermal aging treatments. These studies characterized the development of surface topography and evolution of microstructural features resulting from electromigration. Results of this study indicate that electromigration in two-phase alloys will be significantly influenced by the initial microstructural features.

**9:15 AM**

**Effects of Reinforcements Addition on Microstructural Evolution in Eutectic SnBi Solder Joints under Current Stressing**: *Ruihong Zhang*[1]; Mengting Han[1]; Fu Guo[1]; Guangchen Xu[1]; [1]Beijing University of Technology

Composite approaches have been efficient in enhancing the reliability of lead-free solder joints in many aspects. However, the effects of reinforcement particles on solder joints under current stressing have been paid little attention. In this study, eutectic SnBi solder was used as the matrix soldering material to examine the effects of reinforcements addition on the microstructural evolution in solder joints under current stressing. Such electromigration features were compared with non-composite SnBi solder joints from our earlier studies. Polyhedral oligomeric silsesquioxanes (POSS ®), Cu, and Ni particles with different volume fractions were chosen as reinforcements for the eutectic SnBi solder. The electromigration behavior of the composite solder joints were investigated under the current density of $1.0\times10^4 A/cm^2$ at 50°C and 80°C. Our

first-stage study reveals that the POSS particles can slow down the separation of Bi- and Sn-rich phases and retard the solder depletion at the cathode of joints.

**9:30 AM**

**Electromigration Study of Flip Chip Packages under Extra-High Current Density Tests with Temperature Control**: *Jia-Hong Ke*[1]; Yu-Wei Lin[1]; C. R. Kao[1]; [1]Department of Materials Science and Engineering, National Taiwan University

Electromigration phenomenon in flip chip solder joints under high temperature conditions has been widely studied. However, electromigration tests were seldom carried out under proper temperature control. In this study, a cooling system with a PID controller was applied in order to maintain the chip temperature at 60 degree C, and different extra-high current densities ($5.5\times10^4$ A/cm$^2$, $5.0\times10^4$ A/cm$^2$, and $4.5\times10^4$ A/cm$^2$) were used. The objective of this study is to investigate the effects of controlled temperature and extra-high current density on flip chip solder joints. The results showed that low controlled temperature could retard void formation in solder joints even under extra-high current density tests. Instead, substantial IMC dissolution at the cathode and IMC accumulation at the anode was observed.

**9:45 AM**

**Interaction between Electromigration and Diffusionally Accommodated Interfacial Sliding at Hetero-Interfaces**: *Praveen Kumar*[1]; Indranath Dutta[1]; [1]WSU

Thin film structures in microelectronic devices can deform via unusual, scale-sensitive phenomena due to thermo-mechanical loads sustained during service. In particular, large shear stresses may develop at interfaces during thermo-mechanical excursions, allowing the interface to slide via diffusionally accommodated processes (i.e., creep). Such interfacial creep can be further augmented by superimposed electric currents (via electromigration) as the thin film features shrink to the nanometer regime in modern microelectronic devices, potentially causing severe reliability problems. In this paper, we present experimental evidence of the interaction between electromigration and interfacial sliding, based on a model Pb-Si system. It was noted that sliding rates were enhanced at interfaces where the shear stress was in the same direction as electron flow, and less at interfaces where stress and electron flow opposed each other. The kinetics of these interactions and their importance in microelectronics are assessed using a new constitutive law for electromigration-assisted interfacial sliding.

**10:00 AM Break**

**10:15 AM**

**Microstructures and Crystal Orientation of ß-Sn for Sn-Ag and Sn-Cu Solder Joints under Electromigration**: *Sun-Kyoung Seo*[1]; Sung K. Kang[2]; Moon Gi Cho[1]; Hyuck Mo Lee[1]; [1]KAIST; [2]IBM T.J. Watson Research Center

Recently, it is known that the anisotropic properties of the ß-Sn phase in Pb-free solders strongly affect the reliability of solder joints. Especially, under electrical current, the ß-Sn grain orientation could be an important factor to determine the lifetime of solder joints because the diffusion rates of Cu, Ni and Ag atoms in ß-Sn are very different depending on Sn grain directions. It is also found that the Sn grain orientation is strongly influenced by solder alloys, Sn-Ag vs. Sn-Cu, as well as by the substrate pad metallurgy, such as Cu/OSP vs. Ni(P)/Au. In this study, the changes in the microstructures and the ß-Sn crystal orientation to occur during electromigration are investigated with Pb-free solder joints. The electromigration test is conducted at 150°C under a constant current density, with two solder joints of Ni(P)/Sn-Cu/Cu and Ni(P)/Sn-Ag/Cu. The electromigration lifetime is compared for the two joint systems.

**10:30 AM**

**Direct Measurement of Back Stress in Tin Strips under Electromigration by Synchrotron Radiation X-Ray**: *Yang Yi Lin*[1]; Albert T. Wu[1]; [1]National Central University

This paper presents in situ measurements of stress evolution in tin strips under DC electromigration using synchrotron radiation X-ray. The back stress gradients at various testing conditions were directly detected by the shift of diffraction peaks. The kinetic parameters were deduced. Samples were also subjected to alternating polarity of current. The effect of Joule heating was observed and the increase in temperature was determined. The protective oxide layer on the surfaces is considered to influence critically the kinetics of stress evolution.

**10:45 AM**

**Discussion on the Mechanism of Electromigration from the Perspective of Electromagnetism**: *Peng Zhou*[1]; William Johnson[2]; [1]UC Irvine; [2]MSE, University of Virginia

From the perspective of electromagnetism, conservation of both momentum and energy in the electro-magnetic fields can show that the effective charge number has a linear dependence on the current density. It is suggested that at low temperatures, the momentum of the electro-magnetic fields, instead of the momentum of the drifting electrons, is transferred to the crystal defects to give rise to the polarity effects; while at high temperatures the momentum of the electro-magnetic fields is progressively transferred to the vibrating atoms and causes the “stressing effect” of the lattice, which can be the origin of the decrease of activation energy for diffusion at high temperatures. A tentative failure analysis will give an equation similar to the Black’s equation with an extra $J^2$-dependent exponential term totake into account the ``stressing effect'' at high temperatures.

**11:00 AM**

**Critical Conditions of Electromigration-Induced Cu Dissolution in Pb-Free Solder Joints**: *Jung Kyu Han*[1]; King-Ning Tu[1]; [1]UCLA

One of the most serious reliability problems in solder joints technology still remains on current crowding region. In order to reduce current crowding effect, thick Cu under-bump-metallization (UBM) or Cu column structure has been investigated. If the thickness of Cu is not thick enough, it could not effectively eliminate current crowding. If Cu is too thick, however, it may cause the increase of thermal-mechanical stress. Therefore, kinetics under electromigration to study how fast Cu dissolves into the solder is of interest. The thickness of intermetallic compounds (IMC) shrinks first and reaches dynamic equilibrium. Once it reaches the dynamic equilibrium, Cu starts to dissolve into solder very fast. Experiment of several different conditions was carried out and the kinetic model is proposed to predict Cu dissolution rate at given temperature and current density. This study also shows critical conditions to stimulate fast Cu dissolution.

**11:15 AM**

**TEM Characterization of the Porous Structure Induced by High Current Density in the Flip-Chip Solder Joint**: *Ming-Yen Tsai*[1]; Yen-Liang Lin[1]; C. Robert Kao[1]; [1]National Taiwan University

Owing to the combined effects of current crowding and local Joule heating during electromigration, asymmetrical consumption of the Ni(V) layer in UBM was initiated near the edge of passivation where the electrons entered into the solder joint. Afterward this consumed Ni(V) layer transformed to a porous structure. This porous structure was non-conductive and eventually propagated all over the Ni(V) layer to make the joint fail. In this study TEM technique was used to analysis the porous structure in detail. Many voids were observed near the interface of Ni(V)/(Cu,Ni)6Sn5 in the porous structure. EDX analysis on TEM showed that almost no Ni signal was detected in the Ni(V) layer except for the interface of Ni(V)/Al. The result of SADP verification of the porous structure showed that Cu6Sn5 and V2Sn3 fine grains distributed in the amorphous Ni(V) matrix. A very thin layer of Ni3Sn4 fine grains formed at the interface of Ni(V)/Al.

**11:30 AM**

**The Enhanced Growth of Sn Whisker on High Melting Temperature Sn-Pb Solder Joint by Current Stressing**: *Ying-Ta Chiu*[1]; Kwang-Lung Lin[1]; Yi-Shao Lai[2]; [1]Department of Materials Science and Engineering, NCKU; [2]Central Labs, Advanced Semiconductor Engineering, Inc., Kaohsiung

In this study, the Sn whisker formation behavior of 95Pb5Sn/63Sn37Pb composite solder joint was investigated. The test samples were stressed with a current density of 5×10^3 A/cm^2 at 150°C for 100, 200 and 300 hours and then were kept under 50% RH at 24°C for 38 days. The results reveal that no Sn whiskers were found on the polished surface of 95Pb5Sn/63Sn37Pb composite solder joint after current stressing. However, Sn whisker formed on the surface of the high-Pb matrix after treatment at 24°C with 50% RH for 38 days. The interesting phenomenon is that the length of Sn whisker increases as the current stressing time increases. It is believed that the Sn whisker formation on the surface of high-Pb matrix is resulted from the residual compressive stress relieced after annealing for 38 days. The mechanism of the growth of Sn whisker was discussed in this present work.

**11:45 AM**
**Study of Joule Heating Effects in Lead-Free Solder Joints under Various Current Densities Using Infrared Thermography**: *Guangchen Xu*[1]; Fu Guo[1]; Andre Lee[2]; K.N. Subramanian[2]; Neil Wright[2]; [1]Beijing University of Technology; [2]Michigan State University

Temperature distribution in the eutectic Sn-Bi solder joints that does not exhibit thin-thick divergence associated with current crowding issues was investigated using the non-contact infrared thermography technique. These studies evaluated the role of imposed current density and the associated increases in temperature at different regions of solder joint. Findings of this study indicate that the existing temperature differences between center and end region of these solder joints are based on thermal conductivity issues, in spite no having current crowding in the joint geometry employed. These differences arise based on the thermal conduction issues rather than current crowding issues. Implications of these findings on thermal migration, which is believed to be of importance in inter-connects will be discussed.

## Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials IX: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee
*Program Organizers:* Chih-ming Chen, National Chung Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Kejun Zeng, Texas Instruments; Yee-wen Yen, National Taiwan University of Science and Technology; Wojciech Gierlotka, AGH University of Science and Technology; Chao-hong Wang, National Chung Cheng University

Monday AM Room: 203
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Chih-ming Chen, National Chung Hsing University; Yee-wen Yen, National Taiwan University of Science and Technology

**8:30 AM Invited**
**Evaluation of Current Mode and Additives in Copper Via Filling**: Jin-Yong Sim[1]; In-Kyu Lee[2]; *Jae-Ho Lee*[1]; [1]Hongik University; [2]Korea Aerospace University

Copper via filling by electroplating method became a common technology for interlayer metallization in 3D SiP and then it has been extensively studied. The void free copper via can be obtained with varying the additives of the electrolytes and, the current density and mode. The effects of current mode and additives on the copper via filling were investigated. The acid copper electrolytes containing additives such as PEG, SPS, JGB and PEI were examined to fill 5~20μm via hole without void. The different sized vias were successfully filled with copper varying current density and mode after fixing additive conditions. The effect of pulse duty cycle on via filling was also investigated.

**8:55 AM**
**Carbon Nano Tube and Nickel Alloys Composite Electroplating**: Ho-Kyung Um[1]; Heung-Yeol Lee[2]; Tai-Hong Yim[2]; *Jae-Ho Lee*[1]; [1]Hong Ik University; [2]Korea Institute of Industrial Technology

Nickel alloys have been extensively used in packaging and electronic industries. Ni-Fe alloy, was used as shadow mask for its low coefficient of thermal expansion. Even Ni-Fe alloy has high yield stress and hardness, its values are reduced after heat treatment. CNT is one of the eminent nanomaterial and has many advantages in electrical conductivity, yield stress, thermal stability and etc. If CNT is coexisted in Ni-Fe matrix, the disadvantages of Ni-Fe alloy can be reduced. CNT were agglomerated and tangled each other and then their surface needs to be modified to be suspended in the electrolytes. CNT was grinded and pulverized in the ball mill and followed by acid digestion. Surface modified CNT was suspended in the Ni-Fe electrolytes and then CNT and Ni-Fe alloy was codeposited. The optimum condition for the CNT suspension was investigated. The electrical and mechanical properties of composite coating were also investigated.

**9:15 AM**
**A Study on the Microstructure Evolution of Cu/Sn/Cu Bonding Stacks during Bonding and Their Mechanical Properties for the Applications of 3D Packaging**: *Byunghoon Lee*[1]; Sang-Su Ha[1]; Jeong-Won Yoon[1]; Hoo-Jeong Lee[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

For 3D packaging, in which multiple chips are stacked and bonded vertically for a higher level of integration, searching for bonding materials with high mechanical and electrical reliabilities is a key issue. In this study, we chose Sn as a bonding layer and examined the phase evolution process that occurred between Cu and Sn during the bonding process. We also carried out lap-shear tests on the samples bonded at different temperatures and different pressures to investigate how the microstructure changes (such as the formation of different intermetallic compounds) affect the mechanical properties of the bonded samples. Our analysis discloses that the molten Sn aggressively reacted with the Cu to form Cu6Sn5 and then Cu3Sn intermetallic compounds and that the formation of the IMC phases substantially enhances the strength of the bonds.

**9:35 AM**
**Effect of Wet Chemical Pretreatment Conditions on Cu-Cu Bonding Characteristics for 3-D IC Stacks**: *Jae-Won Kim*[1]; Eun-Jung Jang[1]; Myeong-Hyeok Jeong[1]; Seungmin Hyun[2]; Hak-Joo Lee[2]; Young-Bae Park[1]; [1]Andong National University; [2]Korea Institute of Machinery and Materials

Cu to Cu direct bonds for 3-D IC stacks have several advantages such as low electrical resistivity, high electrical reliability, and reduced interconnect signal delay. However, the process temperature is limited up to 400° to prevent CMOS devices from being thermally damaged. High temperature bonding is one of the key bottlenecks for 3D ICs applications due to a deadly impact on device reliability. In this work, the effect of wet chemical pretreatments on quantitative interfacial adhesion energy of Cu to Cu direct bond was systematically analyzed by using 4-point bending test. The interfacial adhesion energy was evaluated for various pretreatment times with HF and HCl pre-treatments. Surface and delaminated interface were characterized by the scanning electron microscope and X-ray photoelectron spectroscopy. The interfacial adhesion energy was systematically dependent on the wet pre-treatment time. Dependence of interfacial adhesion energy on the cleaning chemicals and times will be discussed in detail.

**9:55 AM Break**

**10:15 AM Invited**
**Tin Whisker Growth in Vacuum Thermal Cycling**: *Katsuaki Suganuma*[1]; Alongheng Baated[1]; Seong-Jun Kim[1]; Keun-Soo Kim[1]; Norio Nemoto[2]; Tsuyoshi Nakagawa[3]; [1]Osaka University; [2]JAXA; [3]Nippon Avionics Co., Ltd.

The aerospace electronics have specific requirements as compared with consumer electronics. Reliability is one of the most key facets that the mechanic designers must think of in the first place. Nevertheless, there have been the electronics failures of the satellites caused by Sn whiskers and Sn whisker failure is one of the most serious concerns for the space electronics. This work is the first report on the vacuum influence on Sn whiskers. In the space, surely Sn whiskers will grow under temperature cycling even in vacuum. 42 alloy lead-frames and ceramic chip capacitors with Sn plating were tested both in the vacuum and in air up to 2000 cycles. There are apparent differences in Sn whisker growth. The width of Sn whiskers is the most significant. Vacuum whiskers are thinner than air whiskers. The whisker growth mechanism in vacuum will be discussed.

**10:40 AM**
**Growth and Orientation of Tin Whiskers on an Electrodeposited Tin Thin Film under Three-Point Bending**: *Chih-ming Chen*[1]; Yu-jen Chen[1]; [1]National Chung Hsing University

Tin (Sn) whisker/hillock growth is a result of release of compressive stress in a Sn thin film. Filamentary Sn whiskers were formed on an electrodeposited Sn thin film aged at room temperature, while Sn hillocks were formed as the aging temperature was raised to 80 and 150°. A three-point bending test was performed on an electrodeposited Sn layer to investigate the Sn whisker growth under mechanically applied tensile stress. Sn whisker growth was mitigated on the Sn layer subjected to a tensile stress in bending. This mitigation growth suggests that part of the compressive stress in the Sn thin film was neutralized by the mechanically applied tensile stress. The growth orientation of the Sn whiskers formed on the high tensile stress region was random but directional on the low tensile stress region.

**11:00 AM**

**Synthesis of Nanostructured Carbon Materials Using Commercial Paper Phenolic Board**: *Yi-Wei Lin*[1]; Chih-ming Chen[1]; [1]National Chung Hsing University

Owing to possess the characteristics of low cost and easy fabrication, printed circuit boards (PCBs) have been widely manufactured and used in a variety of electronic products. But the lifetime of consumer electronic products is usually very short. So, they become out-of-date very quickly and then are abandoned. Unfortunately, this produces large amounts of waste PCBs which might have detrimental effects on the environment. In this poster, two potential methods are demonstrated to recycle PCBs and use them as the carbon source to synthesize highly valuable carbon nanomaterials.

## Processing Materials for Properties: Advanced Materials Processing

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division
*Program Organizers:* Brajendra Mishra, Colorado School of Mines; Akio Fuwa, Waseda University; Paritud Bhandhubanyong, National Metal and Materials Technology Center

Monday AM Room: 617
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Akio Fuwa, Waseda University; Tonya Wolfe, University of Alberta

**8:30 AM Keynote**

**Materials Science for the Next Generation**: *David Olson*[1]; Brajendra Mishra[1]; [1]Colorado School of Mines

Advanced materials, with their interfaces, steep gradients and very small dimensional microstructural features require more comprehensive analytical treatments. Future chemical, physical, and mechanical analyses and implementations need further refinement, such as incorporation of the non-linear behavior associated with interfaces and gradients. The use of electronic, magnetic, and elastic assessments to evaluate the material state will be an everyday practice, including the establishment of electronic metallography practices. The application of wave mechanics with wave-perturbing nondestructive tools and practices to assess more than one property measurement should enable a more comprehensive understanding of the material state.

**9:00 AM**

**Increase Production and Quality Improvement of Electrolytic Copper at Tank House in Naoshima Smelter and Refinery**: *Hideki Zen*[1]; Tatsuo Ishida[1]; Makoto Takagi[1]; [1]Mitsubishi Materials Corporation

Operation of copper tank house in Naoshima Smelter & Refinery started in 1969. Increase of electrolytic copper production had been mainly carried out in the past by installing more tank house cells and raising current density of commercial cells. However this time increase of production and improvement of quality was achieved without big amount of capital investment, as follows. 1)22 cells which were used for lead refinery were converted to starter sheet cells for copper refinery. 2)To improve verticalness of starter sheets, electrolysis time for starter sheets was changed from 24hours to 48 hours. 3)Starter sheet preparation machine was modified. 4)Flow amount of electrolyte was controlled at commercial cells. 5)Current efficiency of commercial cells was kept over 96.5%. As a result, production of electrolytic copper was increased by 750 T/M and tank house capacity reached to 19,500 T/M.

**9:20 AM**

**Damage of Surface by Impact of Nitrogen Jet under Pressure and Low Temperature**: *Hicham Laribou*[1]; Claude Fressengeas[1]; Denis Entemeyer[1]; Véronique Jeanclaude[1]; Abdel Tazibt[2]; [1]Laboratoire de Physique et Mécanique des Matériaux; [2]CRITT TJF&U

Processing surfaces with nitrogen jet at low temperature is an environmentally friendly and innovating technology. It has been introduced by NITROCISION® for industrial applications since 2003. The process consists in projecting onto the surface a nitrogen jet at 3000bars and -150°C. Several effects are combined. Reducing the temperature fragilizes the material, the thermal shock induces traction waves through the material, depressurization of the cryogenic fluid during vaporization induces a blasting effect promoting plastic deformation or lifting the material from the substrate, cavitation of gas inclusions in droplets induces high impact pressures.The process does not generate additional waste and it releases neutral gas into the atmosphere. It can be used in a range of industrial applications, including surface treatment, cutting, drilling, striping and cleaning. This work aims at understanding the mechanisms of surface removal as well as the modes of action of the jet on metallic materials.

**9:40 AM**

**Non-Linear Analytical Practices for Interfacial Phenomena and Nano-Size Microstructural Properties and Behavior**: *John Roubidoux*[1]; J.E. Jackson[2]; B. Mishra[1]; D.L Olson[1]; [1]Colorado School of Mines; [2]Generation 2 Materials Technology LLC

When the microstructural dimension approaches nano size and/or the interfacial boundary achieves space-charging behavior, the traditional linear driving force and mobility terms become insufficient to describe physical observations. This situation involves non-linear and higher order phenomena, which if properly analyzed can provide new insights and allow better predictions of small dimensional phenomena. Elastic and electronic wave measurements and practices can be used to assess thin films, nano-structure, surface layers, and other steep gradient potentials driven in materials. This paper presents the analytical basis for these nano microstructural property measurement correlations with elastic and electric wave property measurments. In addition, specific examples will be given which demonstrate the differences between linear and non-linear behavior.

**10:00 AM**

**Modernization Project of Onahama Smelter with New "O-SR Process"**: Osamu Iida[1]; *Teruyuki Matsutani*[1]; Kenji Kiyotani[2]; [1]Mitsubishi Materials Corporation; [2]Onahama Smelting and Refining Company

Onahama Smelter has been maintained its profitability with overcoming several high energy price period over 40 years by developing innovatory technology, while it employs conventional process, such as huge energy-consuming reverberatory furnaces (RFs) and conventional PS-converters. New development of combustible waste material treatment in RFs was commenced in 1990's, which realized drastic fuel saving and accompanying metal recovery. In December 2007, installation of new S-furnace of the Mitsubishi Process was completed. For copper concentrates smelting, S-furnace is solely used, which is directly laundered to two RFs to deliver melts continuouslly and automaticlly. RFs are dedicated to slag/matte separation and combustible waste material treatment. This new "O-SR Process" realized not only higher copper production and more flexibility for various kinds of raw materials, but also more combustible waste material treatment and less fuel consumption in RFs. This paper introduces good example of old-fashioned smelter revived and recent operating status.

**10:20 AM**

**Evaluation of Stress Corrosion Cracking Susceptibility of Drill Pipe Steels in CO2 Saturated Aqueous Solutions**: *Arshad Bajvani Gavanluei*[1]; Bhola Shaily[1]; B Mishra[1]; D. Olson[1]; [1]Colorado School of Mines

Constant extension rate test (CERT), or slow strain rate test (SSRT), has been conducted to investigate the stress corrosion cracking (SCC) susceptibility of high strength low alloy (HSLA) steels in CO2 containing aqueous environments. G-105 and S-135 grades of drill pipe steels have been used to study the effect of temperature and solution chemistry on the stress corrosion cracking behavior of these steels, using a high temperature and high pressure one liter autoclave. SCC susceptibility of HSLA steels has been evaluated by comparing the results of the experiments conducted in an inert environment (air) and various corrosive solutions at different temperatures from ambient temperature to 200°C, based on the NACE TM0198-2004 standard. Using EDS and XRD techniques, corrosion products have been identified. It has been shown that the SCC susceptibility increased with increasing temperature and maximum occurred at 160°C.

**10:40 AM Break**

**10:50 AM**

**Promotion of Recyclable Material Treatment at Mitsubishi Prpcess in Naoshima Smelter and Refinery**: Tetsuro Sakai[1]; Norio Usami[1]; *Masayuki Kawasaki*[1]; [1]Mitsubishi Materials Corporation

The recyclable materials including valuable metals such as copper and precious metals had been treated at Mitsubishi Continuous Copper Smelting

Furnace (Mitsubishi Process) in Naoshima Smelter and Refinery since the start of furnace operation. Early stage of recyclable material treatment was mainly focused on treating the copper bearing materials which did not contain inflammable materials due to a restriction on capacity of furnace waste heat boilers. As the environmental conservation awareness had been rapidly raised in recent years, establishing treatment systems for wide range of recyclable materials was demanded. To cope with the demand, Naoshima Smelter & Refinery started the operation of incinerating and melting plant of recycle waste in 2003. All of produced slag-metal generating from incinerating and melting plant of recycle waste is treated at Mitsubishi Process. Consequently new treatment system is established without secondary industrial waste by uniting two processes.

**11:10 AM**

**A Novel Process on Production of Thin Wall Austempered Ductile Iron Heat-Treated in the Mold**: *Ali-Reza Kaini-Rashid*[1]; Abolfazl Babakhani[1]; Mohammad Reza Ziaei[1]; [1]Ferdowsi University of Mashhad

In this study, the production of thin wall austempered ductile iron, heat treated in the mold and in the presence of Ni and Mo was investigated. The base iron was melted in an induction furnace and treated at 1450°C by plunging and sandwich methods. Ductile irons were cast in two molds: one was a strip metallic mold with the thickness of 3mm and another cylindrical mold with diameter of 2.5cm. After casting, the specimens were austempered at 350°C for 1hr. Then the microstructures of the specimens were examined by optical microscopy. Results indicated that the ausferrite microstructure can be achieved by the control of the cooling rate within temperature range between 280-400°C. Austempering time about 1hr resulted in no trace of martensite and the structure was only composed of bainitic-ferrite and retained austenite.

**11:30 AM**

**Remarkable Oxidation Resistance of Nanocrystalline Fe-Cr Alloys**: *Raman Singh*[1]; Prabhakar Singh[2]; [1]Monash University; [2]University of Connecticut

The paper will present a review of the fundamentals of likely oxidation mechanism of nanocrystalline metallic materials with examples, and validation of the hypothesis that it may be much easier to develop a protective film on nanocrystalline Fe-Cr alloys. A nanocrystalline Fe-10%Cr alloy has been found to possess in excess of an order of magnitude superior oxidation resiatance than its microcrystalline counterpart of same composition. Validation of the hypothesis may have highly attractive economic/industrial implications, for example for high temperature components in solid oxide fuel cells.

**11:50 AM**

**Discrete Element Simulation: An Efficient Tool for Optimizing Powder Processes from the Sintering Stage to the Final Properties**: *Christophe Martin*[1]; Xiaoxing Liu[1]; Jean-Jacques Kadjo[1]; Didier Bouvard[1]; [1]Grenoble-INP

The modeling of processes involving powder materials needs taking into account the particulate nature of the materials involved. The Discrete Element Method (DEM) is well suited for this task. It allows the macroscopic behavior of an assembly of particles to be calculated from the contact forces generated between each particle. Originally devised for geomaterials, we show that DEM simulations have great potential for powder processes in materials science. Using the example of a thin partially sintered ceramic electrode, we first model the process stages from powder packing to sintering to generate a realistic microstructure. This numerical microstructure is then used to compute macroscopic characteristics such as electrochemical or mechanical properties. In particular, the elastic and fracture properties of the electrodes are calculated as a function of pertinent microstructural properties. The DEM simulations are finally used to propose novel microstructures to optimize the electrochemical performance of an electrode for electrochemical applications.

**12:10 PM**

**Cold Gas Dynamic Spraying of Titanium: A Reliable and Environmentally Friendly Coating Deposition Process**: *Wilson Wong*[1]; Stephen Yue[1]; Eric Irissou[2]; Jean-Gabriel Legoux[2]; [1]McGill University; [2]National Research Council Canada

Cold gas dynamic spray is a solid state high kinetic energy coating and free-form technique. Micrometres-scaled metallic powders are projected onto substrates at supersonic velocities to form a coating. This technique has initiated major interest in the aerospace industry due to its potential to manufacture aerospace engine components with minimal material waste, estimated with a capability to reduce waste of up to 90% compared to conventional fabrication methods. On account of the severe requirements in producing these components, cold sprayed coatings must prove themselves reliable to earn recognition in the industry. Thus, in this study, mechanical properties such as the bond strength of cold sprayed titanium coatings were evaluated. Various particle impact velocities were achieved by altering process conditions (temperature and pressure). Substrates of different surface roughness and hardness were investigated (aluminum alloy, pure titanium, and steel). Additionally, the coating properties were studied via scanning electron microscopy and microhardness testing.

## Refractory Metals 2010: Processing and Properties I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Refractory Metals Committee
*Program Organizers:* Brian Cockeram, Bechtel-Bettis; Gary Rozak, H.C. Stark

Monday AM Room: 2A
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Brian Cockeram, Bechtel Marine Propulsion Corporation; Gary Rozak, H. C. Starck, Inc.

**8:30 AM**

**Surface Processing of an Iridium Alloy**: *Evan Ohriner*[1]; George Ulrich[1]; Roger Miller[1]; Wei Zhang[1]; [1]Oak Ridge National Laboratory

The effects of surface processing on the microstructure and properties of DOP-26 iridium alloy (Ir-0.3% W-0.006% Th–0.005% Al) are investigated. The surface treatments include pulse laser heating, grit-blasting with tungsten carbide media, and mechanical surface deformation by manual scribing. The effects of processing parameters on surface morphology, grain structure, and optical emissivity are evaluated. The results are compared to those from numerical modeling of surface processing and material grain growth behavior. Recrystallization and grain growth is evaluated for both as-treated surfaces and following post-treatment annealing as a method to estimate plastic strains associated with surface processing. Laser surface processing results are compared to those for mechanical surface processing.This research was sponsored by the Office of Radioisotope Power Systems, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

**8:55 AM**

**Effect of Tantalum on the Tensile Impact Ductility and Fracture Behavior of Iridium**: *E. P. George*[1]; C. Carmichael[1]; A. Gali[1]; E. Ohriner[1]; [1]Oak Ridge National Laboratory

Iridium has a high melting point (2450C) and excellent corrosion resistance making it ideal for certain refractory applications. Trace elements can have beneficial (e.g., Th, Ce and W) and deleterious (e.g., Si) effects on the mechanical properties of iridium. Here, we investigate the effects of Ta (0.1 to 0.5 wt.%) on an iridium alloy referred to as DOP-26 in the literature (Ir-0.3W-0.006Th-0.005Al). Tensile impact tests were performed using a gas-powered gun at strain rates of ~1000/second and temperatures of ~1000C. Tantalum additions as low as 0.1 wt.% were found to severely embrittle the DOP-26 iridium alloy, with tensile ductility decreasing to ~5%, which is a factor of 3~5 lower than that of the Ta-free alloy. Possible mechanisms for the embrittlement, including grain growth, grain boundary segregation, and formation of low-melting eutectics, are investigated and discussed.Research sponsored by the Office of Radioisotope Power Systems, U.S. Department of Energy.

**9:20 AM**

**Effects of Thermo-Mechanical Processing on Texture and Microstructure of Pure Molybdenum Plates for Optimum Sputtering Performance**: *Gary Rozak*[1]; Peter Jepson[1]; [1]HC Starck Inc

Crystallographic texture and uniform microstructure are important characteristics for sputtering targets in order to produce a uniform thin film. Texture and microstructure develop in Molybdenum plates as a result of the processing methods. Powder metallurgy and arc cast molybdenum samples were produced via various thermo-mechanical processing (TMP) techniques, e.g., hot rolling, extruding or forging. The subsequent texture was evaluated with electron back scattering diffraction (EBSD) analysis and the microstructure was recorded with standard optical techniques. This presentation will report the

effects of TMP conditions on the subsequent microstructure and crystallographic texture.

**9:45 AM**

**The Role of Stress State and Wrought Processing on the Fracture Toughness and Toughening Mechanisms of Wrought Unalloyed Molybdenum, TZM Molybdenum, ODS Molybdenum, and Molybdenum Alloys**: *Brian Cockeram*[1]; [1]Bechtel-Bettis

The ductile laminate toughening mechanism observed for wrought molybdenum results from a lower toughness in the short-transverse orientation that leads to splitting of the microstructure along grain boundaries in the regions of stress concentration. In this work, commercially available molybdenum, molybdenum alloys, and ODS molybdenum are rolled to thinner sheet and then subject to tensile and fracture toughness testing and examination of the microstructure and toughening mechanism. The finer grain size, finer precipitate size, and state of plane stress achieved for the thinner sheet specimens appears to enhance the ductile laminate toughening observed for wrought molybdenum alloys to improve the fracture toughness. The detrimental effect of crack initiation from brittle carbides, oxides, and second phases is diminished under a state of plane stress to result in higher fracture toughness and lower DBTT values. The impact of stress-state and grain size on the toughening mechanism and resulting properties is discussed.

**10:10 AM Break**

**10:25 AM**

**Plastic Strain Concentration at Grain Boundaries in a 50Mo-50Re Alloy**: *Tongguang Zhai*[1]; Jianhui Xu[2]; [1]University of Kentucky; [2]Smith International

A 50Mo-50Re alloy was stretched at room temperature in air at a range of strain rates between 10-6s-1 and 1s-1. It was found that the alloy exhibited brittle fracture at slow strain rates while it became ductile at a fast strain rate. Cracks were predominently initiated from grain boundaries and triple junctions. Peaks of misorientation gradient were observed close to grain boundaries and triple junctions in the deformed alloy with EBSD. Misorientation maps were then calculated from the measured orientation maps using EBSD, indicating that indeed misorientation gradient peaked around grain boundaries and triple junctions in the deformed alloy. This was consistent with the TEM observation of bend contours around grain boundaries in the alloy. The results from the EBSD experiments demonstrated that there was stress concentration around grain boundaries and triple junctions during plastic deformation, thereby leading to crack initiation at grain boundaries or triple junctions in this alloy.

**10:50 AM**

**Processing and Properties of Tungsten-25%Rhenium with and without Hafnium Carbide**: *Todd Leonhardt*[1]; James Ciulik[2]; [1]Rhenium Alloys, Inc.; [2]The University of Texas at Austin

Historically, tungsten-25% rhenium alloy has been produced into wire for the thermocouple market, but recent demands for high temperature structural components and high-temperature friction stir welding tooling has forced the development of novel processing techniques for tungsten-rhenium and tungsten-rhenium with hafnium carbide. With a melting point of 3050°C, a recrystallization temperature near 1900°C, and a ductile-to-brittle transitional temperature (DBTT) of -50°C, tungsten-25% rhenium is a high-temperature alloy with high strength, high toughness, and low erosion properties needed for friction stir welding tooling. The addition of hafnium carbide substantially improves the strength of this alloy at elevated temperatures, although it makes deformation processing of it into useful shapes more challenging. The processing and microstructures of tungsten-25% rhenium and tungsten-25% rhenium with hafnium carbide will be discussed, as well as the effects of different processing conditions on the mechanical properties of the alloys at ambient temperature, 1371°C, and 1926°C.

**11:15 AM**

**Fracture Behavior of Polycrystalline Tungsten**: *Bernd Gludovatz*[1]; Stefan Wurster[1]; Andreas Hoffmann[2]; Reinhard Pippan[1]; [1]Erich Schmid Institute of Materials Science / Austrian Academy of Sciences; [2]Plansee Metall GmbH

Tungsten and tungsten alloys show the typical change in fracture behavior from brittle at low temperatures to ductile at high temperatures, however the parameters influencing the brittle fracture behavior are not well understood. Using techniques like electron backscatter diffraction (EBSD) and auger electron spectroscopy (AES) parameters like grain size, texture and grain boundary segregation were investigated. EBSD was used to record the crack propagation path, which shows transgranular and intergranular fracture. Depending on temperature and alloying content, different sizes of plastically deformed areas were observed along the crack path. The crack propagation was additionally recorded using the potential method which showed an R-curve behavior for these materials. To improve the understanding of the influence of impurities on the cleavage resistance of grain boundaries different microstructured materials were investigated by AES. The effect of the different microstructural features on the fracture behavior and the ductile to brittle transition will be discussed.

**11:40 AM**

**Study the Activated Sintering of Tungsten as a Function of Heating Mode**: *Avijit Mondal*[1]; Kranti V. Reddy[1]; Anish Upadhyaya[1]; Dinesh Agrawal[1]; [1]Indian Institute of Technology, Kanpur

Tungsten is an excellent material for many industrial applications due to its attractive properties. However, its potential of being an excellent high temperature structural material hinders because of poor sinterability. Usually the consolidation of a conventional microcrystalline W powder is difficult and requires very high temperature in electrical resistance sintering under hydrogen atmosphere. Activated sintering of tungsten metals through addition of selected transition metals such as Ni, Fe, Co, Cu has been critically discussed in this paper. The general observations and theoretical treatments have led to the identification of beneficial activators by studying the binary phase diagrams of the metal and the activator element. Microwave processing has been emerging as an innovative sintering method for consolidation of particulate metals. The 2nd part of this study describes recent research findings wherein W, W-1Ni, W-1Fe, W-1Co and W-1Cu have been successfully consolidated using microwave heating which resulted in further acceleration in densification.

## Solar Cell Silicon: Production and Recyling: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee, TMS: Energy Conversion and Storage Committee

*Program Organizers:* Anne Kvithyld, SINTEF; Gregory Hildeman, Solar Power Industries

Monday AM Room: 2B
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Anne Kvithyld, SINTEF

**8:30 AM**

**Electrochemical Production of Affordable Solar Grade Silicon**: *Antony Cox*[1]; Derek Fray[1]; [1]University of Cambridge

Photovoltaic technology has the potential to offer unlimited pollution-free renewable energy. A major barrier in the commercialisation and acceptance of this technology is the high cost of silicon. There are alternatives to silicon but they are less efficient and degrade rapidly. Current methods to extract silicon also produce substantial amounts of CO2, operate at very high temperatures and involve several stages with the use of expensive and highly toxic chemicals. An alternative electrochemical method is discussed which involves electro-deoxidizing inexpensive silica feedstock in a molten salt electrolyte to produce 99.90wt% silicon which is then electrorefined in the same electrolyte to remove trace impurities such as C, Fe, P, B, Ti, Ni, Mn and remaining O to deposit silicon of 99.9999wt% purity. The process consumes less energy than current methods and with the use of inert anodes the only bi-product is oxygen.

**8:55 AM**

**Electrorefining of Metallurgical Grade Silicon in Molten Salts**: *Geir Martin Haarberg*[1]; Shuihua Tang[1]; Karen Osen[2]; Henrik Gudbrandsen[2]; Sverre Rolseth[2]; Ole Edvard Kongstein[2]; Shulan Wang[3]; [1]Norwegian University of Science and Technology; [2]SINTEF; [3]Northeastern University

Electrochemical studies of the behaviour of silicon were carried out in molten salt electrolytes. Electrorefining of metallurgical grade silicon was done in molten chloride and fluoride electrolytes at temperatures from 700 – 850°C. Electrochemical studies such as voltammetry showed that silicon can be anodically dissolved and cathodically deposited from molten salts. Silicon deposits of good quality was obtained by electrorefining in molten KF-

LiF-$K_2SiF_6$ at 760°C. High current densities of ~0.5 A/cm$^2$ could be used, but inclusions of fluorides represent a challenge in this electrolyte.The use of a liquid alloy anode of silicon and copper was found to be beneficial in molten $CaCl_2$ (80 mol%) - with NaCl, CaO, and dissolved Si. Promising electrorefining results for some impurity elements such as aluminium and titanium were obtained in recent experiments. However, the contents of phosphorus and boron were still too high for solar cell applications.

**9:20 AM**
**Preparation of High Purity Silicon by Electrolysis-Vacuum Distillation**: *Jidong Li*[1]; Mingjie Zhang[2]; Yiyong Wang[1]; [1]School of Materials Science and Engineering; University of Science and Technology Liaoning; [2]School of Metallurgy, Northeastern University

In this paper, Mg-Zn-Si alloys were prepared by electrolysis in electromagnetic stirring using high purity silicon dioxide as raw material and high purity silicon obtained was separated from Mg-Zn-Si alloys by vacuum distillation. The conditions such as current density, electrolysis time and electromagnetic stirring effecting on back electromotive force, silicon content and current efficiency were well investigated. Finally, at 1000°,magnetic field intensity of 28mT, the silicon content of 35.7%(mass fraction) in Mg-Zn-Si alloy can be obtained by electrolyzing for 4h in the current density of 0.56A.cm-2. Then, at 1050°,the high purity silicon of 99.98% was obtained by vacuum distillation for 3h.

**9:45 AM Break**

**10:20 AM**
**Hierarchy of Slurry Recycling Options**: *Walter Radeker*[1]; [1]CRS Reprocessing Services LLC

When considering the wire slicing process for cutting PV silicon wafers, recycling slurry is an economic requirement. There are, however, meaningful variations in recycling choices. By exploring these choices, we can help companies make the right choices for them. In general the options are no recycling of the slurry components, use of self-managed on-site systems, or using an outside vendor to manage the reprocessing system, either on-site or off-site. Within this range of offerings, there is a hierarchy designed to satisfy desires that drives how firms think when considering how best to recycle. This hierarchy includes satisfying the desire for risk management, cost improvement, stable process control, and direct process management. Customer data and industry experience tells us that these are all are important drivers that can be arranged in a way to determine the best recycling method for a customer.

**10:45 AM**
**Wetting Properties of Molten Silicon with Graphite Materials**: *Arjan Ciftja*[1]; Merete Tangstad[2]; Thorvald Engh[2]; [1]SINTEF; [2]Norwegian University of Science and Technology

The wetting behavior of molten-silicon/refractory-materials system is important in refining and casting of silicon with respect to production of low cost solar cells. Here we study the wetting properties of molten silicon with several graphite materials used in the PV industry. The sessile drop method is employed to measure the contact angles. Initially molten silicon does not wet graphite materials. The initial contact angles measured are approximately 120°. As soon as the silicon melts, it starts to react with C to form SiC. Equilibrium wetting angles of 0 – 31° are measured. With increasing surface roughness the equilibrium wetting angles decrease. Infiltration of molten silicon is also studied. The results show that infiltration depth of molten silicon increases with the average pore size of graphite materials.

**11:10 AM**
**Mechanical Properties of Fine Grained Polysilicon Grown in Fluidized Bed Reactors**: *Mohamad Zbib*[1]; David Bahr[1]; Wayne Osborne[2]; Grant Norton[1]; [1]Washington State University; [2]REC Solar Grade Silicon LLC

A new method of growing polycrystalline silicon for solar applications is the fluidized bed reactor, which produces a fine grained granular bead from chemical vapor deposition. The beads vary from a few millimeters to a centimeter in diameter. During handling this material may crack; therefore, there is an interest in understanding the toughness and hardness of the material. Using nanoindentation and indentation fracture testing the mechanical properties as a function of grain size and annealing conditions were measured. Post grown annealing does not change the hardness of the beads (9.5 GPa), but does alter the toughness from between 1.1 MPa m^0.5 at 1050°C to 2.3 MPa m^0.5 at 600°C. The grain sizes and small voids within the sample due to the growth mechanisms did not affect the typical fracture path, suggesting that porosity at this scale is not the dominant feature controlling the fracture of this material.

**11:35 AM Panel Discussion**
**Recycling Needs led by Gregory Hildeman, Solar Power Industries**

## Solid-State Interfaces: Toward an Atomistic-Scale Understanding of Structure, Properties, and Behavior through Theory and Experiment: Atomic-Level Structure and Composition

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee
*Program Organizers:* Michael Demkowicz, Massachusetts Institute of Technology; Douglas Medlin, Sandia National Laboratories; Emmanuelle Marquis, University of Oxford

Monday AM Room: 602
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Michael Demkowicz, Massachusetts Institute of Technology; Emmanuelle Marquis, University of Oxford

**8:30 AM Invited**
**Atomic-Scale STEM-EELS Mapping of Structure, Chemistry, Bonds and Electronic Properties across Functional Interfaces**: *Christian Colliex*[1]; [1]CNRS

Aberration corrected STEMs now deliver and raster angström-size electron probes on cross-sectional foils with solid-state interfaces parallel to the incident beam. Bright and annular dark field images deliver views of the structural arrangement of the atomic columns across such interfaces. In parallel, EELS spectroscopy has demonstrated its efficiency for mapping with a high level of spatial resolution, the electronic response of the irradiated specimen. It thus provides maps, atomic column by atomic column, of the nature of the atoms across interfaces between artificially grown layers for electronics, spintronics or oxitronics components. This diagnosis is now extended to the determination of their bonding states through the monitoring of the spectral fine structures which modulate the characteristic edges. Various practical examples will be shown to demonstrate recent successes and present limitations of these new potentialities, claiming for further developments in theory and modelling in order to optimize the information thus accessible.

**9:00 AM**
**Atomic Scale Structure and Composition across G/G' Interfaces in Ni-Base Superalloys**: *Srinivasan Rajagopalan*[1]; J.Y. Hwang[2]; Soumya Nag[2]; A. Singh[2]; G.B. Viswanathan[3]; J. Tiley[4]; Rajarshi Banerjee[2]; Hamish Fraser[1]; [1]The Ohio State University; [2]University of North Texas; [3]UES Inc.; [4]Air Force Research Laboratory

The interface between the ordered γ' and the disordered γ phase in Ni-base superalloys plays a significant role in determining physical and mechanical properties. Using advanced characterization techniques such as aberration-corrected HRSTEM, EELS and 3DAP Tomography, the nature of the order-disorder and compositional transitions across the γ/γ' interface have been investigated in Rene' 88 DT, a multicomponent Ni-base superalloy. The variation of the interface width for different types of γ' precipitates (primary and secondary) has also been studied as a function of cooling rate and aging time. Using measurements based upon HRSTEM and 3DAP observations, the mechanism of γ' coarsening in Rene' 88 DT is rationalized, based upon existing models. Finally, the implications of the observations on the nucleation and evolution of different generations of γ' precipitates are discussed.

**9:20 AM**
**Chemomechanical Analysis of Metal Nanoparticle Interfaces under Extreme Environments via Molecular Dynamics Simulations**: *Hansohl Cho*[1]; Krystyn Van Vliet[1]; [1]Massachusetts Institute of Technology

Metal nanoparticles, capable of exothermic reactions upon mechanical mixing, can offer new means of controlled reactions under extreme environments. Forced mixing of nickel and aluminum via mechanical stress input results in extensive energy dissipation with extreme deformation and alloying reactions.

Here, we present our study of the chemomechanical behavior of the Ni and Al nanoparticles under mechanical impact, up to the point of shock loading, using nonequilibrium molecular dynamics simulations. We find that the interfacial mixing and alloying are accompanied by rapid thermal energy release. Thus, kinetic energy dissipation can be achieved in the extreme mechanical stress states at the nanoscale Ni/Al interface via high speed impact loading. We discuss the effect of nanoparticle radius on the extent of this effect. Finally, we consider the effects of impact loading conditions and boundary conditions that affect the extent and duration of energy dissipation within the Ni and Al nanoparticles.

**9:40 AM**

**Measurement of the Interface Width by Atom Probe Tomography**: *Michael Miller*[1]; Ai Serizawa[1]; [1]ORNL

Accurate measurements of solute gradients at interfaces, precipitate concentrations, and interface widths are important for understanding materials properties. In this study, the interface width was estimated from the distance between the 10 and 90% points of the solute concentrations in the precipitates and the matrix in the proximity histogram. Simulations indicate that a minimum interface width of ~0.15 nm may be obtained under optimum conditions. However, artifacts, such as the overestimation of concentrations, are generated under some data analysis conditions. The accuracy of these measurements will be demonstrated with a combination of simulations and experimental data from reactor pressure vessel steels and nanostructured ferritic alloys.This research was sponsored by the U.S. Department of Energy, Division of Materials Sciences and Engineering; research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

**10:00 AM Break**

**10:20 AM Invited**

**Structure and Chemistry of Nanometer-Thick Intergranular Films at Au-Al2O3 Interfaces**: *Wayne Kaplan*[1]; Mor Baram[1]; [1]Technion - Israel Institute of Technology

The existence of nanometer-thick intergranular equilibrium films at metal-ceramic interfaces has been experimentally verified for the Au-Al2O3 system. The films were formed by equilibrating thin Au films on a sapphire substrate which was previously partially wetted with drops of anorthite glass (CaO-2SiO2-Al2O3). The process resulted in sub-micron equilibrated Au particles residing on glass drops and on the sapphire substrate adjacent to the glass drops. The interfaces between the Au particles and the sapphire substrates were characterized using a monochromated and aberration corrected TEM (Titan 80-300 S/TEM). The thickness of the films was determined from quantitative HRTEM and the composition was determined from combined energy filtered TEM and energy dispersive spectroscopy (EDS). The use of aberration corrected HRTEM proved the existence of structural order within the films, which serves as part of the entropy term balancing the positive and relatively large Hamaker coefficients for the interface.

**10:50 AM**

**The Structure of Uranium Dioxide Grain Boundaries and its Influence on Fission Gas Segregation**: *Pankaj Nerikar*[1]; Chris Stanek[1]; Blas Uberuaga[1]; Susan Sinnott[2]; [1]Los Alamos National Laboratory; [2]Department of Materials Science and Engineering, University of Florida

In many materials, defects preferentially segregate to grain boundaries often resulting in a profound effect on the properties of the material. This is especially important for xenon (Xe) segregation in uranium dioxide. It is well known that Xe migrates to grain boundaries, where bubbles may form resulting in fuel swelling. Atomistic simulations often reveal phenomena that are necessary to fully understand material behavior. The atomic structures of symmetric S5 tilt, S5 twist, and amorphous grain boundaries in uranium dioxide are explored in this work using empirical potentials and density functional theory. We have found that for both the S5 tilt and S5 twist boundary, there is an unsymmetrical structure that is lower in energy than the symmetric structure. Xe segregation to these boundaries is also investigated in this work. Our simulations predict the segregation energy to be sensitive to the local atomic environment of the solute atom in the host.

**11:10 AM**

**Spatially Resolved Compositional Measurements across Interfaces, Phase Separations, and Non-Conservative Faults in Complex Oxides**: Srinivasan Rajagopalan[1]; G.B. Viswanathan[2]; David McComb[3]; Jan Ringnalda[4]; *Hamish Fraser*[1]; [1]The Ohio State University; [2]UES Inc.; [3]Imperial College London; [4]FEI Company

Complex oxides have been the subject of intense study in the recent past. Of particular interest is the ability to characterize interfaces and faults in nanometer-scale thin films and multilayered versions of these materials, requiring structural and compositional resolutions at or near to the atomic scale. An advantageous technique involves the aberration-corrected (scanning) transmission electron microscope; when the spherical aberration of probe is corrected, compositional information can be determined with very high spatial resolution. This paper presents the results of an assessment of the ability to perform spatially resolved measurements of compositional variations across phase separations and faults in strontium titanate thin films, and interfaces between strontium and barium titanate multilayered couples. The role of intrinsic and extrinsic factors affecting these measurements will be discussed. Support from the National Science Foundation for the Materials Research Science and Engineering Center (MRSEC) under contract number DMR-0820414 is gratefully acknowledged.

**11:30 AM**

**Nanoscale Characterization of a Nanostructured Fe-$Y_2O_3$ Composite Material**: *Mathilde Brocq*[1]; Bertrand Radiguet[2]; Fabrice Legendre[1]; Fabien Cuvilly[2]; Philippe Pareige[2]; Jean-Marie Lebreton[2]; [1]SRMP - CEA; [2]GPM-Université de Rouen

To be able to understand nanostructured materials microstructure, a set of nanoscale characterization techniques able to complement each other are needed. A special attention has to be paid on interface since those materials, by definition, contain a significant proportion of interfaces.An iron based material reinforced by nanometric yttrium oxide precipitates was studied because of its interest for ODS (Oxide Dispersion strengthened) steels considered for future nuclear reactors. It was synthesized by reactive ball-milling and annealing. It was then characterized in the as-milled and as-annealed state by Electron Probe Micro-Analysis (EPMA), X-Ray Diffraction (XRD), Mössbauer spectroscopy (MS) and Atom Probe Tomography (APT). Matching results give a precise and reliable description of the two phases of the material and of their evolution during the process. On the other hand, comparison of MS and APT results reveals an aberration in APT reconstruction of interface which will be presented in detail.

**11:50 AM**

**Chemical Interface Width and Triple Line Transport in Metallic Multilayers**: *Guido Schmitz*[1]; Patrick Stender[1]; Constantin Ene[2]; Henning Galinski[1]; [1]Westf. Wilhelms-Universität; [2]Universität Göttingen

Based on the thermodynamics of inhomogeneous systems, it is expected that the chemical transition at interfaces, even between two immiscible materials, cannot be atomically sharp. We could demonstrate by atom probe tomography that the width of interfaces in stable metallic multilayers varies systematically with temperature. Systems Ag/Cu, Cu/NiFe and Fe/Cr were analyzed using a wide-angle tomographic atom probe (WATAP). Owing to the outstanding resolution of this instrument, a broadening of interfaces can be demonstrated on a depth scale between 0.4 and 1.5 nm. Since these metallic systems are immiscible from a thermodynamic point of view, Cahn-Hilliard theory is used to explain the observed temperature dependence.In addition, the large volume of analysis of modern atom probes allows statistically significant observation of atomic transport along one-dimensional defects lines, which are interpreted as triple lines of the grain structure. Mobility of individual triple lines, diffusion coefficients and segregation behaviour are determined.

## Surface Engineering for Amorphous-, Nanocrystalline-, and Bio-Materials: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
*Program Organizers:* Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International Univesity; Sudipta Seal, University of Central Florida; Narendra Dahotre, University of Tennessee

Monday AM Room: 604
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Narendra Dahotre, University of Tennessee

**8:30 AM Introductory Comments**

**8:35 AM Invited**
**Development of Multi-Functional Nanostructured and Composite Coatings for Tribological Applications**: *Ali Erdemir*[1]; [1]Argonne National Laboratory

During last decade or so, great strides have been made in the development and utilization of nanostructured and composite coatings for a wide range of engineering applications. These coatings are much more robust and multi-functional than their earlier predecessors and are well-suited for demanding mechanical and tribological applications. In this paper, the primary focus will be on the design, synthesis, and mechanical and tribological characterization of a new generation of super-hard nanocomposite coatings that can have great application potentials in the transportation and manufacturing sectors. Specifically, we will introduce a crystal-chemical model that can help identify the specific coating ingredients that are needed to form these nanocomposite coatings that will attain ultra-low friction and wear under dry and lubricated sliding conditions. These designer coatings provide friction coefficients of less than 0.05 under severe boundary lubricated sliding conditions and they cannot be scuffed under the heaviest loadings that are available in reciprocating and block-on-ring type test machines. Because of their extreme resistance to wear and scuffing, they are well-suited for a wide range of engine applications as well as machining and manufacturing applications that require high precision and durability in molds, dies, and tools. Recent test results from bench-top and fired engine tests will be presented to illustrate the much superior tribological properties for these designer coatings over a broad range of sliding conditions. A variety of surface analytical tools are also used to understand the fundamental tribological mechanisms of these coatings after tests under severe conditions.

**9:00 AM Invited**
**The Tribological Behaviour of Graded Nanocrystalline Nickel Coatings**: *Sundararajan G*[1]; Nitin Wasekar[1]; [1]ARCI

Pulsed electrodeposition technique is ideally suited to deposit thick, nanocrystalline coatings on a variety of substrates. By controlling the pulse parameters it is possible to obtain coatings which are graded in terms of grain size. In this study, thick Ni coatings have been deposited on a mild steel substrate using pulsed electrodeposition so as to obtain a grain size grading along the thickness direction.At one extreme, the grain size increased from 25 nm at the coating surface continuously to 500 nm at the coating-substrate interface. At the other extreme, the grain size decreased from a value of 500 nm at the coating surface to 25 nm at the coating-substrate interface. A number of intermediate variants within the above two extremes were also obtained. The sliding wear and solid particle erosion behaviour of all these variants were evaluated and the results will be presented and analysed.

**9:25 AM Invited**
**Evolution of Microstructure in Laser Clad Coatings Studied by Orientation Imaging Microscopy**: *Václav Ocelík*[1]; Ivan Furár[1]; Jeff De Hosson[1]; [1]University Groningen/M2i

Laser powder deposition of thick metallic coatings is one of the surface engineering techniques which provide resistant against high loading impact, severe wear and corrosion at high temperatures. In this work Orientation Imaging Microscopy (OIM) based on Electron Back Scatter Diffraction in a Scanning Electron Microscope was employed as a very powerful instrument for the study of the relationship between processing parameters and the microstructure of individual and overlapping laser tracks at different processing speeds. OIM provided new insights into the microstructure of laser clad coatings and very useful information concerning the directional grow of individual grains, the solidification texture and the shape of solidification front during laser processing. Strong correlations between the microstructure, processing parameters as well as mechanical performance were discovered.

**9:50 AM**
**Residual Stresses in PS 304 Tribological Coating**: *Pnina Ari-Gur*[1]; Simon Narasimhan[1]; Mark Croft[2]; Zhong Zhong[3]; Thomas Gnäupel-Herold[4]; Malcolm K. Stanford[5]; Christopher DellaCorte[5]; Phillip B. Abel[5]; [1]Western Michigan University; [2]Rutgers University; [3]Brookhaven National Laboratory; [4]National Institute of Standards and Technology; [5]National Aeronautics and Space Administration

Oil-free turbomachinery need high-temperature hard coatings to withstand the harsh conditions. The $PS_30_4$ coating was developed by NASA for that purpose. It is a composite of Ni-Cr, $Cr_2O_3$, eutectic $BaF_2$-$CaF_2$, and Ag, and is applied by plasma spray. It can be deposited on variety of substrates. One of the metrics for performance is the cohesive strength of the coating. The best cohesive strength is achieved via heat treatment in air. The goal now was to analyze the residual stresses developed following stages of the process (plasma spray, heat treatment, and surface grinding). Stresses with spatial resolution of 0.1 mm were measured, allowing depth profiling of the coatings and into the substrate near the interface. Consistently the coatings exhibit compressive stresses in the circumferential direction and tensile stresses in the axial direction. There is a modest stress relaxation in the coating near the substrate interface. The substrate stresses are generally low.

**10:10 AM**
**Microstructural Assessment Associated with Micropitting in Rolling Contact Fatigue**: *Fang Cao*[1]; Peter Jacobs[1]; Martin Webster[1]; [1]ExxonMobil Research and Engineering

Micropitting is a surface fatigue phenomenon encountered in bearings and gears under lubricated conditions, which leads to their premature failure. The failure mechanisms of micropitting are complicated by the nature of the tribological contact where a number of factors such as load, speed, sliding, specific film thickness, and lubricant chemical composition can play a critical role. In this study, experiments have been carried out in a three-contact roller disc machine to assess the influence of these factors on micropitting of roller samples made from AISI52100 bearing steel. The formation of micropitting under different test conditions was studied in detail by metallographic investigation of the roller samples in both plain-view and cross-section, with an emphasis on the changes of microstructure and chemical compositions associated with the top surface layer of the sample. Possible mechanisms for micropitting formation and alleviation remedies will be discussed based on these experimental results.

**10:30 AM Break**

**10:45 AM**
**Engineering Non-Stick, Pro-Stick/Adhesion and Anti-Corrosion Surfaces with Self-Assembled Monolayer of Phosphonate (SAMP) Technology**: *Eric Bruner*[1]; [1]Aculon, Inc.

Self-Assembled Monolayer of Phosphonates (SAMP) were developed as a technology platform. SAMP coating solutions have formulated for numerous markets including optical, display, electronics, and industrial coatings. These coatings outperform all known alternatives in characteristics such as adhesion, stain resistance, and scratch resistance. Surface treatments must be mechanically and chemically stable under conditions experienced in the intended area of use. SAMPs can impart any of these properties to metals, metal oxides and even some polymer surfaces by drawing on its library of structurally tailored phosphonic acids.The key is covalent bonding, which creates a uniquely strong attachment between the SAMP and substrate. The SAMP is one approximately 1.5 nm thick. It completely covers the material to which it is applied, and assures total surface coverage regardless of the type or texture of that material. The composition of the SAMP determines the properties that it imparts to its substrate.

**11:05 AM**
**Anisotropic Nanaofriction Behavior of Aligned Carbon Nanotube Carpet**: *Jiangnan Zhang*[1]; Yuekai Sun[1]; Lijie Ci[1]; P.M. Ajayan[1]; Jun Lou[1]; [1]Rice University

The adhesion and frictional properties of surfaces coated with vertically and transversely aligned multi-walled carbon nanotube (MWCNT) carpet was investigated in ambient, around 10% RH humidity and nitrogen condition by atomic force microscope (AFM). The measured friction forces are higher for vertically aligned CNT carpet than for transversely aligned CNT carpet in all above environments. The friction force was found to be critically influenced by the relative humidity, with the decreasing relative humidity the friction force drops. The adhesive force data which was determined from force-displacement curves shows the adhesion forces are lower for transversely aligned CNT carpet than for vertically aligned CNT carpet while the adhesion forces drop as the relative humidity was decreased.

**11:25 AM**
**Dry Sliding Wear of Nanocrystalline Al - 12.6 at. % Si**: *I. Baker*[1]; M. Gwaze[1]; Y. Sun[1]; A.T. Dohner[1]; A. Grosse[1]; T. Tran[1]; F.E. Kennedy[1]; P.R. Munroe[2]; [1]Dartmouth College; [2]University of New South Wales

Wear resistance of many materials has been found to be proportional to their hardness. Thus,one might expect wear resistance to increase with decreasing grain size and nanocrystalline alloys to show exceptionally good wear resistance. Since sliding wear has been shown to refine the near-surface grain structure, it could be argued that nanoscale grains would not significantly affect wear behavior. This presentation will describe the results of a study designed to determine whether the wear rates of nanocrystalline eutectic Al-Si, produced by cryomilling followed by compaction using backpressure-assisted equal channel angular extrusion, are lower than material of the same composition produced by drop casting. Pin on disc wear tests were performed in air and argon against a Y-stabilized ZrO2 counterface. Scanning electron microscopy and transmission electron microscopy, including energy dispersive X-ray mapping, were used to examine the worn pin surfaces. Work supported by U.S. NSF Grant CMMI 0651642.

**11:45 AM**
**Wear Resistance and Adherence of TiO2 Sol-Gel Thin Films**: Miguel Alterach[1]; Pablo Favilla[1]; *Mario Rosenberger*[1]; Alicia Ares[1]; Carlos Schvezov[1]; [1]Universidad Nacional de Misiones - CONICET

The wear resistance and adherence of TiO2 films was studied; the films were synthesized by the sol-gel dip-coating technique on a grade 5 titanium alloy. Monolayer and multilayer films were deposited by varying: dip-coating velocity, aging time of sol and the heat treatment parameters. These process conditions had influence on the color (thickness) of the film and the cracks formation. The wear resistance was measured on a ball-on-flat machine using a rotating glass ball as counterface. The worn films were evaluated by the size of the superficial scare, which can be noted by the change of color on the surface. The adherence was measured on a scratch test machine and examined by optical microscope to determine the adherent critical load. The best wear resistance and adherence of the films was measured on a trilayer film fabricated with a heat treatment at 500ºC for 1h for each layer.

**12:05 PM**
**Fretting Corrosion Behaviour of Untreated and Surface Engineered Ti-6Al-4V Alloy**: *Satendra Kumar*[1]; Sankara Narayanan TSN[1]; Ganesh Sundara Raman S[2]; Seshadri S.K.[2]; [1]National Metallurgical Laboratory, Madras Centre; [2]Indian Institute of Technology Madras

Fretting corrosion is one of the major modes of failures observed in hip and knee implants which lead to a reduction in life-time of prosthesis. Surface engineering is a viable option to improve the fretting corrosion behaviour of these implants. The present paper will address the fretting corrosion behaviour of surface engineered Ti-6Al-4V alloy in Ringer's solution and compared with that of the untreated one. The methods used for surface modification are thermal and anodic oxidation. The change in free corrosion potential (FCP) measured as a function of time with the onset of fretting, during fretting and after stopping off the fretting motion is used to evaluate the fretting corrosion behaviour. Thermally oxidized Ti-6Al-4V alloy exhibit a better fretting corrosion resistance than the anodized and untreated one. The study concludes that thermal oxidation is a suitable surface engineering method for improving the fretting corrosion resistance of Ti-6Al-4V alloy.

## The Aluminium Industry – Perspectives on our Future: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
*Program Organizer:* Geoff Bearne, Rio Tinto Alcan

Monday AM — Room: Ballroom 6A
February 15, 2010 — Location: Washington State Convention Center

*Session Chair:* Geoff Bearne, Rio Tinto Alcan

**8:30 AM Introductory Comments**

**8:35 AM Plenary**
**The Challenges that Aluminium Faces as Material of Choice**: *Frank Field*[1]; [1]Massachusetts Institute of Technology; [2]CRU International Ltd

Abstract not available.

**9:05 AM Plenary**
**The Strategic Impact of Changing Energy Markets on the Aluminum Industry**: Robin Adams[1]; *Kelly Driscoll*[2]; [1]CRU Strategies; [2]CRU International Ltd

Aluminum is the most energy-intensive of the major commodity metals and the industry's smelting strategy has always been driven by the search for cheap power. In the past 5 years there have been clear signs of a structural change in energy prices, both for primary energy and electricity. What does this mean for the aluminum industry? In particular how will it affect the competitive structure of existing alumina refineries and aluminum smelters, the locations where new projects are constructed and the long-run equilibrium price for both alumina and metal? The paper will explore recent structural changes in global markets for oil, gas and coal as well as the state of play regarding carbon taxes and climate change. It will examine how these, along with regulatory changes around the world, are impacting on the price of thermal energy in regions with competitive bauxite-alumina potential and on the price power available to the aluminum industry. It will include a special evaluation of the changing situation in China. On this basis it will develop the competitive implications for today's refineries and smelters, the likely locations for tomorrow's successful projects and the all-important impact on long-term price levels in the industry.

**9:35 AM Plenary**
**The Impact of Economic Highs and Lows on Aluminium Smelter Construction**: *Cesar Inostroza*[1]; [1]SNC-Lavalin Inc.

Since 1990, SNC-Lavalin has been a major player in 17 of the 28 largest aluminium smelter projects globally (outside China and Russia). Currently, SNC-Lavalin is engaged in the design and construction of 5 major aluminium smelter projects - Emal, Dubal, Qatalum, Boyne Smelter Upgrade and the Jonquiere Pilot Plant. The cyclical nature of this commodity-driven business is characterized by inherent risks that have an impact on decision making on aluminium smelter construction. The business case for new projects has also demanded the need to look at more difficult and complex projects, which are located in more remote regions, with limited access to a skilled labour force and operating under tighter environmental standards. This presentation will present an overview of the impact of the economic cycles on the main drivers in smelter construction cost, and will attempt to provide mechanisms to mitigate these impacts.

**10:05 AM Break**

**10:25 AM Plenary**
**Challenges and Opportunities Relative to Increased Usage of Aluminum within the Automotive Industry**: *Mark W. Verbrugge*[1]; Paul E. Krajewski[1]; Anil K. Sachdev[1]; James G. Schroth[1]; David R. Sigler[1]; Blair E. Carlson[1]; [1]General Motor Research and Development Center

The various alloys and processes associated with the use of aluminum within the automotive industry are overviewed, and key issues and challenges that stand in the way of increased aluminum use are highlighted.

**10:55 AM Plenary**
**Aluminum's Sustainability Strategy**: *Steve Williamson*[1]; [1]ARCO Aluminum

Lightweight, corrosion resistant and ease of fabrication have been Aluminum's value proposition for over 100 years. With the advent of green-marketing, Aluminum is updating its Sustainability profile accordingly. Tasked by the Aluminum Association's Board of Directors in 2008, the member companies mandated a re-staffing and re-focus of the Association's efforts on a consolidated Sustainability Strategy. The Association had successfully delivered relevant product and end-use segment recycling, safety and productivity activities for decades. Through the traditional product and market focus, the packaging, transportation, and the building and construction markets were served and promoted. Now, staffed with experienced Sustainability managers, the Aluminum Association is embarking on a two-lane approach to a comprehensive Sustainability Strategy. The first objective is to build a robust data set that will enable each product and segment committee to respond to their respective downstream consumers. Simultaneously, the Association is creating a communications plan that will tell Aluminum's Sustainability story to key stakeholders and policy makers. Just a year into the initiative, and a year fraught with significant macroeconomic turmoil, the Aluminum Association's Sustainability Strategy has taken shape and is gaining traction.

**11:25 AM Plenary**
**Aluminum Recycling in a Carbon-Constrained World: Observations and Opportunities**: *Subodh Das*[1]; [1]Phinix LLC

This presentation will discuss the key aspects of aluminum recycling for several market sectors such as transportation, packaging, aerospace and building and construction in light of existing and anticipated debate on climate changes, carbon trading, carbon constraints and sustainability enhancement. The specific topics to be presented will be in the areas of commercialization of recycling –friendly alloys, development of recycling indices and carbon foot print of various aluminum alloys, and some thoughts and suggestions on enhancing recycling rates and use of recycled materials for developing new aluminum markets and products.

**11:55 AM Concluding Comments**

## The Vasek Vitek Honorary Symposium on Crystal Defects, Computational Materials Science and Applications: Computational Materials Science I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
*Program Organizers:* Mo Li, Georgia Institute of Tech; David Srolovitz, Institute for High Performance Computing, Agency for Science, Technology and Research, Singapore; Adrian Sutton, Imperial College London; Vaclav Paidar, Institute of Physics AS CR vvi; Jeff De Hosson, Univ of Groningen

Monday AM Room: 603
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Adrian Sutton, Imperial College London; David Pope, University of Pennsylvania

**8:30 AM Introductory Comments**

**8:35 AM Charles McMahon**

**8:50 AM David Pettifor**

**9:00 AM Keynote**
**Atomistic Modeling of Defects through the Ages**: *Vasek Vitek*[1]; [1]University of Pennsylvania

We discuss the long-term development of the atomic level modeling of defects in materials and relation to our understanding of their mechanical behavior. The three major themes are: Understanding of atomic bonding and the limits this imposes on what can be studied by modeling, interpretation of the results and relation between calculations and experiments. First we discuss the development starting from pair-potentials through many-body central-force potentials, tight-binding methods up to the DFT based calculations and show for each significant contributions albeit on different levels. In interpretation we are particularly interested in paths towards development of theoretical analyses enabled by modeling. Finally, we emphasize that the link with experiment is crucial for both validation of the studies and for motivation of new investigations. Such experiments need to involve all levels, starting with atomic, such as HREM, up to macroscopic, such as studies of orientation dependencies of yielding in single and polycrystals.

**9:25 AM Invited**
**Challenges in Modelling TCP Phase Formation in Ni-Based Superalloys**: *David Pettifor*[1]; Bernhard Seiser[1]; Thomas Hammerschmidt[2]; Aleksey Kolmogorov[1]; Ralf Drautz[2]; [1]University of Oxford; [2]ICAMS

Modelling topologically close-packed (TCP) phase formation in Ni-based superalloys is a challenging task with only the semi-empirical PHACOMP and NewPHACOMP schemes providing some guidance. In this talk we develop a microscopic understanding of the factors that control TCP phase stability by coarse-graining the electronic structure from density functional theory (DFT) to tight-binding (TB) to bond-order potentials (BOPs). These many-body interatomic BOPs are unique in that they depend explicitly on the electron per atom (e/a) ratio, so that they predict the structural sequence from bcc to sigma to chi to hcp that is found, for example, across the W-Re system. The observed stability of the mu and Laves phases requires the additional contribution of size and electronegativity differences. Remaining challenges in modelling TCP phase formation are briefly discussed.

**9:50 AM Invited**
**Dislocation-Based Simulation of the Migration of Low-Angle Grain Boundaries**: *David Srolovitz*[1]; Adele Lim[2]; Mikko Haataja[2]; [1]Yeshiva University; [2]Princeton University

Understanding the mechanisms of grain boundary migration are complicated by the complexity of the structure of general boundaries, the high dimensionality of the boundary parameter space and the large number of dynamic degrees of freedom in the system. We, therefore, focus on low angle grain boundaries, the structure and dynamics of which can be described in terms of lattice dislocations and their glide, climb, and reactions. We perform a series of 2 and 3-dimensional simulations of stress driven migration of low angle grain boundaries where the dynamical variables are the location and Burgers vectors of the dislocations. We investigate the effect of dislocation climb mobility, boundary inclination, and misorientation on the mechanisms and mobilities of such boundaries. Next, we develop analytical models for boundary mobility that are in excellent agreement with both simulation and experimental results.

**10:15 AM Break**

**10:40 AM Invited**
**Discrete Dislocation and Multi-Scale Analyses of Fatigue Crack Growth**: *Alan Needleman*[1]; [1]University of North Texas

Processes at a variety of scales play a role in the fatigue crack growth of crystalline metals. The mesoscale, where discrete dislocation effects need to be accounted for, plays a central role in mediating between atomic scale effects and macroscale dissipation processes that can be modeled appropriately using conventional continuum descriptions. The organized dislocation structures near a crack tip give rise to much higher stress levels to drive atomic scale processes than are predicted by conventional continuum plasticity. Some discrete dislocation plasticity predictions for fatigue crack growth will be discussed along with some recent steps taken to carry out multi-scale analyses of fatigue crack growth in metal single crystals.

**11:05 AM Invited**
**Displacive Processes in Systems with BCC Parent Lattice**: *Vaclav Paidar*[1]; [1]Institute of Physics AS CR vvi

The changes of sample shape are caused by plastic deformation or by martensitic phase transformations. In both cases the mechanisms of atomic rearrangements are based on collective displacements of atomic aggregates. The internal structure of dislocations, carriers of plastic deformation, can be examined using the energies of generalized stacking faults displayed by so called $\gamma$-surfaces calculated for bcc metals by Prof. Vitek already more than forty years ago. This approach can be extended to the shuffling of atomic planes that plays a crucial role in martensitic phase transformations. Similarities and differences between displacive processes of local lattice shearing and atomic plane alternate shuffling will be discussed.

**11:30 AM Invited**

**Coupling of the Continuum Theory of Dislocations and Structural Phase Transformations at the Mesoscale**: *Roman Groger*[1]; Turab Lookman[2]; [1]Academy of Sciences of the Czech Republic; [2]Los Alamos National Laboratory

Collective motion of dislocations often gives rise to correlated dislocation domains and to spatially inhomogeneous microstructure with unconventional mechanical properties. In multiphase materials the evolution of crystal structure can be studied using well-developed concepts such as the mean-field Landau theory. However, theoretical studies of plasticity in multiphase materials are rather scarce owing to a complicated coupling of the dislocation density and the underlying microstructure. In this talk, we propose a self-consistent scheme in which the coupling of the order parameter field with the dislocation density is accomplished using a set of incompatibility constraints. When these physical constraints are incorporated into the Landau free energy functional, one arrives at a mesoscopic model of phase transformations mediated by dislocations. This model serves to bridge the length scales by allowing for the systematic incorporation of data from both atomistic simulations and macroscopic experimental measurements.

## Three-Dimensional Materials Science VI: Three-Dimensional Crystallography and Grain Boundary Analysis

*Sponsored by:* The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Phase Transformations Committee

*Program Organizers:* Alexis Lewis, Naval Research Laboratory; Anthony Rollett, Carnegie Mellon University; David Rowenhorst, Naval Research Lab; Jeff Simmons, AFRL; Stuart Wright, EDAX Inc-TSL

Monday AM Room: 401
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* David Rowenhorst, U S Naval Research Laboratory; Stuart Wright, EDAX-TSL

**8:30 AM Invited**

**Deriving the Grain Boundary Character Distribution and Relative Grain Boundary Energies from Three Dimensional EBSD Data**: *Gregory Rohrer*[1]; [1]Carnegie Mellon University

The proliferation of the dual-beam focused ion-beam scanning electron microscope has allowed for more routine collection of data from calibrated serial sections. Combining this technique with electron back-scatter diffraction (EBSD) allows for a complete description of the complete description of the crystallography of all of the grain boundaries within a polcrystal. The grain boundary character distribution (GBCD) is an important description of the grain boundary network that is thought to be a significant indicator of materials properties. Serial sections of EBSD data have been collected and reconstructed in 8% yttria-stabilized zirconia, yittria, SrTiO3, and nickel. Different methods for calculating the GBCD from 3D reconstructed microstructures are compared with a more established stereological analysis of 2D data. Implications of the results on our understanding of relative grain boundary energies and the structure of grain boundary networks in polycrystals will be discussed.

**9:00 AM**

**Deriving the Relative Grain Boundary Areas and Energies in Nickel from Three Dimensional EBSD Data**: *Jia Li*[1]; Gregory Rohrer[1]; Shen Dillion[2]; [1]Carnegie Mellon University; [2] University of Illinois at Urbana-Champaign

The three dimensional interfacial network of grain boundaries in polycrystalline nickel has been characterized using a combination of electron backscatter diffraction mapping and focused ion beam serial sectioning. These data have been used to determine the relative areas of different grain boundary types, categorized on the basis of lattice misorientation and grain boundary plane orientation. Using the geometries of the interfaces at triple lines, relative grain boundary energies have also been determined as a function of lattice misorientation and grain boundary plane orientation. Grain boundaries comprised of (111) planes have, on average, lower energies than other boundaries. Asymmetric tilt grain boundaries with the S9 misorientation also have relatively low energies. The grain boundary energies and areas are inversely correlated.

**9:20 AM**

**Calculation of Grain Boundary Angles at Triple Junctions in 3D Digitized Microstructures**: *Michael Chandross*[1]; Elizabeth Holm[1]; [1]Sandia National Laboratories

Digitized microstructures are generated in both simulations and experiments. Because of the discrete nature of digitized grain boundaries, it is difficult to measure dihedral angles at triple junctions in these microstructures. We recently developed a method that utilizes linear fits to grain boundaries followed by a comparison of correlation coefficients to accurately calculate triple junction angles (TJAs) in digitized microstructures. We use this method to analyze cross-sections of 3D simulated microstructures as well as various experimental micrographs. Histograms of TJAs show the expected peak about 120 degrees. Simulated microstructures agree well with experiment. Details of the angle distribution indicate the accuracy of grain growth simulations (lattice effects, faceting) or the distribution of grain boundary energies in experimental systems (anisotropy). Further, we note specific peaks that can be used to determine the microstructural dimensionality (i.e. columnar vs. equiaxed) from a 2D cross-section.

**9:40 AM Break**

**10:10 AM Invited**

**Three-Dimensional Grain Boundary Networks: Modeling and Connections to Experimental Data**: *Megan Frary*[1]; [1]Boise State University

The grain boundary network that makes up a microstructure plays an important role in determining the properties of the material. These networks are well-studied in two dimensional systems; however, most microstructures are inherently three dimensional, so an understanding of the role microstructure plays in determining properties must account for the full 3D microstructure. Here, percolation-based models are applied to 3D grain boundary networks with both regular and irregular grain shapes. The microstructures are characterized in terms of the cluster size distribution, mean cluster size, and radius of gyration; grain boundary area must be accounted for when calculating these properties in 3D. The analytical tools developed to characterize simulated microstructures can be applied to experimentally-determined data sets to extract the same metrics from those microstructures. Developing an understanding of 3D connectivity in real microstructures may elucidate the role of grain boundary character distribution on property improvement observed during grain boundary engineering.

**10:40 AM**

**Three Dimensional Analysis of Grain Curvature and Crystallography**: *David Rowenhorst*[1]; Alexis Lewis[1]; George Spanos[1]; Gregory Rohrer[2]; Anthony Rollett[2]; [1]US Naval Research Laboratory; [2]Carnegie Mellon University

The state of serial sectioning has now allowed for statistically relevant structures to be analyzed, often containing thousands of objects within the sampling volume. We will briefly present our methodology for serial sectioning, including crystallographic orientations from EBSD in a single phase, beta-titanium alloy. The resultant reconstruction contains over 4300 grains, within the 200 sections taken. In the results, we will present the analysis of the grain boundary curvature and use this as a measure of the grain growth rate. Furthermore the correlation between boundary curvature to the crystallographic interface of the grain boundary will be examined.

**11:00 AM**

**Synthesizing Annealing Twins in Three-Dimensional Voxel-Based Microstructures**: *Lisa Chan*[1]; Anthony Rollett[1]; Gregory Rohrer[1]; [1]Carnegie Mellon University

In face-centered cubic metals, Grain Boundary Engineering typically involves maximizing the density of high symmetry boundaries to improve grain boundary dependent properties, such as the resistance to intergranular corrosion and fatigue cracking. These high symmetry boundaries are denoted by low sigma values for Coincident Site Lattice relationships. In the studies of increasing the fraction of low sigma boundaries, twinning events have been found to be very effective in introducing low sigma boundaries. In this study, annealing twins are added into a digitally simulated microstructure such that the number fractions and twin cluster statistics match with the values observed

in experimental structures. The resulting texture and grain boundary character distribution are also compared against the experimental measurements.

**11:20 AM**

**Crystallographic Orientation Determined from the Pattern of Solidified Structure**: *Hisao Esaka*[1]; Kei Shinozuka[1]; [1]National Defense Academy

Solidified structure is complex, since the growth directions of dendrites are not in accordance with the observed plane. Many ghost lines are observed. Dendrite of cubic metals has been simplified with rod and four plates with the same thickness. Then, the ghost lines have been analyzed applying the solid analytical geometry. In order to characterize the solidified pattern, angles and the ratio of width of ghost lines have been formulated using the angles, which determine the orientation of dendrite. The computer program has been developed to determine the spatial orientation of dendrite from the characteristic parameters of ghost lines. Applying this computation, it was found that the cross sections of dendrite model performed by 3D-CAD agreed well with the solidified structure. Furthermore, the crystallographic orientation estimated from the solidified pattern agreed with EBSP data of the cast specimen of Al-20 mass% Cu alloy.

**11:40 AM**

**3D Monte-Carlo Simulation of Microstructural Evolution upon Heating of Deformed LCB Titanium Alloy**: *Sergii Shevchenko*[1]; Orest Ivasishin[1]; Elena Pereloma[2]; Azdiar Gazder[2]; [1]Institute for Metal Physics; [2]University of Wollongong

The evolution of microstructure, texture and grain boundary statistics during thermal treatment of deformed LCB titanium alloy was modeled using a 3D Monte Carlo (Potts) approach. The initial microstructure, texture (ODF), spatial distribution of the stored energy of deformation (cold drawn, 70%). The modeling technique allowed to compare how differences in nucleation mechanisms and kinetics affect recrystallization kinetics and microstructure and texture evolution. It was shown that oriented nucleation led to formation of recrystallization texture, which, however, smeared during subsequent grain growth. Also, oriented nucleation resulted in slower recrystallization rate in comparison with the case of not oriented nucleation. Results of the MC Potts modeling were verified experimentally by analysis of textures and microstructures (EBSD orientation maps) at different stages of recrystallization and grain growth.

## Ultrafine Grained Materials – Sixth International Symposium: Processing-Microstructure-Properties I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee

*Program Organizers:* Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Monday AM — Room: 606
February 15, 2010 — Location: Washington State Convention Center

*Session Chairs:* S. Lee Semiatin, U.S. Air Force Research Laboratory; En (Evan) Ma, Johns Hopkins University; Xavier Sauvage, University of Rouen, CNRS; Henry Rack, Clemson University

**8:30 AM Introductory Comments**

**8:35 AM Invited**

**Superior Properties of Ultrafine-Grained Metals Produced by SPD Processing**: *Ruslan Valiev*[1]; [1]Ufa State Aviation Technical University

Over the last years there appeared a number of observations stating that ultrafine-grained (UFG) metals and alloys produced by severe plastic deformation can exhibit unique and extraordinary mechanical and physical properties that differ fundamentally from their conventional, coarse-grained counterparts. The present report considers several new examples of that kind - effect of superstrength revealed in several nanostructured light alloys, appearance of super-ductility in UFG Al alloy already at room temperature and a number of other unique features of physical and mechanical behaviour of nanometals. Findings of such unusual properties are of high scientific interest as these properties are attributed to novel physics of deformation and phase transformations in UFG materials. Practical importance of these works arises from the development of metallic materials of new generation. These aspects are considered and discussed in the report with the emphasis on the phenomena origin and strategies for achievements.

**8:55 AM**

**Nanostructures and Magnetic Properties of FePd Alloys Processed by Severe Plastic Deformation**: *Xavier Sauvage*[1]; Abdelahad Chbihi[1]; Didier Blavette[1]; Dmitry Gunderov[2]; A.G. Popov[3]; [1]University of Rouen, CNRS; [2]Ufa State Aviation Technical University; [3]Institute of Metal Physics

The intermetallic FePd alloy was processed by severe plastic deformation (high pressure torsion) both in the ordered and the disordered state. The resulting materials are nanostructured with a grain size smaller than 100nm. Using x-ray diffraction, transmission electron microscopy and magnetic properties measurements some strain induced disordering was revealed. The ordering kinetics during post deformation aging treatment was also investigated. In the optimum state, nanoscaled ordered domains give rise to a record coercivity of about 1400 Oe.

**9:10 AM Invited**

**High Tensile Strength and Ductility in Nanocrystalline and Ultrafine-Grained HCP Cobalt**: *Xiaolei Wu*[1]; Yuntian Zhu[2]; [1]Institute of Mechanics, Chinese Academy of Sciences; [2]North Carolina State University

Tensile property and work hardening behavior were investigated in both nanocrystalline (NC) and ultrafine-grained (UFG) cobalt produced by the surface mechanical attrition treatment. The yield strength of UFG Co and NC Co is around 3 and 5 times higher than that of the coarse-grained counterpart, with the uniform plastic strains being as high as 8.6% and 6.1% respectively. In particular, the annealing results in a significant increase in ductility and work hardening rate as compared with their deformed states, with plastic strains reaching 13.8% and 8.3% respectively. TEM observations show the presence of basal stacking faults of high density in both UFG and NC grains. The successive formation of SFs and their interplay with dislocations, together with the formation of NC grains during tension enhances the work hardening and uniform ductility.

**9:30 AM**

**Effect of Strain Path and Texture on Grain Refinement in Severe Plastic Deformed Copper**: *Chengfan Gu*[1]; Laszlo Tóth[2]; Rimma Lapovok[1]; Chris Davies[1]; [1]Monash University; [2]Université Paul Verlaine de Metz

OFHC copper were obtained by ECAP, route Bc, up to four passes and cold rolled to a thickness reduction of 97.5% (total true strain of 4.26) respectively. The crystallographic textures and microstructure of obtained samples were measured by X-ray diffraction and EBSD. It has been found that the microstructure in the ECAPed copper was much more refined with respect to the rolling case (at same equivalent strain) leading to smaller grain sizes in ECAP.The grain refinement between ECAP and rolling can be interpreted by the model based on lattice distortion of crystals due to the constraining effects of their neighbors in the polycrystal [Tóth L.S. et al, ICSMA15, 2009]. The model produces subgrains in orientations controlled by the orientation of the grain boundaries and by the rate of lattice rotation. It is applied in this study to reproduce the differences in grain sizes and the texture developments.

**9:45 AM**

**Superior Grain Refinement via Intelligent ECAE Processing of Materials**: *Suveen Mathaudhu*[1]; Laszlo Kecskes[1]; Jae-Taek Im[2]; David Foley[2]; Majid Al-Maharbi[3]; Ibrahim Karaman[2]; K. Ted Hartwig[2]; [1]U.S. Army Research Laboratory; [2]Texas A&M University; [3]Sultan Qaboos University

Severe plastic deformation (SPD) by equal channel angular extrusion/pressing (ECAE/ECAP), has been recognized as a premier processing tool for obtaining ultrafine grained or nanocrystalline (UFG/NC) microstructures in bulk metal samples. While the progress in grain refinement capabilities has been significant, the majority of results have been demonstrated on metals and alloys with f.c.c. crystal structures, and relatively fine starting grains sizes and homogeneous textures. In this paper, we will present results which show that factors such as texture, chemistry, and starting grain size play a significant

role on the grain refinement potential of all metallic systems, including h.c.p. and b.c.c. crystal structures. Examples of enhanced grain refinement will be given for b.c.c. Ta, Nb and W, h.c.p. Mg- and Ti- alloys, and hexagonal Bi2Te3. The results presented will demonstrate concrete examples of superior grain refinement by incorporation of unconventional rotation routes, chemical homogenization, and pre-processing.

**10:00 AM Invited**

**High Pressure Torsion of Pure Metals for Universal Plot**: Kaveh Edalati[1]; *Zenji Horita*[1]; [1]Kyushu University

Several pure metals such as Al, Ag, Au, Cu, Ni, Pt and Fe are processed by high-pressure torsion (HPT). Hardness first increases with an increase in strain and then saturates to a steady state when plotted against equivalent strain. The hardness increase is more rapid as the pressure increases but the saturation levels are the same under different pressures. When the plotting is made with the equivalent strain multiplied by pressure and divided by shear modulus, it is shown that all data points under different pressures fall on a single curve. It is also shown that when the hardness is normalized by shear modulus with an inclusion of the homologous temperature for HPT operation, a universal plot is obtained for all data points of different pure metals covered in this study except for Al where recovery is very fast.

**10:20 AM Break**

**10:35 AM Invited**

**Nanostructures, Grain Refinement and Mechanical Properties in Al–Mg Alloys Subjected to High Pressure Torsion**: *Hans Roven*[1]; Manping Liu[2]; Maxim Murashkin[3]; Ruslan Valiev[3]; Tamas Ungár[4]; Levente Balogh[4]; [1]Norwegian University of Science and Technology (NTNU); [2]Shanghai Jiao Tong University; [3]Ufa State Aviation Technical University; [4]Eötvös University

Recent results on nanostructures, grain refinement and mechanical properties in Al–Mg alloys subjected to high pressure torsion will be reviewed. Typical nanostructures of non-equilibrium grain boundaries, full dislocations and Shockley partials, deformation microtwins and stacking faults, as well as hexagonal and rhombic shaped nanostructures have been identified using high-resolution transmission electron microscopy. Grain size distributions and dislocation densities and densities of planar defects were quantified by X-ray diffraction (XRD) and high-resolution XRD line profile analysis. Microhardness and tensile properties of the Al–Mg alloys were comparatively investigated. The major part of the presentation will focus upon the formation mechanisms of the typical nanostructures, the refinement and strengthening mechanisms associated with the typical nanostructures and faults. A possible formation process of the hexagonal and rhombic shaped nanostructures will be proposed based on molecular-dynamics simulations. The role of Mg solute on the formation, refinement and strengthening mechanisms will also be considered.

**10:55 AM**

**Crystal Size Influences the Propensity for Deformation Twinning**: *Evan Ma*[1]; Ju Li[2]; Qian Yu[3]; Zhiwei Shan[4]; Jun Sun[3]; [1]Johns Hopkins University; [2]University of Pennsylvania; [3]Xi'an Jiaotong University; [4]Hysitron Inc.

Deformation twinning (DT) in crystals is a highly correlated inelastic shearing process which controls the mechanical behavior of many engineering materials. It is known that deformation twinning can become more difficult in small grains and crystals. Using micro-compression and in situ nano-compression experiments, we find that deformation twinning in a Ti–5at%Al alloy is overtaken by less correlated, ordinary dislocation plasticity (ODP), when its physical dimension d is reduced to below a critical dc ~ 1 micrometer. A phenomenological stimulate-slip model has been developed to explain the strong crystal/grain size dependence for DT. The large transition size is far above that (~ 20 nm) for the switch of deformation mechanisms in nanocrystalline materials and easily accessible in experiments, making our findings highly relevant for practical applications.

**11:10 AM**

**Enhanced Strain Hardenability of Uniform, Submicrocrystalline Dual-Phase Steel Processed via Equal Channel Angular Pressing and Intercritical Annealing**: *Young Gun Ko*[1]; C.W. Lee[2]; S. Namgung[2]; D.H. Shin[2]; [1]Yeungnam University; [2]Hanyang University

Tensile deformation behavior is investigated of submicrocrystalline dual-phase steel (DPS) via equal channel angular pressing (ECAP) accompanied by heat treatment in an intercritical region, with a specific focus on strain hardening behavior. A fully martensite structure is used as a preform microstructure because easy dissolution of carbon atoms during ECAP leads to a uniform distribution of each constituent phase (martenisite + ferrite) during intercritical annealing treatment. From tension tests, the submicrocrystalline DPS exhibits greater strain hardenability than what submicrocrystalline steels behave, resulting in a tensile strength of ~ 1 GPa with a total elongation of ~ 20 pct. The strain hardening behavior is here analyzed based on strain gradient model.

**11:25 AM Invited**

**Superplasticity in Nanocrystalline Metallic and Ceramic Materials**: *Amiya Mukherjee*[1]; [1]University of California

Significant differences, including higher flow stresses and enhanced strain hardening rates in nanocrystalline structure, have been observed in the deformation characteristics of nanocrystalline materials at elevated temperatures as compared to their microcrystalline counterparts. Conventional understanding of elevated temperature crystalline plasticity cannot explain these observations. Cooperative grain boundary sliding (CGBS) in superplasticity has shown to account for the majority of macroscopic strain in microcrystalline materials. In this work, nanocrystalline $Ni_3Al$ produced via High Pressure Torsion is deformed superplastically in the TEM. In-situ tensile testing shows the nature of CGBS at the nanoscale through direct observation of this phenomenon. The second part of this presentation is devoted to demonstrating the potential of using spark plasma sintering to consolidate nanocrystalline powder and the subsequent superplastic deformation of the compact to near-finished shape. This procedure has provided one of the lowest temperature, high strain rate superplastic formings of nanocrystalline ceramics to date.

**11:45 AM**

**ECAE Processing of Pure and Mg Alloy Powders: Effect of Confinement, Route, and Temperature**: *Laszlo Kecskes*[1]; Kristopher Darling[1]; Micah Gallagher[2]; Suveen Mathaudhu[1]; David Foley[3]; Robert Barber[3]; Karl Hartwig[3]; [1]US Army Research Laboratory; [2]Dynamic Science, Inc.; [3]Texas A&M University

Severe plastic deformation processing by ECAE has been applied to coarse-grained ZK-60 Mg alloy powders. The effects of confinement, route, and temperature were studied to elucidate the break-down of the prevailing, intermetallic-inclusion-interposed, laminar structure of rolled ZK-60 alloy plate. We relied on routes A, C, or E, at a range of temperatures up to 300oC, to explore inherent limitations of dispersion and homogenization in solid, as-received ZK-60 alloy rods. At the same time, to examine potential improvements to the ZK-60 alloy microstructure, we side-by-side extruded prealloyed ZK-60 powders and co-milled blends of Mg, Zr, and Zn powders with the nominal ZK-60 composition. Scanning electron microscopy, texture measurements, energy dispersive x-ray analysis, and microhardness measurements were applied to characterize the resultant extrudates. A comparison of extrudates from both top-down and bottom-up approaches are discussed.

**12:00 PM**

**Shape Memory Characterization of Aged Ti-50.6Ni**: *Henry Rack*[1]; Astrid Mueller[1]; Erica Sampson[1]; Ruslan Valiev[2]; [1]Clemson University; [2]Ufa State Aviation Technical University

This presentation will present the results of a study which has examined the shape memory response of ultra-fine grained Ti-50.6 Ni aged at temperatures between 400 and 550C for times to 100 hrs. These results will be contrasted with solution treated and aged Ti-50.6Ni with the variations in shape memory transformations temperatures and latent heats of reaction being described in terms of the microstructure observed utilizing x-ray, scanning and transmission electron microscopy.

**12:15 PM Invited**

**The Brittle-To-Ductile Transition in Severely Deformed Low Carbon Steel**: *Masaki Tanaka*[1]; Kenji Higashida[1]; Tomotsugu Shimokawa[2]; [1]Kyushu University; [2]Kanazawa University

The brittle-to-ductile transition (BDT) behavior was investigated in low carbon steel deformed by an accumulative roll-bonding (ARB) process. The temperature dependence of its fracture toughness was measured by conducting four-point bending tests at various strain rates. The fracture toughness increased while the BDT temperature decreased in the specimens deformed by the ARB process. Arrhenius plots between the BDT temperatures and the strain rates indicated that the activation energy for the BDT did not change due to the deformation with the ARB process. It indicated that the decrease in the BDT

temperature by grain refining was not due to the reduction in the dislocation mobility with respect to short-range barriers. Quasi-three-dimensional simulations of discrete dislocation dynamics indicated that the decrease in the number of dislocation sources decreases in the DBT temperature. The roles of grain boundaries will be also discussed in order to explain the decrease in the BDT temperature.



## Ultrafine Grained Materials – Sixth International Symposium: Processing-Microstructure-Properties II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee

*Program Organizers:* Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Monday AM Room: 607
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Donald Lesuer, Lawrence Livermore National Laboratory; Megumi Kawasaki, University of Southern California; Challapalli Suryanarayana, University of Central Florida; Zenji Horita, Kyushu University

**8:30 AM Introductory Comments**

**8:35 AM Invited**

**Flow Stress Anisotropy and Tension-Compression Asymmetry in Ultrafine Grained AZ31B Magnesium Alloy**: *Ibrahim Karaman*[1]; Majid Al-Maharbi[1]; David Foley[1]; Irene Beyerlein[2]; K.Ted Hartwig[1]; Suveen Mathaudhu[3]; Laszlo Kecskes[3]; [1]Texas A&M University; [2]Los Alamos National Laboratory; [3]U.S. Army Research Laboratory

AZ31B Magnesium alloy has been processed at 150-200°C using equal channel angular extrusion (ECAE) up to four passes following four different routes. The flow stress anisotropy and tension-compression (T/C) asymmetry along the three orthogonal directions of the ECAE billet were monitored. The texture evolution during ECAE was measured and predicted using a viscoplastic self-consistent crystal plasticity model. The initial grain orientations with respect to the ECAE die greatly influence the type of deformation modes during ECAE and hence resultant texture and grain morphology. If basal planes are oriented perpendicular to ECAE shear plane, non-basal slip systems are activated which suppresses dynamic recrystallization during ECAE. Flow stress anisotropy and T/C asymmetry after ECAE are highly dependent on crystallographic texture. It is demonstrated that controlling starting texture, ECAE temperature, route and number of passes provide some control over resulting texture, grain size and morphology, and thus, flow stress anisotropy and T/C asymmetry.

**8:55 AM**

**Role of Ultrafine Grain Size in the HCP - FCC Allotropic Transformation in Ti, Zr, and Hf**: Uma Seelam[1]; Gagik Barkhordarian[2]; *C. Suryanarayana*[1]; [1]University of Central Florida; [2]GKSS Research Center

Allotropic HCP - FCC transformations were observed in mechanically milled Ti, Zr, and Hf powders. While the transformation was observed in powders milled under regular milling conditions, no such phase transformation was observed when the powders were milled in an ultra-high purity environment by placing the mill inside an argon-filled glove box. From a critical analysis of the width and intensity of X-ray diffraction peaks and chemical analysis of the milled powders, it was inferred that the transformation was associated with the formation of nanocrystals in the milled powder. It was also concluded that the HCP - FCC phase transformation was, at least partially, due to pick-up of interstitial impurities by the powder during milling of these powders to the nanocrystalline state. Isothermal equation of state was used to estimate the stability of the FCC phase in the Group IV B metals.

**9:10 AM**

**Ultra-High Strength of Nanocrystalline Iron-Based Alloys Produced by High Pressure Torsion**: *Tadahiko Furuta*[1]; Shigeru Kuramoto[1]; Tetsu Osuna[1]; Zenji Horita[2]; [1]Toyota Central R & D Labs., Inc.; [2]Kyusyu University

Elastic and plastic deformation behaviors were investigated in a nanocrystalline Fe-19%Ni-34%Co-8%Ti (in at%) alloy produced by High Pressure Torsion (HPT) at room temperature with a rotating speed of 1 rpm under a pressure of 6 GPa. Applying the HPT process, the average grain size appears to be 20-50 nm with high density crystal defects and such an intense grain refinement have never been reported in bulk iron alloy. The alloy shows ultimate tensile strength (UTS) of about 2.75 GPa and Young's modulus (E) of about 150 GPa with the total tensile elongation of 8 - 10 %. As a result, the ratio of UTS/E for the present alloy is ~0.02, which is substantially higher than conventional metallic materials. It is inferred from this high value of UTS/E that the dislocation motion, which governs the plastic deformation behavior of metallic materials, is inhibited by the grain refinement up to ultra-high stress level.

**9:25 AM Invited**

**Fatigue Behavior of Friction Stir Processed Ultrafine Grained Aluminum and Magnesium Alloys**: *Rajiv Mishra*[1]; Partha De[1]; Rajeev Kapoor[1]; Wei Yuan[1]; [1]Missouri University of Science and Technology

The fundamental micromechanism of fatigue deformation changes from microcrystalline to ultrafine grained (UFG) materials. The well proven concepts of persistent slip bands and extrusion/intrusion features in microcrystalline alloys do not apply directly to UFG materials because of the grain length scale and change in the dislocation micromechanisms. In the recent years, friction stir processing is emerging as a convenient tool for producing very fine grained and UFG materials with high fraction of high angle boundaries. Additionally it results in refinement and homogenization of constituent particles in commercial aluminum alloys. Initial results indicate that the UTS normalized fatigue stress for 107 cycles increases significantly for commercial aluminum and magnesium alloys. An overview of the S-N curves and the changes in micromechanisms will be presented and discussed. A conceptual dislocation dynamics-microstructural length scale framework has been developed to rationalize the enhancement in fatigue strength.

**9:45 AM**

**Influence of Grain Boundary Character and Strain Rate on the Ductility of Ultrafine Grained AA 5052**: *Rajeev Kapoor*[1]; Nilesh Kumar[1]; Rajiv Mishra[1]; [1]Missouri University of Science and Technology

Presented here is a study of ultrafine grained AA 5086 (a solid solution hardening Al-2.5%Mg alloy) obtained by two methods, one through friction stir processing and the other through the continuous equal channel angular pressing technique for four different conditions of 1, 2, 3 and 4 passes. The microstructure and fraction of high angle grain boundaries for the different conditions of materials is determined using electron backscattered diffraction (EBSD) in a SEM-OIM. Tensile tests on miniature size samples are carried out at room temperature at strain rates ranging from 10<-2> to 10<-4> s<-1>. The effect of decreasing strain rate on the total and uniform elongation is studied and compared with earlier work of Sabirov et al. (2008). The work-hardening behavior and its relation to strain localization is compared for the different the differently processed UFG materials. The effect of strain rate on the interaction of microshear-bands with grain boundaries and their influence on both uniform and total elongation is investigated. The influence of increasing fraction of high angle boundaries on both ductility and uniform elongation is presented and compared with earlier works.

**10:00 AM**

**Fatigue Crack Growth Behaviour of Ultrafine Grained Copper**: *Jelena Horky*[1]; Golta Khatibi[1]; Brigitte Weiss[1]; Michael Zehetbauer[1]; [1]Faculty of Physics, University of Vienna

Previous literature on the fatigue crack growth behaviour of ultrafine grained and nanocrystalline metals processed by electrodeposition or cryomilling shows enhanced crack growth rates compared to coarse grained metals especially in the near threshold regime related to a less tortuous crack path (see e.g. T. Hanlon et. al., Int. J. Fatigue 27 (2005) 1147). First investigations on SPD processed materials show the same tendency.The aim of the current work was to shed more light on crack propagation behavior of SPD processed metals, at the example of Cu. HPT processing has been chosen since it achieves a broader range of grain sizes than e.g. ECAP. A number of experiments were carried out

on specimens which were subjected to a certain thermal treatment after HPT processing. The results show correlations between the microstructure, crack propagation rate and ductility of HPT copper.

**10:15 AM**

**Implications of Deformation under Constraint in Development of UFG Microstructure in an Austenitic Stainless Steel**: Chiradeep Gupta[1]; J. B. Singh[1]; Swetha Mulki[2]; *R. Kapoor*[3]; Apu Sarkar[1]; I. Samajdar[2]; J. K. Chakravartty[1]; [1]Bhabha Atomic Research Centre; [2]Indian Institute of Technology, Powai; [3]Department of Materials Science and Engineering, Missouri University of Science and Technology

The widely used methods of production of ufg microstructure rely on the application of SPD by either ECAP or high pressure torsion. These methods essentially are based on the application of shear deformation on the sample to obtain the refinement in microstructure. In metastable austenitic stainless steels however, application of deformation leads to formation strain induced martensite, which may hinder the extent of refinement achievable by SPD. In this work the effects of application of constrained deformation is explored and compared with those carried out without constraint for the propensity for strain induced martensite formation. Preliminary results indicate that constrained deformation, as using plane strain compression, suppress the formation of strain induced martensite. The effect of application of ultra high deformation in both cases are explored in terms of changes in microstructure characterized by TEM and SEM-EBSD and changes in mechanical properties by compression tests at room temperature.

**10:30 AM Break**

**10:45 AM Invited**

**The Ambient-Temperature Mechanical Properties of UFG Ag with Nanotwins Using Microshear Tests**: *Michael Kassner*[1]; Andrea Hodge[1]; [1]USC

The mechanical properties of ultrafine grained (UFG) 150 µm silver interlayers prepared by planar magnetron sputtering were studied using microshear torsion tests. The interlayers have very high ductility in pure shear, comparable to conventional grain sizes, and show a mechanical steady-state. The Hall-Petch behavior at 1.25 µm is consistent with other earlier work. The hardening rates (ds/de) are substantially higher in the UFG Ag. The saturation stress and strain-rate sensitivity at this stress are identical to coarse-grained silver.

**11:05 AM**

**Formation of Ultrafine Grains during Friction Stir Processing of Ti-6Al-4V**: *Adam Pilchak*[1]; James Williams[2]; [1]Universal Technology Corporation; [2]The Ohio State University

Friction stir processing was applied to modify the surface of an investment cast and hot isostatically pressed titanium alloy. In a single pass, the fully lamellar microstructure was refined to sub-micron sized globular α grains when the peak temperature was kept below the β-transus. The microstructure was examined with electron microscopy and electron backscatter diffraction. The stir zone was found to be free from microtexture, which is prevalent in α+β forged products, and is detrimental to fatigue properties. Analysis of the transition zone between the unaffected base material and the stir zone revealed that grain refinement revealed that the sub-micron grains formed by deformation induced lattice rotations within individual α lamellae. Due to the plastic anisotropy of the α-phase, a significant orientation dependence was noted. This talk will describe the grain refinement mechanisms operative during friction stir processing of cast Ti-6Al-4V and discuss them in terms of crystal plasticity theory.

**11:20 AM**

**Coarsening-Induced Fatigue-Crack Initiation in Several Nanocrystalline Nickel Alloys**: Henry Padilla[1]; *Brad Boyce*[1]; Paul Kotula[1]; Elizabeth Holm[1]; [1]Sandia National Labs

Nanocrystalline nickel alloys exhibit impressive fatigue-life performance, even beyond that associated with Hall-Petch strengthening. Fatigue-crack initiation resistance appears to be the key to their enhanced performance, since crack propagation proceeds with ease. Microstructural analysis using Focused Ion Beam dissection of several nanocryrstalline Ni alloys (Ni, Ni-Mn, Ni-Fe) reveals regions of coarsened grains at the crack initiation zone. This fatigue-induced coarsening is thought to be a necessary precursor for fatigue crack initiation. Therefore, for these nanocrystalline alloys, grain-boundary stabilization is a pathway towards improved fatigue performance. Interestingly, alloys which are stable against thermally-induced coarsening are not necessarily stable against fatigue-induced coarsening. A Potts model provides insight into the role of Zener pinning and solute drag on thermally- and mechanically-induced grain growth.* Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract No. DE-AC04-94AL85000.

**11:35 AM Poster Preview**

**Texture and Microstructure Evolution in Ultrafine Grained AZ31 Processed by EX-ECAP**: *Milos Janecek*[1]; [1]Charles University

**11:40 AM**

**Effect of Strain Reversals on Processing by High-Pressure Torsion**: *Megumi Kawasaki*[1]; Byungmin Ahn[1]; Terence Langdon[1]; [1]University of Southern California

Processing by high-pressure torsion (HPT) is a continuous operation in which the sample remains within the HPT facility so that, unlike equal-channel angular pressing (ECAP), it is difficult to develop different processing methods. However, reversing the direction of torsional straining is available in HPT after straining in the forward direction. Experiments were conducted on pure Al using either monotonic-HPT (m-HPT) or cyclic-HPT (c-HPT) for totals of 1 to 4 turns at room temperature under an applied pressure of 6.0 GPa. Detailed observations were taken to evaluate the microstructural damage introduced by reversal straining on the surface of each disk. Microhardness values were measured both along diameters in each disk and over the complete surfaces in order to construct color-coded contour maps of the hardness distributions. Additional microstructural analyses permit an evaluation of the significance of strain reversal during processing by HPT.

**11:55 AM Invited**

**Strengthening Mechanisms in Deformed and Annealed Nanostructured Metals**: *Xiaoxu Huang*[1]; Naoya Kamikawa[2]; Niels Hansen[1]; [1]Risø National Laboratory for Sustainable Energy, Technical University of Denmark; [2]Tohoku University

The yield strength of nanostructured metals processed by plastic deformation to ultrahigh strain has been analyzed by using the boundary spacing as the strengthening parameter in a Hall-Petch equation. However, the deformed microstructure is composed of different structural features such as dislocation boundaries, high angle boundaries and loose dislocations between boundaries. It follows that several strengthening mechanisms may contribute to the yield stress and that a strength-structure relationship might include more structural parameters than just the boundary spacing. In the present study the relative contribution to the yield stress from different strengthening mechanisms are varied by annealing deformed samples to different stages of recovery and recrystallisation followed by a quantification of structural (strengthening) parameters. The structure-strength relationships are then analyzed and it is found that not only dislocation and grain boundary hardening contribute to the yield stress but also mechanisms such as precipitation hardening and dislocation source hardening.

**12:15 PM**

**Composition and Structure of Nitrogen-Containing Dispersoids in Tri-Modal Metal Matrix Composites**: *Clara Hofmeister*[1]; Bo Yao[1]; Yongho Sohn[1]; Timothy Delahanty[2]; Mark van den Bergh[3]; Kyu Cho[4]; [1]University of Central Florida; [2]Pittsburgh Materials Technologies, Inc.; [3]DWA Aluminum Composites; [4]U.S. Army Research Laboratory

Aluminum tri-modal composite reinforced with B4C particulates has been fabricated successfully, and exhibited an extremely high yield strength and tailorable ductility. The fabrication of this composite starts from the cryomilling of 5083 Al alloy powders with B4C particles, which yields agglomerates containing sub-micron B4C particles solidly bonded with nanocrystalline Al (NC-Al) grains. These agglomerates are then blended with coarse grain Al powders, and consolidated to form the bulk composite. In this study, secondary ion mass spectrometry was employed to determine the composition of Nitrogen that linearly varied a function of cryomilling time. Crystalline and amorphous dispersoids containing Nitrogen and other constituents were documented by analytical transmission electron microscopy. The influence of composition and structure of the dispersoids on the strength of the composite will be discussed.

**12:30 PM**

**Nano-Scale Strengthening from Grains, Sub-Grains and Particles in Fe-C Alloys**: *Donald Lesuer*[1]; Chol Syn[1]; Oleg Sherby[2]; [1]Lawrence Livermore National Laboratory; [2]Stanford University

The nano-scale grain-size strengthening effect has been studied relative to the strengthening influence of other microstrucural features such as sub-grains, particles and solid solution additions. In Fe-C alloys these microstructural features can result from phase transformation and deformation processes. The formation of lath and plate martensite during quenching and severe plastic deformation will be shown to result in very high strengths (4600 MPa) which can be correlated with nano-scale grain sizes and interparticle spacing. The development of a nano-scale grain size in the Fe-C alloys will be shown to be controlled by the inter-related influence of deformation, transformation products and ultra-fine particles. Particles, grains and subgrains will be shown to provide significantly higher strengthening than contributions from solid solution effects.

## 2010 Functional and Structural Nanomaterials: Fabrication, Properties, Applications and Implications: Nano-Sensors and Magnetic Properties

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee

*Program Organizers:* David Stollberg, Georgia Tech Research Institute; Nitin Chopra, University of Alabama; Jiyoung Kim, University of Texas - Dallas; Seong Jin Koh, University of Texas at Arlington; Navin Manjooran, Siemens Corporation; Ben Poquette, Keystone Materials; Jud Ready, Georgia Tech

Monday PM Room: 214
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Seong Jin Koh, University of Texas at Arlington; Nitin Chopra, The University of Alabama

**2:00 PM Introductory Comments**

**2:05 PM Invited**

**Gold Nano-Engineered Mercury Sensor for Alumina Refineries**: *Suresh Bhargava*[1]; [1]RMIT University

The toxicity of mercury remains a threat to the environment and public health despite a number of efforts by government bodies world-wide. Mercury is a common environmental pollutant that is neurotoxic and bioaccumulative. Currently, mercury sensors which are primarily based on ultraviolet techniques are plagued by cross-sensitivity issues. However, when the humble Quartz Crystal Microbalance (QCM) is combined with well formed gold nano-engineered surfaces these cross-sensitivity issues can be overcome without compromising sensitivity. Using a single step electrochemical approach, a QCM loaded with nano-engineered gold structures has an increase in sensitivity of approximately 340% to 180% towards 1 to 10mg/m3 of Hg vapor over non-modified sensors when operating at 89°C, respectively. What is more promising is that over many months of continuous testing in the presence of ammonia and high humidity contaminated simulated streams, the sensor showed a maximum of ±6.6% in response magnitude towards the tested concentrations.

**2:25 PM**

**Fundamental Studies and On-Chip Integration of Nanoporous Energetic Silicon**: *Collin Becker*[1]; Luke Currano[2]; Wayne Churaman[2]; Conrad Stoldt[1]; [1]University of Colorado; [2]U.S. Army Research Lab

Porous silicon (PS) is a widely studied material and holds great potential in the realization of novel Microelectromechanical systems (MEMS). When impregnated with any one of a number of oxidizers, porous silicon can function as an energetic material. Because silicon is widely used in micromachining and CMOS processes, PS can be integrated on-chip alongside a MEMS sensor. This new class of energetic can be used for applications requiring on-chip power, propulsion, and fuzing. The strength of the energetic reaction, which is comparable to traditional energetics, is controlled by altering processing parameters to tune PS thickness, porosity, specific surface area, and surface terminations. Currently, the correlation between PS morphology and energy release rate is unclear. Here we characterize the structure of the PS prior to ignition, using Fourier transform infrared spectroscopy, Raman spectroscopy, and scanning electron microscopy. This characterization is coupled with calorimetry data. Lastly, on-chip energetic PS devices are presented.

**2:45 PM**

**Gas Sensing Behavior of Nanostructured $CoSb_2O_6$ Prepared by a Colloidal Method**: *Hector Guillen-Bonilla*[1]; Carlos Michel[2]; Juan Moran-Lazaro[2]; Juan Reyes-Gomez[3]; Dario Pozas-Zepeda[3]; [1]Centro de Enseñanza Tecnica Industrial; [2]Universidad de Guadalajara; [3]Universidad de Colima

Nanostructured powders of $CoSb_2O_6$, possessing the trirutile-type structure, were synthesized by a colloidal method in ethyl alcohol. Microwave radiation was used to evaporate the solvent. The sample heated at 200°C was observed by SEM, revealing the presence microspheres having an average diameter of 2µm. The thermal decomposition at 700°C in air produced hollow nanostructured spheres having abundant nanoporosity; this observation was made by TEM. X-ray powder diffraction was used to identify the crystal structure. To test $CoSb_2O_6$ as a gas sensor material, the powder calcined at 700°C was deposited on alumina as thick films. DC and AC electrical characterization was performed in air, $O_2$ and $CO_2$. Polarization curves were recorded to obtain quantitative information about $CO_2$ and $O_2$ detection. The results indicate that $CoSb_2O_6$ detects changes in the surrounding atmosphere, at a temperature as low as 250°C.

**3:05 PM**

**Carbon Dioxide Gas Sensing Properties of $CoSb_2O_6$ Prepared by a Colloidal Method**: *Hector Guillen-Bonilla*[1]; Carlos Michel[2]; Juan Moran[2]; Juan Reyes[3]; Dario Pozas[3]; [1]Centro de Enseñanza Tecnica Industrial; [2]Universidad de Guadalajara; [3]Universidad de Colima

$CoSb_2O_6$ has a singnificant response in gas detection ($CO_2$). In order to improve its gas sensing properties it is necessary to have a better control on the size and shape of the particles. The colloidal methods are effective to control the microstructure inorganic materials. In this work $CoSb_2O_6$ was synthesized by the colloidal method. Single-phase $CoSb_2O_6$ was obtained after a calcination at 600°C in air; the crystal structure was identified by XRD. By SEM the formation of microcolumns was observed. These particles had a length between 10 to 29 µm. $CoSb_2O_6$ thick-films were tested as gas sensors. The electrical characterization was performed in alternating current (AC) in air and $CO_2$, at frequencies: 100 Hz, 1 kHz and 100 kHz. At 200ºC, the change of the magnitude of the impedance (Z) was 246 to 249 KO. The results indicated that $CoSb_2O_6$ would be a $CO_2$ gas sensing material.

**3:25 PM**

**Multi-Walled Carbon Nanotube Sensor Devices for Gas Sensing Applications**: Raghu Mangu[1]; Suresh Rajaputra[1]; Srikanth Durgamahanty[1]; Dali Qian[1]; Rodney Andrews[1]; *Vijay Singh*[1]; [1]University of Kentucky

Multi Walled Carbon Nanotubes (MWCNTs) based device configurations studied as part of this research work were simple and did not require any manipulation of individual or bundles of tubes, through techniques such as e-beam or photolithography. MWNTs grown inside the pores of insulating AAO matrix by a chemical vapor deposition (CVD), without the use of catalyst, was the first design. MWNTs grown as thick films on Si/SiO2 substrates via CVD process using Ferrocene as a catalyst was the second design. Both device configurations were integrated into resistance sensing devices. Steady state sensitivities as high as 5% and 10% for 100 ppm of NH3 and NO2 respectively were observed. Variations in sensor resistance with exposure to oxidizing and reducing gases were explained on the basis of charge transfer between the analyte and the MWCNTs, the latter behaving as p-type semiconductors.

**3:45 PM Break**

**3:55 PM**

**Electromechanical Coupling Behaviors of Suspended Low Dimensional Materials and Applications to Sensing**: *Hao Lu*[1]; Li Song[1]; P.M. Ajayan[1]; Jun Lou[1]; [1]Rice University

Electrical properties of suspended graphene and nanowires will be studied with coupling of stress induced by AFM (Atomic Force Microscope). Sample behavior will be probed both in air and different liquids. We'll investigate response of the suspended structures with respect to different environments and discuss the possibility of using it as a sensing mechanism.

**4:15 PM**
**Functionalization of Single TiO2 Nanotubes for Bio-Sensor Applications**: *Mingun Lee*[1]; Dongkyu Cha[1]; Jie Hunang[1]; Hyunjung Shin[2]; Moon J. Kim[1]; Jiyoung Kim[1]; [1]University of Texas at Dallas; [2]Kookmin University

Nanoscale structures have been widely investigated for emerging applications such as chemical and biological sensors. Thanks to their nontoxicity, large surface to volume ratio and hollow structure, single TiO2 nanotube devices hold great promise in that role. TiO2 nanotubes fabricated by combining ALD with nano-templates method behave like an n-type semiconductor, where the noticeable shift of their electrical conductance upon exposure to certain chemicals provides a means of detecting the targeted material. To achieve good selectivity between different biomaterials, a critical concern for biosensors, inner and outer surfaces of the nanotubes were functionalized by chemical compounds including biotin. In this study, we will present effects of TiO2 nanotube functionalization in bio-sensor applications. This research has been supported by a grant(code #: 2009K000469) from 'Center for Nanostructured Materials Technology' under '21st Century Frontier R&D Programs' of the Ministry of Education, Science and Technology, Korea

**4:35 PM**
**Enhanced Irreversibility Field and Critical Current Density in Superconducting NbC Integrated with Aligned Carbon Nanotubes**: *Guifu Zou*[1]; Hongmei Luo[2]; Scott Baily[1]; Yingying Zhang[1]; Junyi Zhai[1]; Jie Xiong[1]; Quanxi Jia[1]; [1]Los Alamos National Laboratory; [2]New Mexico State University

We report a novel chemical solution approach to integrate the superconducting NbC with oriented carbon nanotubes (CNTs). The NbC:CNTs composite shows improved irreversibility field (~ 5 T at 4.2 K), far greater than reported 1.2 T at 4.2 K. In addition, very high critical current densities 10^5 A/cm^2 are achieved at 3 T and 6 K. To the best of our knowledge, both the irreversibility field and the critical current density of the NbC composite are the highest reported in the literature. We believe that the aligned CNTs play a key role in the enhancement of pinning of vortex lines to increase the performance of superconducting NbC. Our results suggest that the incorporation of CNTs into superconductors has potential for high-field applications.

**4:55 PM**
**Nanoparticles with Double Perovskite La2BB'O6 Composition**: *Yuanbing Mao*[1]; [1]Washington State University

Magnetic semiconductors with near room temperature ferromagnetism have attracted much attention due to their broad potential applications. For example, double perovskite La2NiMnO6 exhibits a ferromagnetic order with Tc of ~280 K and large magnetic-field-induced changes in the resistivity and dielectric properties. Most previous studies were performed in bulk ceramic and thin film forms and proved that synthetic conditions determine the atomic order of the B-site sublattice and, as a result, the ferromagnetic properties of La2BB'O6 (B = Ni and Co; B' = Mn). Here, we first report the successful synthesis of La2BB'O6 nanoparticles with size ranging from 15-50 nm in diameter. These nanoparticles are characterized by power x-ray diffraction, transmission electron microscopy and Raman spectroscopy. The microstructure of these multifunctional nanostructured materials is investigated by analytical high-resolution TEM. To better understand their processing-structure-property relation, these results are combined with the measurements of their magnetic and electronic properties.

**5:15 PM Concluding Comments**

## Advances in Composite, Cellular and Natural Materials: Cellular and Porous Materials

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee
*Program Organizers:* Yuyuan Zhao, The University of Liverpool; David Dunand, Northwestern University

Monday PM Room: 305
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* David Dunand, Northwestern University; Markus Buehler, Massachusetts Institute of Technology

**2:00 PM Keynote**
**Cellular Materials in Nature**: *Lorna Gibson*[1]; Michael Ashby[2]; [1]MIT; [2]Cambridge University Engineering Department

Cellular materials are widespread in nature. Wood and cork have a honeycomb-like structure with cells that are roughly hexagonal prisms. Trabecular bone, plant parenchyma, adipose tissue, coral and sponge all have a foam-like structure, with polyhedral cells. Natural structures often have a cellular component: skulls and leaves of monocotyledon plants are sandwich structures, with dense outer skins separated by a foam-like core; animal quills and plant stems are nearly fully dense cylindrical shells supported by a foam-like core; and palm and bamboo stems are cylinders with radial density gradients. This talk provides an overview of cellular materials in nature and illustrates how the cellular structure gives rise to increased mechanical performance.

**2:40 PM**
**Multi-Scale Osteointegration of Biphasic Calcium Phosphate Bone Scaffolds**: *Amy Wagoner Johnson*[1]; [1]University of Illinois at Urbana-Champaign

In engineering porous ceramics for bone regeneration, most studies focus on optimizing the macroarchitecture. The few that focus on microstructure use it to enhance osteoconductivity. While microporosity improves bone growth in macropores, we are the first to show osteocytes embedded in bone within micropores <10um in size. Biphasic calcium phosphate (BCP)macroporous scaffolds with microporous rods were implanted in swine mandibular defects. Cells migrated into the interconnected micropores and bone formed throughout the rods, creating a co-continuous BCP/bone composite. Here we characterize the bone growth using several imaging modalities and show the first truly osteointegrated scaffold with integration at both the macro and the micro length scales, leaving no "dead space" or discontinuities of bone. The bone-filled micropores increase the bone-scaffold interface by orders of magnitude compared to bone that is limited to the macropores, and has important implications for the efficacy of such scaffolds in load bearing defects.

**3:00 PM**
**Shape-Memory NiTi Foams**: *David Dunand*[1]; [1]Northwestern University

NiTi (Nitinol) foams with shape-memory or superelastic properties are of interest for biomedical implants and for actuators. Here, we present various processes to create these foams based on sintering of NiTi powders together with space-holders which are removed to create porosity. The spaceholder can be fugitive, e.g NaCl evaporating during the sintering operation, or ice which is freeze-dried before sintering. The spaceholder can also remain during the sintering operation, leading to a NiTi/spaceholder composite. The spaceholder is then removed by chemical dissolution (e.g. NaF) or by electrochemical dissolution (e.g. steel). Microstructure and mechanical properties of these various foams, with equiaxed or elongated porosity (depending on the type of spaceholder), are also reported, focusing on the superelastic and shape-memory effect.

**3:20 PM**
**Mechanical and Biological Properties of Titanium Syntactic Foams**: Xiaobing Xue[1]; Victoria Kearns[1]; Rachel Williams[1]; *Yuyuan Zhao*[1]; [1]The University of Liverpool

Titanium syntactic foam is a novel composite material with hollow ceramic microspheres embedded in titanium matrix. This paper reports on the preliminary studies on the mechanical and biological properties of Ti syntactic foam manufactured by powder metallurgy. The density and porosity

were measured. The compression, three-point bending, indirect and direct contact cell culture tests were conducted. The density and porosity varied with compaction pressure. However, higher pressure resulted in a large number of crushed microspheres. The apparent modulus, compressive and flexural strength increased with increasing sintering temperature or sintering time. Indirect cell contact tests demonstrated that the titanium syntactic foams were non-cytotoxic. In direct cell contact tests the cells attached and spread well on the surface of titanium syntactic foams. These data suggest that this material warrants further investigation in bone replacement applications.

**3:40 PM Break**

**4:00 PM**

**Mechanical Behavior of Nanoporous Pt**: *Antonia Antoniou*[1]; Dhriti Bhattacharyya[2]; Pat Dickerson[2]; Nathan Mara[2]; [1]Georgia Institute of Technology; [2]Los Alamos National Laboratory

Nanoporous Pt (np-Pt) is synthesized by electrochemical dealloying of co-sputtered Ptx¬Si1-x amorphous films. The deposition and dealloying conditions were varied. As the Pt fraction in the amorphous alloy increases, Si dissolution is favored along pre-existing features of the amorphous film (i.e. column boundaries, surface asperities). The morphology of the resulting np-Pt depends on the manner in which silicon is preferentially removed. In addition to the expected isotropic open cell structure, anisotropic structures are also observed such as columnar and radial-type foam. The foam is found to be polycrystalline with 5 nm grains, and voids and ligaments range between 20-30 nm. Mechanical behavior has been examined by nanoindentation and micropillar compression.

**4:20 PM Invited**

**Elastic Modulus Study of Nanoporous Au Foams**: *Andrea Hodge*[1]; Monika Biener[2]; Juergen Biener[2]; [1]University of Southern California; [2]LLNL

The effect of Ag and the relative density on the elastic properties of nanoporous Au (np-Au) foams will be presented for partially as well as fully dealloyed np-Au samples with various ligament sizes. Additionally, Ag coated np-Au samples were synthesized by immersing np-Au in a 1 M Ag nitrate solution, followed by drying and thermal decomposition of the deposited Ag nitrate salt to Ag and NO2 and O2. Cross-sectional analysis revealed that this method yields a homogeneous Ag distribution, and that the Ag concentration can be adjusted within the range of 0-20 a.t.%. Mechanical testing was performed by depth-sensing nanoindentation. It was observed that the effect of the relative density on the elastic properties of np-Au seems to be much stronger than predicted by the Gibson and Ashby relationship. The elastic modulus of np-Au seems to be independent of the ligament size.

**4:40 PM**

**Effect of Partial Filling of Cells on Mechanical Strength of WBK Cores under Compression and Shear**: *Ki-Ju Kang*[1]; Jong-Sun Park[1]; [1]Chonnam National University

Among various fabrication methods for cellular metals, a technique based on 3 dimensional wire-weaving, known as Wire-woven Bulk Kagome (WBK), has taken attention because WBK can be useful for fabricating multilayered Kagome truss-type cellular metal. WBK has been proved that it has high strength-per-weight. Recently, a new idea for strengthening WBK even further was introduced, that is, filling tetrahedron-like cells in its interior space suppress the elastic buckling of struts which is a typical failure mechanism of truss type PCM (periodic cellular metals). In this work, first, theoretical studies using elementary mechanics of material are presented to estimate the strength under out-of-plane compression and shear. And the results are compared with those measured by experiments which were performed with specimens with various slenderness ratios of struts. It has been found that the partially filled WBK exhibited high specific stiffness and strength, absorption of energy in comparison with ordinary WBK.

**5:00 PM**

**Influence of Porosity and Microstructure on Thermal Properties of Laser Processed Ni and Ti6Al4V Alloy**: *Felix Espana*[1]; Vamsi Krishna Balla[1]; Susmita Bose[1]; Amit Bandyopadhyay[1]; [1]Washington State University

Thermal properties of metals and alloys depend on various parameters including composition, microstructure and surface topography. In this study we have fabricated Ni and Ti6Al4V alloy samples with varying porosity between 5 and 20 vol. % using Laser Engineered Net Shaping (LENS™) and tested their thermal properties and performance. It was found that porosity influences thermal performance, in terms of thermal conductivity, conductive and convective heat transfer. However, the finer microstructural features, formed as a result of rapid cooling rates associated with LENS™, prevented degradation of thermal conductivity of these materials. This microstructural influence on thermal properties has been confirmed by post-fabrication heat treatment studies. This presentation focuses on influence of porosity and microstructure of Ni and Ti6Al4V alloy on their thermal performance.

**5:20 PM**

**Mechanical Properties of LCS Porous Steel: Comparison between the Dissolution and Decomposition Routes**: *Miao Lu*[1]; Yuyuan Zhao[1]; [1]The University of Liverpool

Porous metals have many potential applications because of higher strength, stiffness and energy absorption capacity than polymer foams. This paper studied the porous steels produced by the lost carbonate sintering (LCS) process. The LCS porous steels had different mechanical properties when they were produced by the decomposition or dissolution route. The effects of the two routes on the mechanical behavior of the porous steels with different porosities (from 75% to 60%) and pore sizes (from 250μm to 1500μm) were compared. The compression strength and elastic modulus of the porous steel produced by the decomposition route were higher than those of the porous steel produced by the dissolution method. However, the differences became smaller at higher porosities and larger pore sizes. The study provided useful information for tailoring the mechanical properties of LCS porous steels.

## Alumina and Bauxite: Bayer Process Chemistry and Alumina Quality I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
*Program Organizers:* Carlos Suarez, Hatch Associates Inc; Everett Phillips, Nalco Company

Monday PM Room: 611
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Fred Williams, CMIS Corporation

**2:00 PM Introductory Comments**

**2:10 PM**

**Development of Particle Breakdown and Alumina Strength during Calcination**: *Benny Raahauge*[1]; Claus Jensen-Holm[1]; Susanne Wind[1]; [1]FLSmidth Denmark

Since the replacement of rotary kilns with stationary calciners, the impact from hydrate properties and calcination technology on the quality of Smelter Grade Alumina (SGA) have been studied frequently. F.L.Smidth are studying the complex interaction between calcining conditions, particle breakdown and development of alumina particle strength in both bench - scale and full scale calcination units using conventional analytical techniques. The bench – scale unit is simulating the pre-calcination step at 320 – 380°C, common to all stationary calciners and the final calcination stage at 1075°C in Gas Suspension Calciners without Holding Vessel. The calcining capacity of the full scale units are ranging from 2200 – 4500 tpd of SGA and covers stationary calciners with and without Holding Vessel. Representative hydrate samples are calcined and compared from more than eight (8) different alumina refineries. The first results of the above work will be presented with focus on how the calcining conditions impacts the properties of calcined industrial hydrates with respect to particle strength and particle breakdown.

**2:40 PM**

**Effect of Environmental Light on the Raman Spectrum of Sodium Aluminate Liquors**: *Jianguo Yin*[1]; Wangxing Li[1]; Zhonglin Yin[1]; Zhanwei Liu[1]; Zhaohui Su[1]; [1]Zhengzhou Research Institute Aluminum Corporation of China Limited

Raman spectroscopy is a useful instrument to character the structure of sodium aluminate liquors. As the spectrum of sodium aluminate liquors is not very strong, some environmental factors may affect the spectrum and hinder

us from attaining good results. Effect of environmental light on the Raman spectrum of sodium aluminate liquors was investigated. It is concluded that sunlight affects the spectrum of sodium aluminate liquors the most and almost masks it. For the light of fluorescence, it shows strong signal of light in the spectrum. But for the light of desk lamp and display screen, they have a little effect on the spectrum. To attain a good Raman spectrum of sodium aluminate liquors, the optimum operation condition is in a darkroom, or only let weak desk lamp on if necessary.

**3:10 PM Break**

**3:30 PM**

**Effect of $Na_2O$ on Alumina Leaching and Self-Disintegrating Property of Calcium Aluminate Slag**: *Sun Huilan*[1]; Wang Bo[1]; Bi Shiwen[2]; [1]Hebei University of Science and Technology; [2]Northearstern University

Effect of $Na_2O$ content on $Al_2O_3$ leaching rate and self-disintegrating rate of calcium aluminate slag are studied by adding $Na_2O$ into pure calcium aluminate slag. And the reaction mechanism is also discussed by XRD analysis initially. The results indicate that Lattice distortion and the weakness of bond energy which is asymmetry in Molecular are caused by the solid solution of $Na_2O$ into$12CaO•7Al_2O_3$. And this make $Na_2CO_3$ and $H_2O$ easy to penetrate into $12CaO•7Al_2O_3$ crystal, and improve the reaction speed and degree. So the existence of $Na_2O$ will improve the leaching property of calcium aluminate slag. But the self-disintegrating property of slag will be decreased because a great amount of ß-$2CaO•SiO_2$ is formed when the content of $Na_2O$ is higher than 2.5%. Considering from the property of calcium aluminate slag only, the largest allowable content of $Na_2O$ is 2.0~2.5% in slag and 1.2~1.5% in materials.

**4:00 PM**

**Improvement of Product Quality in Circulating Fluidized Bed Calcination**: *Cornelis Klett*[1]; Michael Missalla[1]; Roger Bligh[2]; [1]Outotec GmbH; [2]Outotec (Australasia) Pty. Ltd.

Until the introduction of Circulating Fluidized Bed (CFB) Calciners by Outotec (formerly Lurgi) in 1961 rotary kilns were the standard technology for the calcination of alumina. Since then, stationary calciners such as CFBs are the preferred technologies for new installations due to their superior energy efficiency despite their higher generation in fines. Over the last years Outotec researched ways to minimize particle breakage in its CFB calciners. Particle breakage is strongly dependent on the properties of the hydrate such as the hydrate shrinking behavior. Nevertheless other major influences like mechanical stress are a direct function of the calcining technology.Recent installed calciners and measurements from these have shown that Outotec has made a significant leap forward to reduce the particle breakage close to the level of rotary kilns whilst maintaining their enhanced operating performance and energy efficiency. The results of the recent achievements will be presented in this paper.

## Aluminum Alloys: Fabrication, Characterization and Applications: Numerical Modeling

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
*Program Organizers:* Subodh Das, Phinix LLC; Steven Long, Kaiser Aluminum Corporation; Tongguang Zhai, University of Kentucky

Monday PM Room: 615
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Zhengdong Long, Kaiser Aluminum

**2:00 PM**

**Constitutive Relations for Plastic Deformation in a 5754 Sheet**: *Lin Hu*[1]; Stephen Banovic[2]; Tim Foecke[2]; Mark Iadicola[2]; Anthony Rollett[1]; [1]Carnegie Mellon University; [2]National Institute of Standards and Technology

Constitutive equations for the multiaxial stress-strain behavior of aluminum alloy 5754 sheet have been developed, based on crystal plasticity. Both a Taylor-based polycrystal plasticity code (LApp) and a self-consistent viscoplastic code (VPSC) have been used to fit a single slip system hardening law to the available data for tension, plane strain and equibiaxial stretching. The fitting procedure yields good agreement with the monotonic stress-strain data. When the developed hardening law is used to model tests involving strain path changes, however, the agreement is less good. Furthermore, the simulated texture evolution is too rapid when compared to the experiments. These discrepancies motivate the further development of the constitutive relations to include such effects as grain-to-grain interactions and latent hardening.

**2:20 PM**

**Modeling Processing and Performance of an Al-Zn-Mg Alloy**: *John Chinella*[1]; [1]U.S. Army Research Laboratory

This study evaluates an Al-4.5Zn-1.2Mg-alloy 7020 armor. Microstructures of alloys 7039 and 7020 are shown. Computational thermodynamic modeling predicts: phase structures, equilibrium states, process temperatures, and manufacturing advantages. An artificial age study was conducted to modify the initial T651 condition to T7 tempers. The ageing study determines the hardness levels in response to holding periods of time-temperature. Aging parameters can help establish alloy 7020 processing factors to optimize and meet requirements for resistance to penetration by projectiles, or fragments, and fracture from stress corrosion cracking (SCC) or blast. The 7020-T651 ballistic test results versus armor piercing projectiles reveal protection levels superior to 5083-H131 aluminum and equivalent to Class 1, penetration-resistant rolled homogeneous armor (RHA). Results suggest a 7020-T7 material may be optimized to resist blast fracture and SCC, with levels of fragment protection that exceeds any RHA performance and penetration protection levels of shock-resistant RHA Class 2 armor.

**2:40 PM**

**Perturbed Bi-Particle Model of Deformation of Commercial Aluminum Alloys**: *Yansheng Liu*[1]; Xiyu Wen[2]; Ranall Bowers[1]; Xiaoxuan Li[1]; Shridas Ningileri[2]; [1]SECAT Inc; [2]University of Kentucky

Commercial aluminum alloys contain particles either as impurities or designated components. Bi-particle model is used in the current simulation by ABAQUS software. Perturbation is introduced in order to accommodate random distribution of particles. The model is applied to continuous cast ((CC) aluminum alloy under uniaxial tensile deformation. The behavior of particles and surrounding matrix during deformation is discussed. The result is helpful in understand the effect of particle distribution on the failure of CC aluminum alloys and in improvement of the mechanical properties of the materials. It is concluded that particles with larger effective cross section being perpendicular to tensile axial have more detrimental effect on elongation.

**3:00 PM**

**Modeling the Solidification under Pressure Casting Process for Aluminum Alloys**: *Edward Druschitz*[1]; Alan Druschitz[1]; Robin Foley[1]; [1]University of Alabama at Birmingham

Appling isostatic pressure during solidification has been shown to improve the mechanical properties of aluminum castings due to a reduction in the size of porosity. Existing equations appear to adequately describe the effect of pressure on gas pore size but do not describe the effect of pressure on dendrite arm spacing or cell size. In this paper, microstructural data (pore size, dendrite arm spacing, cell size) are compared for aluminum alloys solidified under 10 MPa pressure and at atmospheric pressure. The measured differences in microstructure are then compared to the relevant equations available in the literature. A model accounting for applied isostatic pressure during solidification is proposed.

**3:20 PM**

**An Integrated Computational Tool for Precipitation Simulation of Multi-Component Aluminum Alloys**: *Weisheng Cao*[1]; Kaisheng Wu[1]; Fan Zhang[1]; Shuanglin Chen[1]; Ying Yang[1]; Y. Austin Chang[2]; Jianzheng Guo[3]; Mark Samonds[3]; [1]CompuTherm LLC; [2]University of Wisconsin - Madison; [3]ESI Group

Modeling of microstructure and mechanical property during precipitation process plays a critical role in understanding the behavior of materials and thus accelerating the development cycles of materials. Nevertheless, an integrated computational tool coupling reliable thermodynamic calculation, kinetic simulation and property prediction of multi-component systems for industrial applications is rarely available. In these regards, a software package named as PanPrecipitation is being developed. It is seamlessly integrated with the thermodynamic calculation engine–PanEngine, which provides accurate thermodynamic properties and mobility data necessary for precipitation simulation. The generic system design together with multi-level kinetic and hardening models enable a range of applications. Its functionalities and advantages will be demonstrated by simulation of a number of aluminium

alloys. In addition, the integration of PanPrecipitation with ProCAST for pre-process or/and post-process simulation will be also discussed.

**3:40 PM Break**

**3:55 PM Invited**
***2010 LMD Young Leader Professional Development Award Winner*: Ultrasonic Welding of Aluminum Wires for Cables Harnesses in the Automotive Industry**: *Frank Balle*[1]; Guntram Wagner[1]; Dietmar Eifler[1]; [1]University of Kaiserslautern, Institute of Materials Science and Engineering

The cable harness of a modern upper class car, actually made of copper wires, has a weight of about 50 kg. The application of aluminum wires will be a promising method to reduce significantly the weight of cable harnesses in the range of about 40% with a simultaneous reduction of material costs. The ultrasonic metal welding process is already well established in the automotive industry to join copper wires. The challenge to substitute copper by aluminium is the affinity of aluminium to adhere at the ultrasonic welding tools during the solid state joining process. In this work suitable welding parameters were determined and special surface coatings for the ultrasonic welding tools were developed to ensure a reproducible manufacturing of joints between aluminum wires or between Al-wires and Al-connectors. Mechanical and electrical properties of ultrasonic welded Al-wires-joints as well as microstructural investigations of the joining zone by using computer tomography and electron microscopy will be discussed.

**4:15 PM**
**Phase-Field Simulations of Microstructure Formation in A356 during Casting**: *Markus Apel*[1]; Antoine Carre[1]; Bernd Böttger[1]; [1]Access e. V.

Today the phase field method emerges as a tool which can be applied to simulate the phase formation and solidification morphologies in technical alloys. In this presentation we present a combined study, sand casting experiments using a pure Al7%Si0.3%Mg alloy, accompanied by phase-field simulations of the microstructure formation during solidification. By calibrating the heat extraction rate, the simulations can be adjusted to yield approx. the same cooling curves as they are measured for the real castings.The simulated microstructures are in good agreement with the experimental ones. In addition, we will discuss the solidification path predicted by the phase-field simulation in comparison to Scheil-Gulliver type calculations. Further simulations take Fe and Cu impurities into account. The microsegregation pattern of Si, Mg and of the impurity elements will be discussed with respect to further heat treatments.

**4:35 PM**
**Modeling Non-Isothermal Annealing in Precipitate Hardening Aluminum Alloys: Microstructural Simulation**: *Panthea Sepehrband*[1]; Shahrzad Esmaeili[1]; Haiou Jin[2]; [1]University of Waterloo; [2]Novelis Global Technology Centre

A new approach is introduced for computational modeling of microstructural evolution during non-isothermal annealing in cold-rolled precipitate hardening aluminum alloys. Microstrcutural states are simulated using a Monte Carlo technique and on the basis of a competition between recovery and recrystallization for reduction of stored energy. The initial amount of stored energy, which is related to the level of deformation, is distributed inhomogeneously within the deformed grains, as dictated by the microstructural inhomogeneities and the grain structure. The effects of deformation-induced and pre-existing inhomogeneities, as well as precipitate coarsening and grain boundary pinning on the competitive recovery-recrystallization process are included in the algorithm of simulation. The method is implemented to predict the microstructural evolution during a non-isothermal annealing process that leads to fine-grained AA6xxx sheets. A good quantitative agreement is observed comparing the model predictions with the results from a comprehensive set of microstructural characterization experiments.

**4:55 PM**
**Prediction of Microstructure and Mechanical Properties in Aluminum Castings after Heat Treatment**: *Jianzheng Guo*[1]; Weisheng Cao[2]; Sam Scott[3]; Tony Kronenberger[4]; Joe Hirvela[4]; [1]ESI US R&D; [2]CompuTherm LLC; [3]ESI Group NA; [4]CPP-Minneapolis

A comprehensive numerical model is being developed for the calculation of the final microstructure and mechanical properties of aluminum casting alloys after heat treatment. After specifying the alloy chemical composition, solidification process, and heat treatment parameters, the model predicts the microstructure and potential defects through various stages of the component lifecycle: casting and heat treatment. The model takes into account the relationship between the different input parameters and the link to basic metallurgical features. Such a model can be used for tailoring mechanical properties and component performance with the correct choice of chemical composition and manufacturing process parameters. The effects of cooling history during casting and heat treatment processes are numerically and experimentally investigated. The microstructure and mechanical properties are predicted and compared with experimental measurements.

**5:15 PM**
**Study of a Geometrically Necessary Dislocations Field near the Interface of a Deformed Aluminum Bicrystal**: *Alankar Alankar*[1]; Ioannis Mastorakos[1]; David Field[1]; [1]Washington State University

We reiterate the study by Sun et al. (2000) on a lattice curvature field near the interface of a deformed aluminum bicrystal using our crystal plasticity finite element method (CPFEM) model. The CPFEM model shows evolution of statistically and geometrically necessary dislocations (GND) on all the octahedral slip systems in FCC material. The evolution of statistical dislocations is modeled in form of loops comprising pure edge and pure screw dislocations. Geometric character of dislocations is modeled using divergence of the flux of dislocations. In the simulation, a bicrystal with specific crystallite orientations is deformed in a channel die compression with thickness reduction perpendicular to the common interface. The model results show that due to lattice incompatibility at the interface, GNDs are created with high field density near the interface which tends to diminish away from the interface. The results are compared with the work presented in the above reference.

## Aluminum Reduction Technology: Aluminium Smelter: Environment, Health and Safety

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee
*Program Organizers:* Charles Mark Read, Bechtel Corporation; Gilles Dufour, Aluminerie de Deschambault

Monday PM Room: 608
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Bob McCulloch, Bechtel Corporation; Robert Baxter, Bechtel Corp

**2:00 PM Introductory Comments**

**2:05 PM**
**Modern Potline Gas Treatment Technology for High Amperage Pots - The Alcoa Fjardaal Experience**: Alain Moras[1]; Neal Dando[1]; *Bernard Cloutier*[2]; Philippe Dumortier[2]; Hugues Vendette[2]; [1]Alcoa; [2]Solios Environnement Inc.

Alcoa's latest greenfield smelter at Fjardaal, Iceland has a fluoride emission permit of 0.35 kg F/T Al which is about half the future (2010) European standard of 0.6 kg F/T. To facilitate meeting this requirement, the potline gas treatment centers (GTCs) were designed with dual pot suction systems to double pot extraction flow rate during pot work periods with balancing outlet dampers and venturi flowmeters and an alumina feed calibration system. Both GTCs have been in full operation since Q1 2008 with the following performance: GTC HF emission in the range of 0.2 -0.3 mg per Nm3 at an alumina wt% fluoride loading ~1.7%. No bags have been changed (23000 bags in total for the 2 GTCs), indicating there are no zones of high velocity within the filter casings. Additional design and control features will be presented and performance data will be discussed.

**2:30 PM**
**Heat Recovery from the Exhaust Gas of Aluminum Reduction Cells**: *Martin Fleer*[1]; Odd-Arne Lorentsen[2]; William Harvey[3]; Halldor Palsson[4]; Gudrun Saevarsdottir[3]; [1]Reyst, Reykjavik Energy School of Sustainable Systems; [2]Norsk Hydro; [3]Reykjavik University; [4]University of Iceland

Waste heat losses from aluminum smelting processes are around half of the total energy input and have the potential to be harnessed for useful purposes. The heat loss is by several paths of which the exhaust gas carries the second largest energy amount and is the most accessible. An experimental analysis of

the exhaust gas was conducted at the 270,000 tpy Nordural aluminum smelter in Iceland. The district heating potential of heat recovery from the exhaust gas was assessed for at the local community of Akranes. For this smelter the potential is up to 60 MWt. The chemical composition and dew point of the exhaust gas was analyzed. Particulates were isokinetically sampled and analyzed for chemical properties and size distribution. Scale buildup on a fouling probe was similarly examined. Data and commentary are presented that illustrate the difficulties and considerations for waste heat recovery from exhaust gas streams

**2:55 PM**

**Increased Energy Efficiency and Reduced HF Emissions with New Heat Exchanger**: *Anders Sorhuus*[1]; Geir Wedde[1]; Ketil Rye[2]; Gaute Nyland[2]; [1]Alstom; [2]Alcoa Mosjøen

The first full scale heat exchanger has now successfully demonstrated several months of stable operation at a smelter in Norway. Potentially 100 GWh per year can be recovered from this smelter alone for use in the district heating system. In hot climates recovered energy may be used to produce fresh water in desalination plants which may be equally beneficial. A sharp rise in fluoride emissions (HF) is seen as pot gas temperatures exceed 100ºC. Dilution of the pot gas with ambient air is used to achieve acceptable GTC gas temperatures (110-115ºC) and emission levels. In these cases installing the heat exchanger will not only reduce the size of the GTCs, but also large savings in the power consumption and reductions in HF emissions are obvious. On top of this the value of the recovered energy can justify the heat exchanger installation by itself.

**3:20 PM**

**Reduction Line-5 DC Electrical Hazard**: *Mohsen Shukralla*[1]; [1]Aluminium Bahrain (Alba)

Two of Reduction line-5 employees got slight electrical sensation while doing their routine jobs on the pots; the first one was on April while the second was on May 2009. Despite the fact that no body was injured in both accidents, the issue of electrical shock was taken very seriously. In both cases, the measurements showed the operating floor, which was supposed to be on Floating potential, is on Earth potential. After extensive measurements, checks and taken corrective actions, the hazardous areas were contained in a zone of 3 slabs which accounts for 9 pots. Danger zone notification was raised on the affected areas and all the safety precautionary measures were taken to safe guard all the employees. This paper shall describe the problem of electrical Dc hazard in pot line-5 and actions taken to resolve it. The detailed shock test measurements along with safety precautions taken are also discussed.

**3:45 PM Break**

**3:55 PM**

**2008 Global Anode Effect Survey Results**: *Jerry Marks*[1]; Chris Bayliss[2]; [1]J. Marks & Associates; [2]International Aluminium Institute

The International Aluminium Institute (IAI) conducts annual surveys of global primary aluminum producers on various performance parameters. Anode effect performance data has been collected annually since 2000. Prior to 2000 periodic surveys were conducted covering anode effect performance back to 1990. The current paper discusses progress toward a new 2020 PFC reduction objective. In addition other primary aluminum production survey statistics will be discussed on parameters such as energy consumption, fluoride emissions and safety performance.

**4:20 PM**

**The Applicability of Carbon Capture and Sequestration in Primary Aluminium Smelters**: *Stephan Broek*[1]; Sanjiv Save[1]; [1]Hatch Ltd

Climate Change is affecting every industry including the Primary Aluminium industry. The International Energy Agency has developed several strategies how to abate GHG emissions on large industrial scale. One of the tools identified to abate $CO_2$ emissions from large sources is carbon capture and sequestration (CCS). Earlier papers have touched on the topic of using CCS in primary smelters and in this paper further details are shown of technological aspects of CCS, but also on some of the specific conditions that have to be dealt with when applied in primary aluminium smelters. Conclusions are presented.

**4:45 PM**

**Application of a Method for the Determination of PFC Emissions during Aluminum Pot Startup**: *Jean-Nicolas Maltais*[1]; Josette Ross[1]; Alain Marcoux[1]; [1]Rio Tinto Alcan

Because of their global warming effect, PFC emissions are a major focus for the aluminum industry. The significant reductions achieved in the main source of PFCs, anode effects, had led to questions about the quantification of PFC emissions beyond anode effects. Rio Tinto Alcan has developed a precise industrial method for analysing PFC emissions. It uses active sampling through sorbent material and analysis by a system which includes thermal desorption, gas chromatography and mass spectroscopy. Measurement campaigns have been carried out on P155 and AP30 pots, in partnership with Environment Canada, to define the rate and quantity of PFC emissions during the startup high voltage period.This paper compares results with those from anode effects in the same smelters, and with pot start up measurements from other smelters. The wide variability of measured emission slopes highlights that pot startup practices have an impact on the PFC emissions rate.

**5:10 PM**

**Aluminum Fluoride – A Users Guide**: *Stephen Lindsay*[1]; [1]Alcoa, Inc.

Aluminum fluoride is an important raw material that is primarily manufactured via reactions of aluminum hydrate with fluorspar or with fluosilicic acid. Impurities generally have a minor impact upon smelting processes and metal products, but there can be exceptions. Likewise there are some physical properties of concern with this raw material. In this paper the author illustrates the important factors to consider when selecting and using sources of AlF3.

**5:35 PM**

**Dissolution Behavior of Aluminum Dross in Aluminum Electrolyte**: Weiqin Fu[1]; Zhaowen Wang[1]; Youjian Yang[1]; *Xianwei Hu*[1]; [1]Northeastern University

Aluminum dross generated during electrolytic refining process of aluminum could be added into aluminum electrolysis cells for recycling because of its high aluminum contents. In this paper, the constituents and compositions of the aluminum dross were determined by X-ray and chemical analyses and AlN and Al2O3 were proved to be the main constituents. On that basis, the phenomena of dissolution process of AlN and the aluminum dross in cryolite-based salt melts were observed through a transparent quartz cell. It was found that AlN was dispersed into the melts as small flocculent aggregate, and then dissolved into the melts gradually. The solubility of AlN was estimated to 2.0-2.5wt% at 1258 K. The aluminum dross was observed to form agglomerates on the bath surface, and then dissolved into the melts by chemical reaction with the adjacent electrolyte.Keywords: aluminum dross; dissolution behavior; solubility; transparent cell

**5:55 PM Concluding Comments**

## Aluminum Reduction Technology: Aluminium Smelter: Equipment

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee, TMS: Aluminum Processing Committee

*Program Organizers:* Charles Mark Read, Bechtel Corporation; Gilles Dufour, Aluminerie de Deschambault

Monday PM Room: 609
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Kevin Watson, Bechtel Corporation; John (Jack) Judson, Bechtel Corporation

**2:00 PM Introductory Comments**

**2:05 PM**

**Improving Heat Dissipation and Cell Life of Aged Reduction Lines at Aluminium Bahrain (Alba)**: *Abdulla Ahmed*[1]; K.S.R. Raghavendra[1]; Barry Welch[2]; [1]Aluminium Bahrain (Alba); [2]University of New South Wales (UNSW) & Welbank, Consulting Limited

The side worked end-on-end pre-bake anode technologies installed at beginning of 1970's gave pot life of 1600 days. During the early nineties, the potlines were retrofitted from side-break to point-fed, which was accompanied

by an increase of 8% in line current. The retrofits included gas collection system, changing anode setting pattern and installation of Alumina and Aluminium-fluoride feeders. However, pot life reduced to 1350 days due to increase of heat input by 5%. After introducing certain remedies, the pot life increased to above 2000 days. As the cells were still not operating at their full potential, a heat balance study was conducted in 2005. Several modifications were introduced in collector bar design, use of Silicon Carbide and Carbon composite inserts. Following this development a study has been made on the potential of applying the well-established principles of air-cooling in selective location around the pot shell to improve sidewall ledge protection.

**2:30 PM**

**Update on the Evaluation of HF Emission Reduction Using Covered Anode Trays**: *Jean-Pierre Gagne*[1]; Rene Minville[1]; Neal R Dando[2]; Gilles Dufour[3]; Mike Gershenzon[2]; Pierre Champoux[3]; Alain Moras[3]; [1]STAS; [2]Alcoa Technical Center; [3]Alcoa Canada

During the production of aluminum from conventional prebake Hall-Héroult electrolysis, carbon anodes have to be replaced on a regular basis. The anode butts are usually placed on uncovered trays for transportation and cooling, a practice that contributes to overall hydrogen fluoride (HF) emission from the smelter. In 2000, special anode tray covers developed by Alcoa Deschambault were implemented to significantly reduce fluoride emissions. In 2004, the Alcoa-STAS R&D team developed a second generation of anode tray covers for the new Alcoa Fjardaál plant. In 2009, the Alcoa STAS R&D Team designed and fabricated an experimental setup which allows the accurate full-scale measurement of temporal HF emissions from cooling anode trays. This paper presents a description of the setup, a summary of the procedure and an update of the results obtained with the use of the anode tray covers.

**2:55 PM**

**Automated Anode Gauging**: Said Al Maqbali[1]; C. Smith[1]; J. Raman[1]; M. Angirash[1]; S. Abdullah[1]; S. Thirunavukkarasu[1]; R. Kulkarni[1]; P. Marchand[2]; S. David[2]; P. Boucher[2]; [1]Sohar Aluminium Company; [2]ECL™

Anode change is one of the most critical operations of the reduction process. A continuous improvement project was conducted by Sohar Aluminum in collaboration with ECL to evaluate and optimize the laser based auto gauging system developed by ECL on Pot Tending Mmachines(PTM). This has eliminated risks to the operator from the hot butts being moved by the PTM as well as exposure to the radiant heat of the open pot. It also reduced the time taken for changing the anodes by 25 %. From a process point of view, it reduced improper anode settings by 40%. The process capability (based on mV reading 24 hours after the anode change) increased from a Cpk of 0.47 with manual gauging to 0.62 with laser auto gauging, resulting in better current distribution in the anodes and hence, better smelting efficiency.

**3:20 PM**

**Keeping the Pace of Continuous Improvement by Retrofitting Pot Tending Machines**: *José Barry*[1]; Fidias Roriguez[1]; Jesus Imery[1]; [1]CVG Venalum

Pot tending machines play a key role in the productivity of smelters, and as aluminium companies are squeezing any aspect of their processes to improve profits, in many cases old pot tending machines can not cope with new requirements. Hence, companies have to evaluate either retrofitting or installing new pot tending machines to carry on with their global improvement strategies. CVG-Venalum tackled this issue by developing its own pot tending technology; some of the improvements implemented are an enhanced design of the tool trolley, an automated anode height measurement, PLC and VFD based control system, electronically controlled clamp-tightening torque, and weight measurement modules integrated to the new control and supervisory system. The low cost of the two prototypes constructed at local workshops, as well as their enhanced performance, boosted this project, so CVG-Venalum management decided to carry on the retrofitting of the twenty original pot tending machines serving 720 pots

**3:45 PM Break**

**3:55 PM**

**New Concepts for Bulk Materials Plants for the Aluminium Producing Industry – From Raw Material Receiving to Electrolysis Cells**: *Stefan Skirde*[1]; [1]Coperion GmbH

Today's new smelters are often built at remote places with ever increasing plant capacity. The various plant layouts require different solutions for an economic bulk material handling system from ship to cell for a continuous production. A very common way for the receipt of raw material delivered by ships such as alumina and petrol coke is with a vacuum ship unloader. The product is conveyed at high capacities with a pipe conveyor directly to a storage silo, which can hold at least one complete shipload. Alumina quality will be maintained with an Anti-Segregation system. Further transports can be of pneumatic or mechanical nature depending on its best suitability.The electrolysis cell is the core element of the smelter where the raw material is transformed to valuable metal and requires an automatic and reliable feed of alumina in a dense mode.

**4:20 PM**

**Alfeed, a New Alumina Feeding System to Aluminium Pots**: *Sivert Ose*[1]; Anders Sorhuus[1]; Odd Bjarno[1]; Geir Wedde[1]; [1]Alstom

A new alumina distribution and feed system to the pots, Alfeed, has been developed and is now installed at one of the largest aluminium smelters in the world. At this smelter alone, more than one million ton of alumina per year will be distributed in more than 15 km of transport airslides. The Alfeed system contains several new features which are improvements above existing systems: • Slim design - making retrofit easy • Completely enclosed and self regulating system • Keep pot hoppers automatically topped up all the time • Recycles fines back to the pots • Transports coarse material to the pot • Minimal attrition, segregation or scaling of the material. The Alfeed has been developed during several years. With the successful delivery to one of the world largest smelters, Alfeed has now evolved into a commercial product using standard integrated design and robotic production methods.

**4:45 PM**

**Electrolysis Pot J Hooks New Design: Positive Impacts on Performance and Environment**: Nicolas Dupas[1]; *Damien Rose*[1]; [1]ECL

J hooks are crucial elements of modern aluminium smelting. Their task is to support and ensure the anode rods clamping by the anode clamps, and guide the pot tending machines extraction tool during the anode changing phase. Although they appear as simple components, their performances are critical to a successful production process. For 40 years, their design has almost remained unchanged, their simplicity and criticality making measurable improvements difficult to obtain. To create the new design, finite elements studies and computer assisted production methods have been used. The new concept's validation and its fine tuning has been performed on specially designed test benches, taking into account normal and abnormal conditions of use, and also during extensive field test with collaborating smelters.By working on the structural design, materials and shape of the J hooks, it has been possible to improve their performances and reduce their overall impact on the environment.

**5:10 PM**

**Cleaning and Maintenance of Crucibles and Siphons/Tubes**: *Dominique Prive*[1]; Pascal Côté[1]; Robin Boulianne[1]; [1]STAS

To optimise metal transportation from the potroom to the cast house, crucibles and siphons/tubes must be regularly cleaned and maintained to remove metal/bath deposits that accumulate inside, reducing the crucible capacity after several operations. Cleaning after cooling to room temperature is quite common but has time and cost implications. Moreover, heating cycles and cleaning operations in crucibles with improper equipment may damage the refractory. It is now possible to clean hot crucibles within a very short period of time, thus minimising the time when they are out of service. At the same time, siphon/tube cleaning and preheating are carried out and usually conducted in a common location with the crucible cleaning operation in the smelter. All those operations are automated and require minimum activities from an operator.This paper presents the latest available technologies in a fully integrated solution for the cleaning and maintenance of crucibles and siphons/tubes.

**5:35 PM**

**Erosion of Ferrous Alloys by Liquid Aluminum**: *Mandeep Sidhu*[1]; Milo Kral[1]; [1]University of Canterbury

Cast iron and/or cast steel pipes, which are commonly used to transfer molten aluminum, degrade by material loss and cracking. This is a problem for the aluminum production industry because of the cost of replacing pipes. Service conditions are severe, not only because liquid aluminum can react with nearly all metals and their oxides but also because of cycles of steep thermal gradients. The scope of this research was therefore to investigate the degradation of ferrous alloys under the demands of liquid metal transfer conditions. Pipe alloys

currently employed in service were selected as benchmarks for comparison with candidate alloys. Two specialized experiments were developed to expose candidate materials to erosive and thermal conditions similar to actual service. The various samples of ferrous alloys were compared by their erosive weight loss (by exposure to flowing aluminum) and response to thermal cycling (by cyclic exposure to static aluminum). Electron microscopy was used to characterize the reaction interfaces and microstructural changes to shed light upon the erosion and thermal fatigue mechanisms. The main outcome of this work is to recommend the best candidate material for the service conditions.

**5:55 PM Concluding Comments**

## Biological Materials Science: Bio-inspired Materials Design and Processing II: Bioceramics and Biomineralization

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
*Program Organizers:* John Nychka, University of Alberta; Jamie Kruzic, Oregon State University; Mehmet Sarikaya, University of Washington; Amit Bandyopadhyay, Washington State University

Monday PM Room: 205
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Amit Bandyopadhyay, Washington State University; Mehmet Sarikaya, University of Washington

**2:00 PM Invited**
**Novel Sol-Gel Bioactive Glasses for Tissue Engineering**: John Mitchell[1]; [1]Oregon Health and Science University

The interaction of biological fluids with certain compositions of glass materials has been shown to promote the crystallization of new mineral phases on the glass surfaces. Novel glasses synthesized in our laboratory, by sol-gel fabrication methods, have been shown to promote such biomimetic deposition in as little as 4 hours after immersion. The resulting materials are biocompatible and bioactive, inducing favorable cell and tissue reactions in vivo. This presentation will provide an overview of these amorphous materials and present examples of our investigations into their potential use in a myriad of situations, including: the prevention of dental hypersensitivity; the prevention of bone loss and the promotion of new bone growth; the enhancement of dental restorative materials; and the delivery of therapeutic compounds in vivo.

**2:30 PM**
**Factors Affecting the Dissolution of Resorbable Bioactive Glasses**: Satadru Kashyap[1]; Hamidreza Pirayesh[1]; *John Nychka*[1]; Ding Li[2]; Fuqian Yang[2]; [1]University of Alberta; [2]University of Kentucky

Bioactive glasses used in bone replacement therapy can be made to be entirely resorbable (e.g., bioactive glass 45S5). Factors affecting dissolution are numerous: residual stress, composition, crystallinity, immersion medium, and surface morphology. Here we present results on effects of residual stress, crystallinity, and manufacturing method on the dissolution behaviour of glasses with fixed overall composition (i.e., 45S5).

**2:50 PM**
**Mechanical and Biological Characterization of Dense Nanocrystalline HA Consolidated by Field-Assisted Sintering**: *Tien Tran*[1]; James Shackelford[1]; Joanna Groza[1]; [1]University of California, Davis

As the main inorganic component of bone, hydroxyapatite (HA) should ideally be a premier candidate in the selection of biomaterials. When grain sizes are restrained to the nanoscale regime, protein adsorption and cell adhesion are enhanced, while strength, hardness, and wear resistance are improved. Unfortunately, low phase stability, poor sinterability, and a tendency towards exaggerated grain coarsening challenge full densification of nanocrystalline HA by conventional sintering techniques. The field-assisted sintering technique (FAST) is capable of heating rates up to 1000°C/min, minimizing the time powders are exposed to sub-densification temperatures when some coarsening may occur. Transparent nanocrystalline specimens greater than 99% dense have been sintered in less than 20 min. Simulated body fluid immersion tests were conducted to probe the effects of sintering-related dehydroxylation on surface apatite deposition. Fracture toughness values in the range of 1.4-1.9 MPa√m were measured by single-edge v-notch bend.

**3:10 PM Invited**
**Nanoscale Calcium Phosphates in Bone Implants and Drug Delivery**: *Susmita Bose*[1]; [1]Washington State University

Synthetic calcium phosphates (CaPs) have been used in orthopedics and dentistry because of their excellent biocompatibility and chemical similarity with bone. Most commonly used synthetic CaPs are bioactive hydroxyapatite (Hap, Ca10(PO4)6(OH)2) and bioresorbable tricalcium phosphate (TCP, Ca3(PO4)2). These nanoscale inorganic ceramics can be used as both bone implant as well as a drug delivery vehicle, especially for bisphosphonate based drugs, which is a common drug being used to treat osteoporosis. In our research we have shown that both nanoscale feature as well as addition of dopants can influence biological and mechanical properties of CaP based bone graft material. This invited presentation will discuss nanoscale CaP processing, mechanical, biological property characterization as well as their use in bisphosphonate based drug delivery.Keywords: calcium phosphate, bioceramics, bone graft, drug delivery, processing

**3:40 PM Break**

**3:50 PM Invited**
**Crab Shell Osteogenesis**: *Otto Wilson*[1]; [1]Catholic University of America

There is a kinship that exists among biologically derived hard tissues. This inter-relatedness is expressed via comparable mechanisms for organic matrix organization and biomineralization processes. The ability to use biogenic hard tissues such as coral and nacre in augmentation of damaged bone tissue further attests to the unique bonds which are shared by hard tissues in nature. Crab shell cuticle is another candidate material for enhancing the healing and remodeling of bone. Extensive work has been performed utilizing crab shell component chitin and its chemical derivative chitosan. However, whole crab shell poses some interesting possibilities for use in bone tissue engineering due to similarities with bone tissue. A comparison of the unique chemical and physical characteristics of bone and crab shell with a focus on the respective organic matrices and mineral phases will serve as the focus for this talk. (This work was supported by NSF grant DMR-0645675).

**4:20 PM**
**Unveiling the Formation Mechanism of Pseudo Single-Crystal Aragonite Platelets in Nacre**: *Xiaodong Li*[1]; Zaiwang Huang[1]; [1]University of South Carolina

We demonstrate direct evidence that a single-crystal-like aragonite platelet is essentially assembled with aragonite nanoparticles. The aragonite nanoparticles are readily oriented and assembled into pseudosingle-crystal aragonite platelets via screw dislocation and amorphous aggregation, which are two dominant mediating mechanisms between nanoparticles during biomineralization. These findings will advance our understanding of nacre's biomineralization process and provide additional design guidelines for developing biomimetic materials.

**4:40 PM**
**Osteoinductive Potential of Biphasic Calcium Phosphate Scaffolds with Multi-Scale Porosity**: *Amy Wagoner Johnson*[1]; [1]University of Illinois at Urbana-Champaign

Calcium phosphates are synthetic biomaterials that are used in a wide range of dental and orthopedic applications to replace or restore bone because of their excellent bioactivity and osteoconductivity. Here we evaluate the effect of the growth factor BMP-2 on multi-scale osteointegration for biphasic calcium phosphate scaffolds with multi-scale porosity. Results show osteoinductive effects for scaffolds with and without BMP-2, as evidenced by cell migration and bone formation in micropores less than 10um in size with average interconnection size of 2um. Scaffolds with BMP-2 had significantly more cells in pores at 3, 6, and 12, but not 24 weeks. There is no effect of BMP-2 on the volume of bone formed within macropores in this defect model. Data suggest that osteoinductivity is imparted to the scaffolds via in vivo modifications related to the microporosity. These mechanisms have yet to be resolved, but possible mechanisms will be disscused.

**5:00 PM**
**Growth of Nacre in Abalone**: *Maria Lopez*[1]; P.Y. Chen[1]; K. Chumbimuni-Torres[1]; J. Wang[1]; J. McKittrick[1]; M.A. Meyers[1]; [1]UCSD

The processes of aggregation of minerals to the growing surfaces in red abalone (Haliotis rufescens) are analyzed. Flat pearl implantation method is used to observe the transient phases of calcium carbonate and the steady-state growth of aragonite tiles. Calcium-Ion Selective Electrode is used to monitor the variation of concentration of Ca++ between the extrapallial layer of mantle and the shell. These results are correlated with the growth of tiled aragonite. The morphology of the organic interlayer is characterized by scanning electron microscopy and atomic force microscopy. Its response to mechanical stresses is evaluated by means of nanoindentation. The nanoscale holes in the organic interlayer are measured. These measurements enable a realistic modeling of the formation of the terraced cones that comprise the principal biomineralization mechanism in this gastropod. Research support: NSF Biomaterials Program (DMR 0510138).

**5:20 PM**
**Bioinspired Synthetic Laminates**: *Gustavo Hirata*[1]; Sandra Diaz[1]; Po-Yu Chen[2]; Marc Meyers[2]; Joanna McKittrick[2]; [1]Center for Nanoscience and Nanotechnology; [2]UC San Diego

Bioinspired by the abalone shell microstructure, we have fabricated inorganic/polymer multistacked layers by combining pulsed laser ablation and magnetron sputtering. Smooth thin polymer (PMMA) films were grown by laser ablation and alternatively inserted between inorganic layers deposited by DC or RF magnetron sputtering in order to fabricate multi-layers of several biocompatible materials: CaCO3 (aragonite), ZrO2 ZrN and TiO2. The inorganic films are composed of nanocrystalline or amorphous particles with different degrees of porosity as observed by TEM and AFM. High resolution TEM analysis at the inorganic/organic interface revealed well formed inorganic films which are separated by the polymeric layer (10-50 nm). The hardness values showed an increase for the inorganic film/polymer stacked system as compared with the single film. A more detailed analysis of the results together with AFM/nanoindentation measurements will be presented. This research is supported by ARO Grant W911F-08-1-0461 and NSF Grant DMR 0510138.

## Bulk Metallic Glasses VII: Structures and Mechanical Properties II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
*Program Organizers:* Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee

Monday PM Room: 213
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* A. Greer, University of Cambridge; Dan Miracle, Air Force Research Laboratory

**2:00 PM Keynote**
**Anisotropy in Metallic Glasses**: *A. Greer*[1]; [1]University of Cambridge

Metallic glasses when fully relaxed are expected to be isotropic. However, they can be anisotropic as a result of their production method (for example, melt-spun ribbons appear to be strongly anisotropic), or anisotropy can be induced by various treatments such as annealing under stress or in a magnetic field. Examples of anisotropy will be reviewed and its structural origins will be considered, linking with anelasticity and viscous flow. The potential anisotropy of metallic glasses is often ignored, and this can lead to misleading conclusions. On the other hand, anisotropy can be also be useful.

**2:30 PM**
**Condensed Bond Enthalpies in Metallic Elements, Alloys and Compounds**: *Dan Miracle*[1]; James Dahlman[2]; Amanda Dahlman[2]; Garth Wilks[3]; [1]Air Force Research Laboratory; [2]SOCHE; [3]General Dynamics, Inc.

Bond dissociation energy and bond enthalpy are widely used measures of the energy contained in a bond between atoms in molecular gases. We propose a new quantity, the condensed bond enthalpy, to specify the energy contained in a bond between elements in the condensed state. We develop an approach for determining the condensed bond enthalpy in metallic elements, alloys and compounds using readily available bulk thermodynamic quantities and crystallographic data. The usefulness of these values is demonstrated by application to a range of problems in physics and materials science.

**2:40 PM Invited**
**Investigation of Homogeneous and Inhomogeneous Plastic Flow in Metallic Glasses**: *Katharine Flores*[1]; Wendelin Wright[2]; Wolfgang Windl[1]; [1]The Ohio State University; [2]Santa Clara University

As metallic glasses mature and are implemented, understanding the relationship between their atomic structure and plastic deformation mechanisms is increasingly relevant for both manufacturing and reliability. We have examined the mechanical behavior of a Zr-based BMG at the micro- and sub-microscale and observed a tendency towards more stable shear band behavior and mixed homogeneous-inhomogeneous flow at the smallest length scales. Shear band stability shows a strong dependence on the relative stiffness of the specimen. Implications of such stiffness and length-scale dependant behavior for the design of micro-scale devices and BMG composites will be discussed. These observations are discussed in light of structural changes observed in molecular dynamics simulations of Zr-Cu binary glasses during deformation under different stress state conditions. In these simulations, we characterize local density fluctuations and the coordination of the surrounding atoms as a means of describing the structure at points of incipient failure.

**3:00 PM**
**Aging and Plastic Flow in Metallic Glasses: Monte Carlo Simulations Based on the Activation-Relaxation Technique**: *David Rodney*[1]; Christopher Schuh[2]; [1]Grenoble Institute of Technology; [2]Massachussetts Institute of Technology

The yield stress in disordered solids marks a transition between aging at low applied stresses and plastic flow at higher stresses. The transition involves thermally-activated processes that are difficult to account for at the atomic scale because they occur on long timescales. Here, we overcome this limitation through Monte Carlo simulations in which the elementary thermally-activated events are determined using the activation-relaxation technique. We show that aging increases the stability of the glass, both thermodynamically (the internal energy decreases) and dynamically (the aged glass is surrounded by higher energy barriers). By contrast, plastic flow brings the glass into a high internal energy state only marginally stable, being surrounded by a high density of low-energy barriers. The strong influence of plastic deformation on the glass state is also shown through a polarization of the microstructure, evidenced by an asymmetry of the distribution of thermally-activated plastic strains in glasses after shear deformation.

**3:10 PM Invited**
**Inhomogeneous to Homogeneous Transition in an Au-Based Metallic Glass during Microcompression at Elevated Temperatures**: Shuangxi Song[1]; *T.G. Nieh*[1]; J.C. Huang[2]; J.S.C Jang[3]; [1]The University of Tennessee; [2]National Sun Yet-sen University; [3]National Central University

Transition from inhomogeneous to homogeneous deformation was characterized in Au49Ag5.5Pd2.3Cu26.9Si16.3 metallic glass pillars under compression at 373.1 – 384.2 K or 0.93 – 0.96Tg. Localized shear bands were formed well before macroscopic yielding at low temperatures. High-temperature compression were conducted on these pillar samples near the glass transition temperature to investigate the homogeneous deformation behavior. In contrast to local shear at low temperatures, samples were observed to deform uniformly. The strength was observed to decrease with increasing temperature and decreasing strain rate. Plastic flow behavior can be described by a shear transition zone (STZ) model. The activation energy and the size of the basic flow unit were both deduced and compared favorably with the free volume theory.

**3:30 PM Break**

**3:40 PM Invited**
**Deformation and Fracture Behavior of Metallic Glassy Alloys and Glassy-Crystal Composites**: *Dmitri Louzguine*[1]; Alexei Vinogradov[2]; Alain Reza Yavari[3]; Guoqiang Xie[4]; Akihisa Inoue[1]; [1]WPI-AIMR, Advanced Institute for Materials Research, Tohoku University; [2]Department of Intelligent Materials Engineering, Faculty of Engineering; [3]Institut National Polytechnique de Grenoble; [4]Institute for Materials Research, Tohoku University

The present work demonstrates the deformation behaviour of Zr-Cu-Ni-Al bulk glassy alloys as well as Ni-Cu-Ti-Zr crystal-glassy composites. Fracture

of Zr60Cu16Ni14Al10 bulk glassy alloy is featured by nearly equal fraction areas of cleavage-type and vein-type relief. The observed pattern of alternating cleavage-like and vein-type patterns illustrates a result of dynamically self-organizing shear propagation at the final catastrophic stage. One can suppose that the presence of a small volume fraction of eutectic colonies imposing a heterogeneity into a glassy Zr-Ni-Cu-Al matrix may help to block the plastic slip events. We also present a Ni-Cu-Ti-Zr crystal-glassy composite material having a superior strength paired with a considerable ductility of 15%. The metastable crystalline phase promotes a strain-induced martensitic transformation leading to enhanced plasticity at room temperature. Underlying mechanisms of plastic deformation are discussed in terms of the interplay between dislocation slip in the crystalline phase and shear deformation in the glassy matrix.

**4:00 PM**

**Effects of Hydrogen on Structural and Mechanical Behavior of Zr-Based Bulk-Metallic Glasses**: *Chih-Pin Chuang*[1]; Wojciech Dmowski[1]; Yun Liu[2]; Terry Udovic[2]; Yang Ren[3]; Peter Liaw[1]; Jaihung Huang[4]; [1]University of Tennessee; [2]NIST Center for Neutron Research; [3]Advanced Photon Source, Argonne National Lab.; [4]National TsingHua University

We report the hydrogen-induced phase transformation of Zr-based bulk metallic glasses (BMGs) and their effect on mechanical behavior. The hardness of hydrogen-charged alloys was increased, but was accompanied by the significant reduction in the toughness and strength. The changes of the local atomic structures of amorphous alloys were investigated by the radial-distribution-function (RDF) analysis using high-energy X-ray scattering. The results showed only the increase of the Zr-Zr inter-atomic distance, rather than the expansion of the whole amorphous matrix. Inelastic neutron scattering was used to determine the hydrogen-occupation site in amorphous alloys. The hydrogen optical frequency exhibits a very broad band (~ 70meV FWHM) at around 142 meV, and only shows the consistency of hydrogen atoms being occupied at the tetrahedral-like site. The observation from the X-ray scattering and inelastic neutron scattering suggests the existence of a stable, amorphous hydride phase, which could be responsible for the embrittlement of Zr-based BMGs.

**4:10 PM Invited**

**Flow and Fracture Studies on Bulk Metallic Glasses**: *John Lewandowski*[1]; [1]Case Western Reserve University

The effects of changes in pressure on the flow stress have been determine in both tension and compression with pressures up to 9 GPa. The dependence of the flow stress on the pressure will be compared to the pressure dependence of the elastic constants in order to rationalize the behavior observed. In addition, work continues on determining the effects of changes in elastic constants on both the compressive plasticity and toughness. Results will be presented for a number of different metallic glass systems.

**4:30 PM**

**Structural Defects in Metallic Glass Structures as Shear Transformation Zones**: *Dan Miracle*[1]; Garth Wilks[2]; Amanda Dahlman[3]; [1]Air Force Research Laboratory; [2]General Dynamics, Inc.; [3]SOCHE

Beyond simple estimates of the size and general nature of shear transformation zones (STZs) responsible for the initiation of deformation in metallic glasses, specific details regarding the atomic structural characteristics, frequency and distribution of STZs are presently unavailable. The purpose of this paper is to explore structural defects within the efficient cluster packing (ECP) model as possible STZs. Two specific point defect pairs will be developed in detail. The size of the candidate defect pairs will be determined, the energy associated with a defect pair will be used to estimate the frequency with which these defects may occur, and the free volume associated with these defect pairs will be calculated. The characteristics of these defect pairs will be compared with other theoretical and experimental studies regarding expectations for STZs in metallic glasses.

**4:40 PM Invited**

**Structure of Ca-Mg-Zn Bulk Metallic Glasses**: *Oleg Senkov*[1]; Daniel Miracle[1]; Emma Barney[2]; [1]Air Force Research Laboratory; [2]ISIS, Rutherford Appleton Laboratory

Amorphous structure of ternary $Ca_{60}Mg_{10+X}Zn_{30-X}$ metallic glasses (X = 0, 5, 10, and 15 at.%) was studied using neutron scattering and Reverse Monte Carlo (RMC) techniques. The scattered neutron intensities were measured in the scattering vector range Q from 0.1 to 100 $Å^{-1}$ and the total structure factors, S(Q), and the radial distribution function, G(r), were determined for each of the tested specimens. The RMC method was used to model the amorphous structure of the metallic glasses, which has the best fit to the experimental S(Q) and G(r). The computed amorphous structures were then analyzed and the Mg and Zn – centered atomic clusters were identified as the main features of these structures. Atom pair distances and partial coordination numbers were determined as functions of the alloy compositions and the results were compared with recently proposed cluster packing models.

**5:00 PM**

**Shear Bands Evolution in Cold-Rolled Bulk Metallic Glasses**: Q.P. Cao[1]; J.W. Liu[1]; L.Y. Chen[1]; X.D. Wang[1]; *Jianzhong Jiang*[1]; [1]International Center for New-Structured Materials (ICNSM)

Two bulk metallic glasses have been rolled at room temperature with different thickness reduction. Shear band evolution has been monitored during cold-rolling process. The dependences of microstructure and tensile mechanical property on the strain and rolling directions have been investigated. It is revealed that deformation-induced transformation does not occur up to 92% in thickness reduction except for shear bands. Shear band formation in conjugated directions is achieved in the specimen rolled in two directions, while rolling in one direction induces shear band formation only in one single direction. The efficient intersection of shear bands in conjugated directions results in work-hardening behavior and obvious plastic strain in tensile tests, which is further confirmed by in-situ SEM observation. Based on the experimental results, it is deduced that normal-stress-modified maximum shear stress criterion rather than shear plane criterion can describe the conditions for the formation of shear bands in uniaxial tension.

**5:10 PM Invited**

**Sample Size Dependent Deformation in Amorphous Metals**: Dominik Tönnies[1]; *Cynthia Volkert*[1]; [1]University of Göttingen

Recent studies show that sample size can have a strong influence on the deformation behaviour of metallic glasses. Below a critical dimension on the order of several hundred nanometers, some metallic glasses change from deforming by shear band formation to deforming by apparent homogeneous flow. This transition will be discussed in detail for the case of nanoindentation and micro-compression testing of amorphous $Pd_{80}Si_{20}$. In this alloy, samples with diameters larger than 400 nm deform by shear band formation at stresses consistent with the behaviour of bulk specimens, whereas smaller samples undergo homogenous deformation and strain softening. The transition in deformation mode with sample size will be discussed in terms of the available energy for shear band formation and will consider results from measurements of shear band spacing, offset, and overlap in small volumes.

**5:30 PM**

**Understanding Microstructure-Induced Ductility in Porous Bulk Metallic Glasses via Molecular Dynamics Simulations**: *Yunfeng Shi*[1]; [1]Rensselaer Polytechnic Institute

Bulk metallic glasses (BMGs) have excellent strength, processibility and resistance to corrosion for load-bearing applications. However, most monolithic BMGs are intrinsically brittle in unconfined loading conditions. One promising method for achieving ductility is to introduce porous microstructures into the BMGs. The precise mechanistic details of the confinement effect of pores on shear band development are still poorly understood. Here we present a simulation study focusing on the early-stage plasticity of porous BMGs. We will investigate possible toughening mechanisms including proliferation of shear band initiation, blunting the sharp shear off-sets at the pore surfaces and restriction of the shear band propagation from pore confinement. The relevance of various mechanisms depends on the pore size, shape and spacing. This study will also shed lights on designing porous microstructure that optimize both the ductility and strength.

**5:40 PM**

**Viscous Flow and Superplastic Deformation Behavior of Pt-, Pd- and Au-Based Bulk Metallic Glasses**: Jinn Chu[1]; *Yen-Chen Chen*[1]; Jason Shian-Ching Jang[2]; Tsong-Ru Tsai[3]; Hidemi Kato[4]; [1]National Taiwan University of Science and Technology; [2]National Central University; [3]National Taiwan Ocean University; [4]Tohoku University

Viscous flow and superplastic deformation behaviors of bulk metallic glasses (BMGs) in the supercooled liquid region (SCLR) are interesting to study, because BMGs are readily flowed under an applied pressure. The relative displacement as a function of temperature is obtained with the thermomechanical analyzer

(TMA) for the $Pt_{40}Ni_{20}P_{40}$ BMG rod with various compressive stresses at the heating rate of 10K/min. The viscosity under different stresses is thus computed from TMA results. The superplastic deformation behavior of Pd- and Au-based BMGs are examined in the SCLR at various strain rates in air. In addition, we demonstrate that various micro patterns of BMGs can be imprinted from mold in air. The forming component is characterized by scanning electron microscope and its optical property is further evaluated. The results show the BMGs exhibit excellent flowability and have potential micro/nano-device application.

**5:50 PM**

**Indentation Size Effect of Bulk Metallic Glass: A New Observation**: *Jae-il Jang*[1]; Byung-Gil Yoo[1]; Jun-Hak Oh[1]; Yong-Jae Kim[1]; [1]Hanyang University

Since the popular models for indentation size effect (ISE) are mainly based on dislocation strengthening, one might imagine that the models do not hold valid for bulk metallic glasses (BMGs) which do not contain crystalline defects. Despite the expectation of the size-independent hardness or strength in BMGs due to the absence of the dislocation, some experiemtal indentation studies reported the possible ISE. Additional controversy over the size effect of BMGs arose in recent studies performing microcompression tests: While some suggested 'smaller is softer,' 'smaller is stronger' is reported by others. Also, very recent micro-pillar test showed that the BMG strength is size-independent. To shed light on this dispute, we systematically analyzed the ISE of BMG through nanoindentations with various three-sided pyramidal indenters having different indenter angles. The purpose of this presentation is to report our recent experimental results, which led us to somewhat surprising observation on the issue.

## Characterization of Minerals, Metals and Materials: Characterization of Iron and Steel II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee

*Program Organizers:* Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF; Jiann-Yang Hwang, Michigan Technological University

Monday PM Room: 307
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Jian Li, CANMET-MTL; Jaroslaw Drelich, Michigan Technological University

**2:00 PM Introductory Comments**

**2:05 PM**

**Mechanisms of Composite Agglomeration of Fluoric Iron Concentrates**: You-ming Hu[1]; *Qian Li*[1]; Guang-hui Li[1]; Yong-bin Yang[1]; Yuan-bo Zhang[1]; Tao Jiang[1]; [1]Central South University

It is well-known that fluoric iron ores are very difficult for sintering and pelletizing. The yield and quality of sinter are greatly improved when using the composite agglomeration process (CAP) assisted with heat airflow, enhancing MgO contents and so on. For traditional sinter of fluoric iron concentrate, there is lower viscosity of binding phase and higher fluidity of liquid phase, the sinter is formed with large thin-walled holes and strength of sinter is deteriorated consequently. The novel process forms composite agglomerate in which acid pellets are embedded in basic sinter. The pellets are solid phase bonding and there forms Fe2O3 and Fe3O4 interconnect crystal. For basic sinter, after adding MgO, the viscosity are increased and fluidity decreased, and there form complex calcium ferrites (SFCA) and magnesium ferrite, they are crystallized perfectly and the sinter microstructure is very well.

**2:30 PM**

**On Plastic Notch Effects in Quenched and Tempered Steels**: Pasquale Russo Spena[1]; *Donato Firrao*[1]; Paolo Matteis[1]; [1]Politecnico di Torino

In 1971, Firrao and Spretnak performed a large experimental campaign to assess the plastic stress concentration factor at fracture as a function of the elastic stress concentration factor and of the tempering temperature, by using 25.4 mm wide, 1.14 mm thick AISI 4340 steel sheet tensile specimens with variable tip radius central notches. The availability of finite element methods allows now to re-examine those results and overcome the simplifying assumptions that were originally used to evaluate notch stresses. The elastic stress concentration factors are obtained by three-dimensional solutions, which also evidence the gradual evolution from plane-stress to plane-strain that occurs by decreasing the notch radius while keeping the thickness constant. Moreover, both the actual stress state and the stress concentration factor in the notch immediately before the failure are evaluated by elastic-plastic solutions.Finally, the original conclusions on the notch sensitivity of the examined steel are re-assessed and re-interpreted.

**2:50 PM**

**Calculating Model Establishing and Application of Nitriding and Denitriding to 304 Stainless Steel in AOD**: *Chunfei Shen*[1]; Qiao-lei Shi[1]; Yang Li[2]; Zhou-hua Jiang[2]; [1]Baoshan Iron and Steel Co., LTD; [2]Northeastern University

Nitriding and denitriding behavior of 304 stainless steel melts was researched in AOD refining process. Nitrogen solubility behavior of nitriding was mainly controlled by interphase reaction, and nitrogen solubility behavior of denitriding was controlled by both liquid phase transferring and interphase reaction. And the calculation models of nitriding and denitriding were established. Meanwhile, the comparisons of measured values and model calculation of nitrogen contents were also done. The results of AOD nitriding model showed that 85% of these comparisons had absolute error less than 100×10-4 % after removal of outlying data points. The same level of absolute error was achieved by 80% of the comparisons in AOD denitriding model in reduction period and desulfurization period. After removal of outliers, 90% of the comparisons achieved the level after desulfurization period of AOD.

**3:15 PM**

**Cyclic Deformation Behavior of a Medium Carbon Steel in the VHCF Range**: *Dietmar Eifler*[1]; Michael Koster[1]; Guntram Wagner[1]; [1]University of Kaiserslautern

An ultrasonic testing system (UTS) for the fatigue assessment of steels in the very high cycle fatigue (VHCF) range was developed. The UTS allows to measure characteristic data to de-scribe the cyclic deformation behaviour of medium carbon steels at ultrasonic frequencies. Cyclic loading leads to a defined change of process parameters of the UTS like generator power, dissipated energy and specimen temperature, indicating that irreversible changes in the materials microstructure occur. The measured data, strongly depend on the microstructure of the material. Specimen failure can be predicted with the applied technique in an early state. The very sensitive measuring methods indicate irreversible microstructural changes by a significant rise of process parameters before final failure occurs. In SEM investigations subsurface fatigue cracks emanating from phase boundaries between ferrite and cementite were observed in the VHCF range. Koster Michael, Wagner Guntram, Eifler Dietmar

**3:40 PM**

**Methods to Characterize Very Thin Passive Film Formed in SCW Corrosion Tests**: *Jian Li*[1]; D. Guzonas[2]; Wenyue Zheng[1]; [1]CANMET-MTL; [2]Atomic Energy of Canada Limited

Developing the Generation IV nuclear reactor is an international collaborative effort. One of the most important Gen IV nuclear reactor designs is the supercritical water (SCW) concept, in which the water coolant runs at about 625oC at the outlet. Under this temperature and high pressure condition, the cooling water is at supercritical state, which could pose significant demand for in-core materials. High-temperature mechanical properties, corrosion resistance and radiation damage are some of the key challenges. At present, no single commercial material can meet all the requirements of the severe SCWR in-core conditions. Corrosion experiments performed under SCW condition yields various kinds of corrosion modes, some pose great challenge to characterize using conventional techniques. A combination of various advanced characterization techniques proofed to be necessary to understand the root cause of materials failure.

**4:05 PM**

**Mechanical Properties of Heat Treated HSLA Bolt Steels**: *Hamed Fathi Doost*[1]; Ali Nazari[1]; [1]Islamic Azad University (Saveh Branch)

Tensile properties, Fracture toughness and Charpy impact energy of four types of HSLA steels which are used in 10/9 and 12/9 grade bolts has been studied. All of the four groups has been annealed at 850 degree of centigrade, water- and oil-cooled and then tempered at 500, 550 and 600 degree of centigrade. The

best heat treating cycle has been optimized and metalographic studies has been conducted to investigate the created structures. Scanning electron microscope fractography also used to verify fracture mode. Some empirical relations have been proposed for prediction fracture toughness of HSLA steels using tensile properties and Charpy impact energy data.

**4:30 PM**

**Effect of Stacking Fault Probability and ε Martensite on Damping Capacity of Fe-16%Mn-2% Si Alloy**: *Girish Bm*[1]; Satish Bm[2]; K. Mahesh[2]; [1]MVJ College of Engineering ; [2]East Point College of Engineering and Technology

The Damping capacity of a Fe-16%Mn-1%Mg-2%Si (wt. %) alloy has been studied with respect to cold rolling and annealing after cold rolling. The damping capacity of 8% cold rolled specimen is much higher than 0% cold rolled specimen. In the 0% rolled specimen, the stacking faults are responsible for the damping capacity characterized by the large amplitude dependence. The improvement in damping capacity by cold rolling is attributed to the formation of e martensite. As the temperature increases, the damping capacity improves, reaching its maximum around 1000ºC. A further increase in the temperature, however, degrades the damping capacity. The area of the γ/ε boundaries is the major factor for damping capacity. Heat treatment at 1100ºC lessens the number of e martensite plates, leading to area reduction of γ/ε boundaries despite an increase in amount of e martensite.

**4:55 PM**

**Study of Pre-Strain Effect on Indentation Fracture Toughness of HSLA Steel Using Continuum Damage Mechanics**: *Sabita Ghosh*[1]; Mita Tarafder[1]; Goutam Das[1]; Soumitra Tarafder[1]; [1]National Metallurgical Laboratory

Since last three decades Ball Indentation Technique (BIT) has been used to evaluate mechanical properties like ultimate tensile test, yield strength, strain hardening exponent, strength coefficient and true stress-true strain curve of various engineering materials. BIT can be used when a tensile test cannot be performed: on welded joints or components under service. There has been an urge among the researchers to get an idea about the fracture toughness (KJC) value non-destructively. Various models have been developed to estimate KJC using indentation flow curve. The present work highlights the study about the effect of pre strain on indentation KJC of HSLA steel from the flow curve using continuum damage model. To establish the damage model for the evaluation of KJC under compression, attempt has been made to simulate the ball indentation experimentation to find stress triaxiality just beneath the indentater. ABAQUS software package has been used for finite element simulation.

**5:20 PM**

**The Influence of Different Heat Treatment Cycles on Controlled Surface Graphitization in CK45 Steel**: *Ali-Reza Kiani-Rashid*[1]; Y Hamedi[2]; H.R. Shishegar[2]; [1]Ferdowsi University of Mashhad; [2]Sharif University of Technology

Controlled graphitization has become known as a practical method for improvement of wear resistance and machining properties in steels. In this paper, the effect of heat treatment on microstructure of Ck45 steel has been investigated. Austenitising was carried out at 920°C for 5 hours. Besides, isothermal transformation was conducted at 750°C in the time range of 1-20 hours. The microstructure of the steel considerably changes by this heat treatment process which exhibits the effects of temperature, appropriate austenitising duration and isothermal transformation. Conducted experiments show a suitable distribution of semi-spherical graphite particles especially on the surface of the steel. Also, analyses demonstrate that the amount of formed graphite in the austenitising temperature 920°C is more than graphite in single heat treatment temperature of 750°C.

**5:45 PM Concluding Comments**

**5:50 PM Question and Answer Period**

## Coatings for Structural, Biological, and Electronic Applications: Applications of Coatings I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Biomaterials Committee, TMS: Thin Films and Interfaces Committee

*Program Organizers:* Nuggehalli Ravindra, New Jersey Institute of Technology; Gregory Krumdick, Argonne National Laboratory; Roger Narayan, Univ of North Carolina & North Carolina State Univ; Choong-un Kim, University of Texas at Arlington; Nancy Michael, University of Texas at Arlington

Monday PM Room: 309
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Nancy Michael, University of Texas

**2:00 PM Introductory Comments**

**2:10 PM Invited**

**A Load-based Depth-sensing Micro-Indentation Technique for Spallation Detection of Thermal Barrier Coatings**: *Bruce Kang*[1]; Jared Tannenbaum[1]; Mary Anne Alvin[2]; [1]West Virginia University; [2]National Energy Technology Lab

In this research, we present a micro-indentation technique for NDE spallation detection of thermal barrier coating (TBC) materials. Based on a multiple-partial unloading procedure during indentation, stiffness responses of TBC coupons subjected to thermal cyclic loadings were found to correlate well with morphology changes and increase of residual out-of-plane stress at the bond coat/TGO/top coat interfacial region. A TBC thermal exposure test plan was carried out where time-series cross-sectional microstructural analyses of damage accumulation and spallation failure associated with the evolution of the bond coat/TGO/top coat composite were evaluated and correlated with the measured stiffness responses at various thermal cycles. The results show that prior to the large-scale TBC spallation, the micro-indentation method is capable of identifying TBC spallation site(s). This technique can be viewed as a viable NDE technique for as-manufactured and process-exposed TBCs. A portable instrument for on-line spallation detection/prediction of industrial-size TBC turbine components is also developed.

**2:40 PM**

**Temperature and Scale Dependent Deformation and Creep Behavior of Polymer Derived Si-C-O Ceramics**: *Ming Gan*[1]; Vikas Tomar[1]; [1]Purdue University

Due to their high thermal resistance and outstanding mechanical strength under elevated temperatures, Si-C-O polymer derived ceramics (PDCs) have a wide application in thermal barrier coatings. Effect of temperature and length scale on the elastic modulus and creep strength a Si-C-O based PDC was investigated by nano and micro-scale tests from room temperature up to 750ºC. The results showed that elastic modulus of the material first decreases as a function of temperature up to 100ºC and then increases with further increase in temperature at both nano- and micro length scales. The modulus showed reduction by one tenth while switching from the nanoscale to the microscale. During nanoscale and microscale creep experiments, effects of peak load, loading rate, and holding time on the creep behavior were investigated. A strong size effect was observed at initiation stage of creep, which leads to higher stress exponents under all testing conditions.

**3:05 PM Break**

**3:20 PM Invited**

**Surface Modification of Nanostructured Materials for Functional Medical Devices**: *Roger Narayan*[1]; Nancy Monteiro-Riviere[2]; Robin Brigmon[3]; Michael Pellin[4]; Jeffrey Elam[4]; [1]University of North Carolina & North Carolina State University; [2]North Carolina State University; [3]Savannah River National Laboratory; [4]Argonne National Laboratory

Nanoscale materials may play a leading role in treatment of chronic disease (e.g., controlled release of chemotherapeutic agents for treatment of cancer). Nanoporous alumina provides several advantages over conventional polymeric materials for use in functional medical applications. In this study, atomic layer

deposition was used to coat all the surfaces of nanoporous alumina membranes in order to improve the biocompatibility and reduce the pore size in a controlled manner. In vitro studies revealed that titanium oxide-coated nanoporous alumina membranes did not exhibit statistically lower viability compared to the uncoated control materials. 20 nm pore size titanium oxide-coated nanoporous alumina membranes exposed to ultraviolet light exhibited activity against Escherichia coli and Staphylococcus aureus bacteria. Nanostructured materials prepared using atomic layer deposition may be used to delivering a pharmacologic agent at a precise rate within the body. These materials may also serve as the basis for orthopedic implants or self-sterilizing medical devices.

**3:50 PM**
**Preparation of the Biomimetic Calcium Phosphate Coating on CoCrMo Implant Alloys via an Effective Chemical Activation**: *Luning Wang*[1]; Jingli Luo[1]; [1]University of Alberta

An ion adsorption based chemical method was applied to generate calcium phosphate coatings on CoCrMo implants. The surface was fully covered by calcium phosphate nucleation crystallites after immersion in supersaturated calcium solution (SCS) for 1 h at room temperature, rinsing by water and completely dry in air. The treated specimen was readily for growing calcium phosphate coatings once it immersed in SCS at 37°X. A homogenous calcium phosphate coating formed on the surface after 2 hrs immersion. It shows that the coating mainly contains octacalcium phosphate phase with minor hydroxyapatite. The coating layer is about 4 μm thickness and with high bonding strength (26.2 ± 1.2 MPa). The in vitro electrochemical characterization also indicates that the coated specimen shows higher corrosion resistance and more noble corrosion potential than the uncoated CoCrMo implants. This method shows an efficient way to prepare a bioactive CoCrMo surface for medical application.

**4:15 PM Invited**
**Molecular-Scale Surface and Interfacial Coatings Utilizing Self-Assembled Monolayer of Phosphonates (SAMP) Technology**: *Eric Bruner*[1]; [1]Aculon, Inc.

Nanoscale thick Self-Assembled Monolayer of Phosphonates (SAMP) coatings can be classified into three functional areas; non-stick, pro-stick/adhesion, and anti-corrosion. Coating solutions and processes were engineered for numerous markets including optical, display, electronics, and industrial coatings. These coatings outperform all known alternatives in characteristics such as adhesion, stain resistance, and scratch resistance. SAMP treatments can be used for a variety of applications including imparting hydrophobicity, adhesion, or corrosion inhibition to numerous substrates. Aculon's proprietary methodology can impart any of these properties on metals, metal oxides and even some polymer surfaces by drawing on its library of structurally tailored phosphonic acids.The SAMP is one approximately 1.5 nm thick, and it completely covers the material to which it is applied, and assures total surface coverage regardless of the type or texture of that material. The composition of the SAMP determines the properties that it imparts to its substrate.

**4:45 PM**
**Piezoelectric Measurements and Microstructural Characterization of 'Smart' AlN Thin Films Fabricated by Pulsed Closed Field Unbalanced Magnetron Sputtering**: *Masood Hasheminiasari*[1]; J. Lin[1]; J.J. Moore[1]; B. Mishra[1]; [1]Colorado School of Mines

A 'Smart' die coating based on AlN piezoelectric thin film sensor embedded into a tribological coating system is been investigated in this research. Thin film sensor design and its overall incorporation within the coating system are explained briefly. Piezoelectric AlN thin film with highly preferred (002) orientation has been deposited by pulsed closed field unbalanced magnetron sputtering (P-CFUBMS) system using Cr as the electrode/adhesion layer. The effect of working pressure on residual stress and (002) orientation of AlN thin film is discussed together with gas ratio effect on the piezoelectric measurements. The piezo-responses of AlN thin films were characterized by a lock-in-amplifier, which measures the output piezo-voltage of AlN thin film under cyclic loading conditions using a micro-actuator.

**5:10 PM**
**Plasma Spray Coatings for Aerospace Applications**: *David Koch*[1]; D.M. Fell[1]; David Field[1]; [1]Washington State University

While plasma-spray coatings have been vital process for application of high technology, high perforamce coatings in aerospace engines, etc., application has been limited to most metals, and a few ceramics. Research to apply various oxides, borides, carbides, nitrites, and couple silicides powders as high temperature coatings appear to have positive results after the plasma system was modified with a reactive-atmospheric-processing (RAP) apparatus and techniques. Zirconium Carbide (highest known melting point material at 6400°F {3540°C}), Titanium Carbide, and Titanium Boride coatings were applied with Plasma-RAP variations, with each coating chemistry XRD confirmed and discussed. Further characterization to discuss includes SEM, EDX, porosity content, coating thickness; plus microhardness and abrasion/wear testing.

**5:35 PM**
**Using Artificial Neural Network to Optimize Thickness and Hardness of TiN Layers Deposited by PACVD Method**: Mohammad Sadegh Mahdipoor[1]; Farzad Mahboubi[1]; *Niloofar Kamkar Zahmatkesh*[1]; Mahdi Raoufi[1]; [1]Amir Kabir University

Recently, TiN coating deposited by different methods especially plasma-assisted chemical vapor deposition (PACVD) is used to protect tools from wear. The parameters of used methods for deposition of layers can be so effective on their final characteristics. The aim of this study was to investigate the effective parameters of PACVD method on the final thickness and hardness of TiN coatings and predicting these values in different situations using Artificial Neural Network (ANN). The deposition temperature, deposition time and duty cycle were investigated. The experimental data was used for training of ANN and a multilayer cascade forward back-propagation neural network was designed. The optimization was performed by minimizing the generalized interval between the predicted values and the optimized ones that were obtained experimentally. The predicted values obtained from the trained ANN are found to be in close agreement with the experimental results.

## Computational Thermodynamics and Kinetics: Grain Growth

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee
*Program Organizers:* Jeffrey Hoyt, McMaster University; Dallas Trinkle, Univeristy of Illinois at Urbana-Champaign

Monday PM Room: 308
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**2:00 PM Invited**
**Abnormal Grain Growth in the Presence of a Static Particle Dispersion**: *Elizabeth Holm*[1]; Todd Hoffmann[1]; Anthony Rollett[2]; Christopher Roberts[2]; [1]Sandia National Laboratories; [2]Carnegie Mellon University

Dispersions of second phase particles are often used to inhibit grain growth in polycrystalline metals, so it stands to reason that they might also prevent abnormal grain growth. We used mesoscale grain growth simulations to test this hypothesis for two cases: an initially unpinned polycrystal, and an initially pinned polycrystal. When we added inert, static second phase particles to an initially unpinned structure, abnormal grain growth occurred as if in the absence of particles; only when the abnormal grains impinged and began to grow normally did particle pinning occur. When we induced pinning by placing inert, static second phase particles along grain boundaries, abnormal grain growth occurred even in systems that did not grow abnormally in the absence of particles. We explain this particle-assisted abnormal grain growth in terms of boundary fluctuations and mobility advantage. These results have important implications for designing microstructures that are resistant to abnormal growth.

**2:30 PM Invited**
**Effect of Stresses on Grain Boundary Thermodynamics: Theory and Atomistic Simulations**: T. Frolov[1]; *Y. Mishin*[1]; [1]George Mason University

Thermodynamic properties of grain boundaries (GBs) are usually described in terms of the Gibbs absorption equation which was originally derived for fluid-fluid and solid-fluid interfaces. By contrast to such systems, GBs can support elastic shear stresses applied parallel to the GB plane. We derive a generalized

adsorption equation that includes additional terms representing the effect of the applied shear stresses and the excess strains conjugate to them. These new terms permit computation of the tensor of excess elastic compliances of GBs and can give rise to stress-strain, stress-segregation and other types of cross-effects. The new thermodynamic relations are applied to atomistic simulation of GBs in Cu and Cu-Ag alloys under applied tensile, compressive and shear stresses. We study the effect of such stresses on GB free energy, interface stress, solute segregation and other thermodynamic properties.

**3:00 PM**

**Controlling Crystal Structure in Phase Field Crystal Modeling**: *Michael Greenwood*[1]; Nikolas Provatas[2]; Joerg Rottler[1]; [1]University of British Columbia; [2]McMaster University

The phase field crystal (PFC) method has emerged as a promising candidate for modeling materials with atomistic resolution on mesoscopic time scales. With the currently available (phenomenological) free energy functionals, however, only lattices with triangular (2D) or bcc (3D) symmetries can be studied. We extend the free energy functionals through classical density functional theory to generate a broader class of crystalline structures in both 2D and 3D. Specifically, the correlation functions in these energies are constructed using from the desired lattice and basis structure and the probability densities of the atomic positions within these lattices. We apply this method to crystallization from the liquid state andstructural solid-solid transformations.

**3:20 PM Break**

**3:30 PM Invited**

**Molecular Dynamics Simulation of Grain Growth in 3D Nanograined Ni**: *Stephen Foiles*[1]; Elizabeth Holm[1]; [1]Sandia National Laboratories

Grain growth is one of the fundamental phenomena in materials processing. There is current interest in potential differences in grain growth in nanograined materials compared to conventional materials. Molecular dynamics simulations of the grain growth in a fully 3D nanocrystalline sample have been performed at a variety of temperatures and system sizes. It is observed that a high density of twin boundaries is formed during the growth process consistent with recent experimental findings. The grain growth kinetics are observed to obey the conventional parabolic time dependence until a temperature dependent stagnation is observed. The origin of this stagnation is discussed.Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC0494AL85000.

**4:00 PM Invited**

**Evolving Microstructures in Lipid Bilayers: Novel Insights from Materials Science**: *Mikko Haataja*[1]; [1]Princeton University

Lipid bilayer membranes are semi-permeable, two-dimensional fluid-like sheets which control the exchange of matter and information between living cells and their surroundings. Far from being a featureless mixture of lipids and proteins, compositional lipid microdomains within these membranes are believed to facilitate many important cellular processes. The physical mechanisms which control the sizes and lifetimes of these spatially-extended domains ("membrane microstructure") are rather poorly understood at the moment. In this talk I will argue that a combination of thermodynamics of immiscible systems (driving phase separation) and incorporation of active cellular lipid transport mechanisms to and from the membrane impeding phase separation (analogous to irradiation effects in immiscible binary alloys) provides a compelling physical picture of the spatio-temporal microstructure of the membrane. In more general terms, I will argue that tools from materials science can provide important and novel insight into complex "microstructural evolution" phenomena in biophysics.

**4:30 PM**

**Twinning Nucleation Mechanisms in Hexagonal-Close-Packed Crystals**: *Jian Wang*[1]; John Hirth[1]; Carlos Tome[1]; [1]LANL

We have studied mechanisms for (-1012) twinning in hexagonal-close-packed crystals at an atomic scale using topological analysis and atomistic simulations. Two twinning mechanisms were found: a normal-twinning mechanism in which a stable twin nucleus is created by simultaneous nucleation of multiple twinning dislocations, and a zonal-twinning mechanism in which a stable twin nucleus is created by simultaneous nucleation of a partial dislocation and multiple twinning dislocations. Atomistic simulations, using density-function-theory for Mg, Zr and Zn and an empirical potential for Mg, were performed to study the kinetics and energetics associated with the two twinning mechanisms. The results show that the zonal-twinning mechanism is energetically favorable relative to the normal-twinning mechanism because the zonal dislocation has a smaller Burgers vector.

**4:50 PM**

**Phase Field Modelling of Austenite Grain Growth in the Heat Affected Zone**: *Morteza Toloui*[1]; Matthias Militzer[1]; [1]UBC

Austenite grain growth in the heat affected zone (HAZ) of girth welds in a microalloyed linepipe steel is simulated using phase field modelling. The steep temperature gradient in the HAZ leads to thermal pinning that can adequately be replicated with the phase field approach. An effective grain boundary mobility has been determined from experimental grain growth studies during rapid continuous heating tests thereby accounting for pinning of TiN and NbCN as well as solute drag when Nb particles dissolve. Using the effective mobility austenite grain growth has been predicted in the HAZ employing typical time-temperature profiles and the resulting thermal gradients as a function of the position from the fusion line. Predicted grain structures have been compared with the grain structures observed in the HAZ.

**5:10 PM**

**Affinities for Grain Contacts in 3D Grain Growth**: *Burton Patterson*[1]; Alan Sprague[1]; Veena Tikare[2]; Cristina Cardona[3]; Daniel Chappell[1]; Robert T. DeHoff[4]; [1]University of Alabama at Birmingham; [2]Sandia National Laboratories, New Mexico; [3]San Diego State University; [4]University of Florida

The tendencies for preferred contact among grains of different numbers of faces is quantified using an affinity term describing the actual frequency of occurrence of grain pairs relative to the frequency expected from random contact. Thus, the affinity term describes these tendencies as multiplicative factors of occurrence greater or less than the random state. Data from 3D Monte Carlo simulations shows preferred contact between grains with high and low numbers of faces, with affinities as high as 8 times random. Mid-class grains of approximately 14 faces show merely random contact, i.e., no preference or avoidance, with grains of higher and lower numbers of faces. Affinities also have been determined for different topological characteristics on separated grains. These affinity characteristics provide insight into the topological mechanism of grain growth.

**5:30 PM**

**Atomistic Comparison of Volume-Dependent Melt Properties from Four Models of Aluminum**: *Chandler Becker*[1]; Matthew Kramer[2]; [1]Materials Science and Engineering Laboratory, National Institute of Standards and Technology; [2]Materials and Engineering Physics, Ames Laboratory

With the increasing use of simulations in materials research and design, it is important to quantify differences between, and accuracy of, models used in these simulations. Here we present the results of such a comparison for four embedded-atom models of aluminum that were optimized to have good liquid properties, particularly the melting temperatures. The effects of temperature and volume are systematically examined in the melts for bulk thermodynamic quantities, pair correlation functions and structure factors, and diffusion coefficients for each interatomic potential. Where possible, these are then compared with experimental values. We find quantitative differences in the properties determined from the various interatomic potentials despite the fact that they were fit with similar sets of data.

## Cost-Affordable Titanium III: Low Cost Materials and Processing

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
*Program Organizers:* M. Ashraf Imam, Naval Research Lab; F. H. (Sam) Froes, University of Idaho; Kevin Dring, Norsk Titanium

Monday PM Room: 618
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* F. H.(Sam) Froes, University of Idaho; M. Ashraf Imam, Naval Research Laboratory



**2:00 PM**

**Applications of the FFC Cambridge Process**: *Richard Dashwood*[1]; Rohit Bhagat[1]; Ben Jackson[2]; Randhir Singh[2]; Peter Lee[2]; Douglas Inman[2]; David Dye[2]; Martin Jackson[3]; [1]The University of Warwick; [2]Imperial College London; [3]The University of Sheffield

The FFC Cambridge is a potential alternative to the Kroll process and, when a mixed oxide precursor is used, is a low-cost method for producing alloy powder. This paper will demonstrate the relative ease with which novel and challenging alloy compositions such as Ti-Mo, Ti-W, and Ni-Ti, or complex geometries, such as titanium foams, can be produced. Highly alloyed compositions (i.e. 50 wt.%Mo) can be produced with homogenous chemistry, refined microstructure and low oxygen content (as low as 600 ppm in equiatomic Ni-Ti). Another benefit of the process derives from the nature of the oxygen removal in that the geometry of the cathode precursor is unaltered through to the metal product, albeit with some shrinkage. By combining a novel precursor manufacturing route with the FFC Cambridge process, open cell titanium foams have been produced. The foams possess good mechanical properties and high permeability, making them especially attractive for biomedical applications.

**2:25 PM**

**Very Low Cost Manufacturing of Titanium Alloy Components**: James Withers[1]; *R. Storm*[1]; V. Shapovalov[1]; D. Myers[1]; R. Loutfy[1]; [1]MER Corporation

MER has developed a new process for the manufacture of near net shape components of titanium alloys at a cost significantly lower than conventional manufacturing. Based on the MER plasma transferred arc (PTA) rapid additive manufacturing process, the low cost route is a single melt process that utilizes very low cost raw materials, and has been demonstrated with Ti-6Al-4V. The melt processing of the PTA manufacturing results in fully dense structures without the need for post hipping. The cost of near net shape manufactured components of Ti-6Al-4V is projected to be <$16/lb, compared to >$125 for ram graphite castings or >$50/lb for flat sheet. The mechanical properties obtained for the low cost material will be reviewed, and examples of shapes produced will be shown. The low cost route is expected to find major usage in both commercial and defense applications where light weight, high strength, and corrosion resistance are important.

**2:50 PM**

**Development of High Strength Titanium Alloy Bar Stock from $TiH_2$ Powder**: *Curt Lavender*[1]; Elizabeth Stephens[1]; Eric Nyberg[1]; Vladimir Moxson[2]; Volodymr Duz[2]; [1]Battelle - Pacific Northwest National Laboratory; [2]ADMA Products Inc.

A new low-cost method to produce $TiH_2$ for use in blended elemental powder metallurgy has been under development and has been synthesized into many alloys. This paper reports on the results of use the $TiH_2$ powder for the production of the Ti6Al4V and the difficult to cast Ti1Al8V5Fe alloys via cold iso-static pressing, sintering and rod rolling. Static and dynamic mechanical properties of the as rolled and heat treated materials were characterized by room temperature tensile, compression and fatigue tests. Elastic constants were determined by ultrasonic techniques. This paper will summarize the characterization of the material and discuss the suitability for use in automotive suspension applications.

**3:15 PM**

**Titanium Reduction through Carbothermic Reduction and Molten Salt Electrolysis**: Xiaohui Ning[1]; Chengjun Gao[1]; Shuqiang Jiao[1]; *Hongmin Zhu*[1]; [1]University of Science and Technolgoy Beijing

Titanium dioxide can be reduced by carbon to form oxycarbide TiCxOy. In a certain range of components, titanium oxycarbide is an electronic conductor, and is able to be dissolved into molten salts through anodic electrolysis. During the anodic electrolysis of the titanium oxycarbide, titanium dissolves as as an ion of titanium, while carbon and oxygen leave the anode as carbon oxide. By controlling the conditions of carbothermic reduction, and molten salt electrolysis, pure titanium can obtained on the cathode.

**3:40 PM Break**

**3:55 PM**

**High Temperature Electrolysis of Ti and Its Alloys with a DC-ESR Unit**: *Toshihide Takenaka*[1]; Hidetaka Matsuo[1]; Mitsuru Sugawara[1]; Akihiro Matsuyama[1]; Masahiro Kawakami[1]; [1]Toyohashi University of Technology

Direct electrowinning of Ti metal above its melting point should bring the simplification of its production process. From this viewpoint, high temperature electrolysis of Ti and its alloys has been investigated by using a direct current electro-slag remelting (DC-ESR) unit. Liquid Ti, Ti-Al and Ti-Fe were obtained by this method in a CaF2-CaO-TiO2, CaF2-CaO-TiO2-Al2O3 and CaF2-CaO-TiO2-FeO bath, respectively. It was shown that Ti deposition was strongly affected by the state of the Ti ion in the bath and by the heat generation during the electrolysis. Strong influence of the bath composition on the Ti content in the Ti-Al was not seen, whereas Ti-Fe alloy was deposited only in the CaF2-CaO-TiO2-FeO bath of a limiting composition.

**4:20 PM**

**Development of Novel Alloying Techniques for Cost-Affordable Titanium**: *Peter Collins*[1]; Santhosh Koduri[2]; John Sosa[2]; Hamish Fraser[2]; Jim Sears[1]; [1]Quad Cities Manufacturing Lab; [2]The Ohio State University

As the means of producing cost-affordable Titanium powder mature, there exists simultaneously the need to advance fundamental understandings regarding microstructure-property relationships in Ti-powder and opportunities to develop novel techniques for alloy development. Thus, an overview of the need will be given with an emphasis of the emerging models which incorporate composition and microstructure into property predictions. Regarding the latter, two techniques for alloy development will be described. The first makes use of LENS™, an additive manufacturing technique where it is possible to us deposit composition gradients among Ti alloys using either pre-alloyed or element blends of powder, and thereby tailor the properties and microstructures to achieve local design minimums. The second exploits Kinetic Metallization to produce alloys from elemental blends. An overview of these techniques and current research will be given, specifically as related to the Quad Cities Manufacturing Lab – a fully integrated titanium powder processing facility.

**4:45 PM**

**Thermal Plasma Synthesis of Titanium Diboride**: *Muralidharan Ramachandran*[1]; Sutham Niyomwas[2]; Ramana Reddy[1]; [1]The University of Alabama; [2]Prince of Songkla University

A novel processing technique using thermal plasma was used to investigate the production of fine $TiB_2$ particles. The thermodynamic feasibility of the powder production process and the equilibrium composition were calculated using thermodynamic software. The yield of $TiB_2$ as a function of molar ratio of $CH_4$ was calculated using a constant molar ratio of $TiO_2$:$B_2O_3$ = 1:1. Results showed that the maximum yield was obtained at a molar ratio of $TiO_2$:$B_2O_3$:$CH_4$ = 1:1:6. Experiments were conducted using the thermal plasma reactor with varying molar ratios of $CH_4$. At the molar ratio of $TiO_2$:$B_2O_3$:$CH_4$ = 1:1:5, highest yield of $TiB_2$ was obtained. Effect of process parameters such as the feed rate and the plasma input power for the production of $TiB_2$ powders were investigated.

**5:10 PM**

**Electrochemical Productioin of Titanium from Oxycarbide Anodes**: *Ole Kjos*[1]; Geir Haarberg[1]; Ana Martinez[2]; [1]Norwegian University of Science and Technology; [2]SINTEF Materials and Chemistry

The use of titanium metal is increasing worldwide, but the high cost of metal production is limiting its use. This high cost is mainly due to the complicated and inefficient existing production method, the Kroll process. A new production

route is therefore desired, and an electrochemical approach would seem like a sensible way to go. Due to the properties of titanium, no existing electrochemical process is suitable. This work has focused on investigating the use of titanium oxycarbide anodes as a source of titanium ions in an electrolytic production process from a molten salt. The anodes were manufactured from a purified $TiO_2$-rich slag, which was heatet with carbon to form a TiC-TiO solid solution. These anodes were then used in electrolysis experiments in different molten chloride and fluoride electrolytes. The anode reacts to CO-gas and form $Ti^{3+}$ ions in the melt.

## Electrode Technology for Aluminum Production: Cathodes - Materials and Operation

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
*Program Organizers:* Ketil Rye, Alcoa Mosjøen; Morten Sorlie, Alcoa Norway; Barry Sadler, Net Carbon Consulting Pty Ltd

Monday PM Room: 616
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Manfred Banek, SGL Carbon GmbH

**2:00 PM Introductory Comments**

**2:05 PM**
**Carburation Phenomenons at the Cathode Block/Metal Interface**: Martin Lebeuf[1]; Marc-André Coulombe[1]; Patrice Chartrand[2]; Benedicte Allard[3]; *Gervais Soucy*[1]; [1]Université de Sherbrooke; [2]École Polytechnique de Montréal; [3]Carbone Savoie

The industrial cathode blocks wear more rapidly with increasing current density and bath acidity, the current trend in the aluminium industry. The importance to understand, measure and control the degradation mechanisms is obvious when considering the cost of replacing such potlines. One of the accepted wearing mechanisms is the aluminium carbide formation and dissolution process. Six experiments were performed to study this mechanism and the effect of current density, time and electrolyte exposition of the surface on the amount of aluminium carbide produced. The aluminum carbide formation has been studied using XPS, SEM-EDS, as well as XRD. The bath chemistry, before and after electrolysis, was characterized by XRD and XRF. Increasing current density, time, and surface unexposed to the electrolyte enhance the accumulation of aluminium carbide in the few first millimeters in the cathode block. The relation between the aluminium carbide formation and the final bath chemistry will be discussed.

**2:25 PM**
**Erosion Measurements of High Density Cathode Block Samples through Laboratory Electrolysis with Rotation**: *Yoshinori Sato*[1]; Pascal Lavoie[2]; Pretesh Patel[3]; [1]SEC CARBON LTD.; [2]Light Metals Research Center, University of Auckland; [3]The Light Metals Research Centre, University of Auckland

The increasing trend in line current over recent years has seen the erosion rate of graphitized cathode blocks also increasing, which has led to the problem of decreasing trends in the pot life. This has led some smelters to adopt cathode blocks with high density, especially pitch impregnated cathode blocks as an anti-erosion measure. SEC CARBON and the Light Metals Research Center tried to evaluate the erosion of high density cathode block samples with by using an apparatus which was designed with a rotating cathode to promote physical erosion and electrochemical erosion at the same time. Results have shown that pitch impregnated materials were found to have no benefit in regards to erosion resistance. This paper will primarily discuss the impact of formulation, pitch impregnation and processing techniques on the electrochemical erosion resistance of graphitized materials.

**2:45 PM**
**Thermo-Mechanical Characterisation of Graphitic and Graphitized Carbon Cathode Materials Used in Aluminium Electrolysis Cells**: *Donald Picard*[1]; Wadii Bouzemmi[1]; Bénédicte Allard[2]; Houshang Alamdari[1]; Mario Fafard[1]; [1]Aluminium Research Centre - REGAL; [2]Carbone-Savoie

In the conventional Hall-Héroult electrolysis process, both the carbon anode and cathode operate at up to 980°C under a very corrosive environment. Since the lifetime expectancy is an important economic point to take into consideration it is imperative that all process related phenomena present in the cells be well understood, e.g. the thermo-mechanical behaviour of carbon cathode materials used into the cell linings. To do so, an experimental program based on uniaxial compressive tests, for a temperature range from 25°C to 1000°C, has been conducted on graphitic and graphitized cathodes. The Young's modulus, compressive strength and damaging coefficients at various temperatures are examples of measured properties. The properties of both cathode block types were compared and analysed. Mainly, the damaging at high temperature can be neglected in the graphitic cathode block but not in the graphitized one. The Young's modulus evolution with temperature also differs from the two materials.

**3:05 PM**
**Electrical Resistance of Graphitic and Graphitized Cathode Materials at Elevated Temperatures**: *Jilai Xue*[1]; Jun Zhu[1]; Yunxia Song[1]; [1]Unversity of Science and Technology Beijing

Electrical resistance is of great importance for cathode performance and energy consumption in aluminum reduction cells. The values of the electrical resistance for the cathodes are material and temperature dependent. The electrical resistance of graphitic and graphitized cathode materials before and after electrolysis was also measured at temperatures from 30°C to 965°C. An increase in electrical resistance was found with all samples after the electrolysis under identical conditions, and the graphitized one had the lowest resistance value among the testing samples. SEM inspection showed more salt penetration and micro-cracking in the semi-graphitized cathode than in the graphitized one, which may be the causes for the change in electrical resistance after electrolysis. Higher graphitization and lower porosity can reduce the electrical resistance of the cathodes. The electrical resistance of TiB2-carbon composite material exhibited a higher value than those of graphitic and graphitized cathodes at elevated temperatures.

**3:25 PM**
**Development of High Density Graphitized Cathode Blocks for Aluminium Electrolysis Cells**: *Sten Yngve Larsen*[1]; Xian-An Liao[1]; Hermann Gran[1]; Stian Madshus[1]; Johan Arnold Johansen[1]; [1]Elkem Carbon AS

New types of coke were successfully introduced one decade ago for production of semigraphitic (E-30), graphitic (E-100) and especially graphitized (E-G) cathode blocks. Compared to cokes historically used for production of cathode blocks, these cokes are different in both structure and chemical composition. The present paper mainly describes the characteristics of E-G blocks. Compared to the graphitized cathode blocks based on traditional cokes, E-G blocks have been found to be denser, less porous, more resistant to wear in aluminium electrolysis as well as having a slightly higher sodium expansion rate and a higher coefficient of thermal expansion. An average apparent density in excess of 1.72 g/cm3 was achieved without pitch impregnation. In addition E-G blocks have more evenly distributed properties in different directions than the traditional graphitized cathode blocks. These characteristics are expected to contribute to a longer pot life and a more stable pot operation.

**3:45 PM Break**

**4:00 PM**
**Sodium Diffusion in Cathode Lining in Aluminium Electrolysis Cells**: *Zhaohui Wang*[1]; Jørn Rutlin[2]; Tor Grande[1]; [1]Norwegian University of Science and Technology (NTNU); [2]Norsk Hydro Aluminium AS

A qualitative understanding of the diffusion of sodium into the cathode lining in aluminium electrolysis cells, including the carbon cathode, the ceramic sidelining and refractory lining, is important for the improvement of cathode cell design and performance. In this paper, a mathematical model is used to describe sodium diffusion and reaction behavior through the entire cathode lining. In addition to the degradation mechanisms in different lining materials, a mass balance of sodium consumption in a typical cell is considered and used for the verification of the model. The simulation gives additional insight into the transport of sodium in the carbon cathode and demonstrates that sodium transport in the cathode goes through solid state diffusion. The model is also used to simulate the consequences of changes in the carbon materials in the cathode lining.

**4:20 PM**

**Characterization of Sodium Expansion of Industrial Graphitic and Graphitized Cathodes**: *Jilai Xue*[1]; Liancheng Wu[2]; Qingsheng Liu[1]; Qingren Niu[2]; Wei Wang[1]; Xin Hou[2]; Jun Zhu[1]; Hua He[2]; [1]University of Science and Technology Beijing; [2]Ningxia Qingtongxia Energy Aluminium Group, China Power Investment Corporation

A series of experiments with various industrial cathodes ranging from 35% to 100% graphitic and fully graphitized carbons were carried out using a modified Rapoport apparatus. Sodium expansion during aluminum electrolysis was measured against time at 960°X. For a higher cryolite ratio, the cathodes with lower graphitic carbons exhibited more sodium expansion than the fully graphitized carbon, while for a lower cryolite ratio, such a difference become less profound among the tested samples. XRD characterization shows that the d002 value of all types of the cathode samples increases after aluminum electrolysis, and the increased graphitization (p002) in the cathode carbons can reduce the sodium expansion. SEM-EDS inspection reveals that the sodium and fluorides penetration within the cathode samples has different patterns for varying graphitization of the carbon materials. The results can provide useful information for operational evaluation of the industrial cathode products as well as for improvement in cathode performance.

**4:40 PM**

**Electrochemical Investigation of Potassium Intercalation into Graphite**: Dongren Liu[1]; *Wangxing Li*[2]; Zhanhong Yang[1]; Shilin Qiu[2]; Yingtao Luo[2]; [1]Central South university; [2]Zhengzhou Research Institute of Chalco

Electrochemical intercalation of potassium into graphite in molten KF was investigated by means of cyclic voltammetry, chronoamperometry, galvanostatic electrolysis and open-circuit potential measurements. It was found that potassium intercalated into graphite proceeding via the mechanism of intercalation between graphite layers. The intercalation compound formed in graphite matrix in molten KF was quite unstable. The intercalation process was found to be governed by both the diffusion of potassium ion in graphite bulk and the phase transition kinetics. The transfer coefficient of the intercalation reaction was calculated according to the parameters resulting from nonlinear fitting of the current-time transient curves with a quasi-reversible equation. Analysis with scanning electron microscope analyses shows graphite was severely eroded by intercalation of potassium. These findings could provide a deeper understanding of the basic mechanism of potassium interaction with cathodic carbon block in the Hall-Héroult process when potassium cryolite would be used as low temperature electrolyte.

**5:00 PM**

**Corrosion Resistance of Cathode to NaF-KF-ALF3-Based Electrolyte**: *Hengwei Yan*[1]; Wangxin Li[2]; Shilin Qiu[2]; Ji Li[1]; [1]Central South University; [2]Zhengzhou Research Institute of Chalco

The KF-AlF3-based electrolyte is a kind of promising bath in low-temperature aluminum reductuion, but it has strong corrosion to cathode material. In this paper,the cathode expansion rate in KF-AlF3-based electrolyte was tested for semi-graphite cathode and graphitization cathode, after electrolysis, the cathodes were analyzed by SEM-EDS. The experimental results show that when KF content is higher than 7wt.%, the semi-graphite cathode were corroded seriously after electrolysis, but for graphitization cathode, despite KF content in electrolyte reach 20wt.%, the expansion rate still keep in an acceptable level. The result suggest that the graphitization cathode can be used with KF-AlF3-based electrolyte in aluminum reductuion process.

**5:20 PM**

**Development Status of Processing Technology for Spent Potlining in China**: *Xiping Chen*[1]; [1]Zhengzhou Research Institute of Chalco

With the development of Chinese aluminum industry, wastes from smelters are severely restricted by the government. Different processing technologies for spent potlining (short as SPL) have been developed in the past five years. Main SPL processing technologies are Detoxifying with limestone, coal cinder by Pyro-process, Detoxifying with bauxite in Aluminum Sintering Process and Floatation of SPL. Three typical technologies were put into industrial application in Pingguo Smelter, Shangdong Smelter and Yichun Smelter, respectively. SPL plant in Pingguo which adopts Pyro-process has a capacity of 15000 metric tons per year. SPL-bauxite Sintering Process used in Shangdong can treat 6000 metric tons SPL every year. Floatation of SPL was put into industrial test in Yichun this year and the test scale is 1000 metric tons SPL per year. Different products which were received from three processes can meet national standard, at the same time SPL can be sufficiently detoxified after processing.

## Electrometallurgy - General Session: Session I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Aqueous Processing Committee
*Program Organizer:* Michael Free, University of Utah

Monday PM — Room: 310
February 15, 2010 — Location: Washington State Convention Center

*Session Chair:* Michael Free, University of Utah

**2:00 PM**

**A Sandwich Structure Lead-Based Composite Porous Anode for Zinc Electrowinning**: Jiang Liangxing[1]; *Lv Xiaojun*[1]; Lai Yanqing[1]; Li Jie[1]; Liu Yexiang[1]; [1]Central South University

In order to improve the electric conductivity and mechanical performance of lead-based porous anode (PA) for zinc electrowinning, a sandwich structure composite porous anode (CPA) was prepared by counter-gravity infiltration and its structure was optimized. SEM observation shows that under the optimized infiltration conditions, the core and foam outside can integrate together. The results of anodic polarization show that the pore diameter and thickness of foam influence the anodic potential, and the optimized value of them is 1.43-1.6mm and 2mm, respectively. Mechanical performance measurements show that pore diameter and thickness of foam has negligible effect on tensile strength, but the thickness of core metal has great effect on it. According to the practice in zinc electrowinning, the thickness of core metal was designed to 2mm, and the tensile strength is about 8MPa, which is 3 times of PA. With the optimized structure, the electric conductivity is 2 times of PA.

**2:25 PM**

**Constant and Pulse Voltage Applications in $CaWO_4$ Reduction**: Orhan Goksu[1]; Ishak Karakaya[1]; *Metehan Erdogan*[1]; [1]Middle East Technical University

It has been shown that metallic tungsten powder can be obtained by electrochemical reduction of solid $CaWO_4$ in molten $CaCl_2$-NaCl eutectic mixture at 600°C. In this study, constant and pulse voltage electrolysis experiments were performed to optimize electroreduction kinetics and to obtain information about the mechanism of above process. It was found that pulse voltage applications yield faster reduction rates when average voltage of pulse voltage application was the same as constant voltage application. In addition, analysis of the current vs. time graphs of constant and pulse voltage electrolysis studies showed that reduction occurs at potential differences higher than 2.25 V between $CaWO_4$ cathode and graphite rod anode. Cyclic voltammetry studies and thermodynamic computations were performed to support above result.

**2:50 PM**

**Electrochemical Formation of Mg–Li-Y Alloys at Solid Magnesium Electrode from LiCl–KCl –YCl3 Melts**: *Pengkai Wang*[1]; Huimin Lu[1]; Feng Shi[1]; [1]Beihang University

In this paper, the electrochemical formation of Mg–Li-Y alloys from LiCl–KCl –YCl3 melts at 823K was conducted with solid Magnesium cathode. The cyclic voltammetry, chronopotentiometry and chronoamperometry were used to study the electroreduction mechanism and controlling step of Li and Y electrodepositing on Mg cathode. The results indicated that Li(I) and Y(III) could be reduced at -2.1V(Vs.Mo) and -1.1V(Vs.Mo), the electroreduction processes of Li and Y were quasi-reversible diffusion controlled reactions including one and three electrons transfer respectively. The X-ray diffraction and scanning electron microscopy analyses also showed Mg–Li-Y alloys could be obtained by potentiostatic electrolysis at 3.2V, formation of Mg-Li-Y alloys controlled by applied current, and Lithium and Yttrium contents by electrolysis time. At this moment, the cathodic density was 2A/cm2; Li and Y could co-deposit and diffuse into the Mg cathode to form the Mg-Li-Y alloys.

**3:15 PM**
**Electrochemistry of Tantalum Pentachloride in the Room Temperature Ionic Liquid 1-Butyl-3-Methy Imidazolium Hexafluorophosphate**: *Xiaoxiang Zhang*[1]; Huimin Lu[1]; Tao Zhang[1]; [1]Beihang University

Electrochemical behavior of 0.25M tantalum chloride on platinum substrate was investigated in the 1-butyl-3-methy imidazolium hexafluorophosphate ([Bmim]PF6) by cyclic voltammetry, chronopotentiometry and chronoamperometry. Cyclic voltammetrys of tantalum pentachloride in the employed ionic liquid at 100ºC exhibit four reduction peaks, the first reduction peak contributes to the reduction of trichloride anion(Cl3-) at -0.4V vs. Pt, the second corresponds to the reduction of the tantalum (V) to tantalum (III) at -0.9V vs. Pt, the third is attributed to the reduction of the tantalum (III) to tantalum metal at -1.25V vs. Pt and the fourth is the formation of various tantalum subchlorides at -1.6V vs. Pt. The results have shown that the electrodeposition of tantalum from tantalum pentachloride in [Bmim]PF6 is quite complicated process, but tantalum can be electrodeposited in thin layers.

**3:40 PM Break**

**3:50 PM**
**ZrB2 Produced with Molten Salt Electrolysis**: *Selda Özkan*[1]; Servet Timur[1]; Mustafa Urgen[1]; [1]ITU Metallurgical and Materials Engineering Department

In this study, a combination of cathodic arc physical vapor deposition (PVD) and molten salt electrolysis methods were used to obtain ZrB2 layers on AISI 304 grade stainless steel surface. Prior to boriding, steel surface was coated with metallic zirconium via cathodic arc PVD method and then the zirconium coated steel was borided by molten salt electrolysis. Well adherent, dense, ZrB2 layers are formed on the substrates. The thickness of the ZrB2 layer showed dependence on the thickness of the metallic Zr coating layer and the time of boriding. During the boriding process the alloying elements of the substrate also took part in the coating structure depending on their diffusion characteristics.

**4:15 PM**
**Effects of Ultrasound on Cell Voltage of Aluminum Electrolysis in Cryolite-Alumina Melts**: *Jilai Xue*[1]; Shao Hua[1]; Jun Zhu[1]; [1]University of Science and Technology Beijing

Aluminum electrolysis aided with ultrasound was carried out in cryolite-alumina melts in a laboratory cell, and a layer CO2 gas bubbles was formed on the anode. Cell voltage varied as the bubbles generated on the anodes surface, and reduced by applying ultrasound on the anode. The effects of ultrasound frequency and anodic current density were investigated with ultrasound on and off, alternatively during the electrolysis process. The cell voltage can be lowered by 5% with ultrasound, which demonstrate the potential energy saving for aluminum reduction process.

**4:40 PM**
**Evaluation of the Corrosion Behavior of Laser Welded and GTAW Welded Austenitic Stainless Steel 316L in Lithium Bromide and Comparing the Inhibition Effect of Chromate, Bromate and Molybdate on the Corrosion Behavior of Austenitic Stainless Steel 316L in Lithium Bromide**: *Ahmad Momtaz*[1]; [1]German University in Cairo

The corrosion behavior of ASS 316L laser welded and GTAW welded in lithium bromide concentrations of 0.05, 0.1, 0.5, 1, 3, 4.6, 6, 8.06 and 9.79 M was evaluated using electrochemical methods and optical microscopy. The inhibitors used were: sodium chromate, sodium bromate and sodium molybdate. Optical microscopy showed that ferrite was formed in the austenite matrix exhibting austenite ferrite solidification. Open circuit potential showed that laser welded had more noble potentials than GTAW welded at most of the concentrations. The laser welded showed lower corrosion currents and corrosion rates than GTAW welded. Moreover, laser welded showed better pitting resistance than GTAW welded, while GTAW welded showed better repassivation behavior than laser welded. The sodium chromate approved to be the most effecient inhibitor. Moreover, laser welded is inhibited more effeciently by the three types of inhibitors than GTAW welded. Laser welding is better than GTAW welding for ASS 316L.

## Failure of Small-Scale Structures: Deformation and Failure

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
*Program Organizers:* Marian Kennedy, Clemson Univeristy; Brad Boyce, Sandia National Laboratory; Reinhold Dauskardt, Stanford; Zhiwei Shan, Hysitron Inc

Monday PM Room: 206
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Reinhold Dauskardt, Stanford University

**2:00 PM Invited**
**SIZE MATTERS: Nano-Scale Mechanical Properties of Single Crystals, Nanocrystalline Metals, and Amorphous Metallic Glasses**: *Julia Greer*[1]; Ju-Young Kim[1]; Dongchan Jang[1]; Michael Burek[2]; [1]California Institute of Technology; [2]University of Waterloo

When microstructural or external sizes of materials are reduced to nano-scale, they exhibit unique behaviors. We fabricate nanopillars with different initial microstructures ranging from 50 nm to 1 micron by using Focused Ion Beam and E-beam lithography/electroplating approaches. Their strengths in uniaxial compression and tension are subsequently measured in in-situ mechanical deformation instrument, SEMentor. We discuss nano-mechanical behavior in three distinct material classes: single crystals, nano-crystalline metals, and metallic glasses. We observe SMALLER is STRONGER phenomenon in single crystals while nano-crystalline metals exhibit SMALLER is SOFTER trend. Metallic glasses show both strength increase and ductility when reduced to nano-scale. Unlike in bulk, nano-scale materials also exhibit numerous discrete strain bursts. We attribute these dissimilarities to free surface effects, leading to unique dislocation behavior in crystals, grain-boundary activity in nano-crystalline solids, and shear transformation zones in metallic glasses, serving as fundamental reason for observed failure and plasticity mechanisms.

**2:25 PM**
**The Role of Grain Boundaries in the Creep of Sub-Micrometer Thick Cu and Cu/$Si_3N_4$ Microbeams at 300 K**: *Robert Klassen*[1]; Yong Liu[2]; [1]The University of Western Ontario; [2]Eaton Corporation

Constant-load creep tests were performed at 300 K on 250 μm long cantilever microbeams of Cu and Cu/$Si_3N_4$ containing a sputter-deposited Cu layer between 200 and 1300 nm thick. A nano-indentation tester was used to apply the load and to measure the displacement of the microbeams during the creep tests. The internal stress and strain rate within the microbeams was determined by FE modelling incorporating user-defined material constitutive expressions. The reduced average grain diameter within the thinnest Cu microbeams was found to enhance the contribution from grain boundary diffusional creep and reduce the contribution from obstacle-limited dislocation glide. The activation energy and the athermal flow stress were found to be larger for Cu/$Si_3N_4$ than for the free-standing Cu microbeams of the same thickness. This suggests that the presence of the $Si_3N_4$ layer invokes a higher dislocation density which results in stronger obstacles to dislocation glide.

**2:40 PM Invited**
**Competing Roles of Deformation and Void Formation during Rapid Thermal Cycling of Metal Interconnects**: *Robert Keller*[1]; David Read[1]; Roy Geiss[1]; [1]NIST

Development of methods for testing the thermomechanical reliability of metal interconnects has led to the observation of a transition in material response during rapid thermal cycling, as induced by alternating electric current cycling at 100 Hz. In tests of partially encapsulated copper lines in vacuum, thermal cycling over temperature ranges of approximately 250°C and higher resulted in the formation of damage largely consistent with dislocation slip due to thermomechanical fatigue. Thermal cycling over temperature ranges of approximately 225°C and lower resulted in the formation of damage consistent with diffusive mechanisms, i.e., voids. This observed range of transition temperature appears to be similar to the optimum hold temperatures seen for the formation of stress voids in highly constrained interconnects. We discuss

this transition in terms of local conditions of stress, temperature, constraint, heating/cooling rates, and localized variations in microstructure.

**3:05 PM**
**Visualization of Failure Mechanisms in Nanocrystalline Thin Films**: Krishna Jonnalagadda[1]; John Sharon[1]; Kevin Hemker[1]; *Kaliat Ramesh*[1]; [1]Johns Hopkins University

One of the main reasons for early failure of nanocrystalline metals is the presence of voids. The mechanical behavior of nanocrystalline metals with voids was investigated via in-situ optical and scanning electron microscope tensile experiments. The 200 nm thick, electron transparent, nanocrystalline aluminum films were patterned with small 5-10 micron holes to understand failure via void growth. Under tensile loading, the void size increased, becoming elliptical, and a small crack nucleated normal to the loading direction. Under displacement controlled loading the crack growth was slow until complete failure of the film. Experiments are also conducted in which the crack growth is arrested and the deformation mechanisms are investigated with TEM. Due to high stress concentration at the crack tip both grain growth and void nucleation are expected along the crack front. Furthermore, the void growth rate and its change in shape are modeled considering 2D plane stress in the film.

**3:20 PM**
**Tuning the Mechanical Properties of a Nanoporous Gold**: *Hai-Jun Jin*[1]; Lilia Kurmanaeva[1]; Jörg Weissmüller[1]; [1]Forschungszentrum Karlsruhe GmbH

Conventional approach towards tailoring the mechanical properties of materials is to manipulate their atomic structure or microstructure. However, instead of these "permanent" changes, here we introduce a new concept to develop "smart" materials which allows prompt and reversible variation of its mechanical properties. We demonstrate in bulk nanoporous gold samples prepared by dealloying, with very small structure size and large surface area, that both the yield stress and the brittleness can be "tuned" in an electrochemical environment. Compression tests were preformed in situ with samples immersed in the electrolyte and with potentiostatic control. Potential changes allow a reversible variation of the flow stress by as much as the factor two. Furthermore, the samples are obviously more brittle at positive potential. The underlying mechanisms will be discussed in terms of surface dislocation nucleation and dislocation motion concerning the roles of the surface stress, surface energy and even the surface diffusivity.

**3:35 PM Break**

**3:50 PM Invited**
**Failure of Protein Materials in Extreme Conditions and Disease**: *Markus Buehler*[1]; [1]Massachusetts Institute of Technology

Biological protein materials feature hierarchical structures, ranging through the atomistic, molecular to macroscopic scales, forming functional biological tissues as diverse as spider silk, tendon, bone, skin, hair or cells. Here I will present computational studies, focused on how protein materials deform and fail due to extreme mechanical conditions, disease and injuries. Based on a multi-scale atomistic simulation approach that explicitly considers the architecture of proteins including at the chemistry level, we have developed predictive models of protein materials, validated through quantitative comparison with experimental results. This bottom-up approach enables us to extract fundamental physical concepts that control the properties of protein materials. I will present studies of several major classes of protein materials, including cellular alpha-helix rich protein networks, beta-sheet structures as found in spider silk and amyloids, as well as collagenous tissues that form tendon and bone. Materials failure in the contact of genetic diseases will be discussed.

**4:15 PM**
**Deformation and Fracture in Human Skin**: *Kemal Levi*[1]; Victoria Hsiao[1]; Ruiliang Jia[1]; Reinhold Dauskardt[1]; [1]Stanford University

The outermost layer of human skin, the stratum corneum (SC), is a critical small-scale biological structure with important chemical barrier and mechanical properties. The tissue is 10-25 μm thick and has cellular and intercellular components that vary from tens of microns to submicrons in size. We describe micromechanical techniques to probe the biomechanical properties of the SC together with optical and atomic force microscopy integrated with digital image correlation. We demonstrate how environmental, enzymatic and chemical treatments influence SC components and their resulting mechanical properties. We also describe novel thin-film methods to probe the resistance to intercellular delamination and the stresses that arise naturally in SC as a result of treatment or environmental conditions. Finally, a biomechanics framework to account for the SC drying stress as a mechanical driving force for dry skin damage is presented. This research presents a new approach to characterize the fundamental biomechanics of human SC.

**4:30 PM Invited**
**Size Dependent Deformation in Polymers - Experiments and Theory**: *Chung-Souk Han*[1]; [1]North Dakota State University

Similar to the size-dependent deformation observed in metals at length scale ranges from microns down to nanometers,many polymers exhibit size dependent deformation at these length scales as well. While for metals such size dependent deformation phenomena are commonly attributed to geometrically necessary dislocation densities,this notion can not be applied to polymers and a sound theory explaining these phenomena in polymers seems not to be available. Experimental investigations where such size dependent deformation has been observed are reviewed here and include microbeams, indentation testing, and foam and composite materials.In addition a related micromechanically motivated higher gradient theory incorporating a Frank energy type component to the deformation potential is suggested for the prediction and rationalization of such size dependent deformation in polymers.This theory is discussed with respect to the reviewed experiments and it is found that it is capable to link some molecular properties of the polymer to its size dependent deformation.

## Global Innovations in Manufacturing of Aerospace Materials: The 11th MPMD Global Innovations Symposium: Innovations in Casting Technologies

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Shaping and Forming Committee, TMS: High Temperature Alloys Committee
*Program Organizers:* Deborah Whitis, General Electric Company; Thomas Bieler, Michigan State University; Michael Miles, BYU

Monday PM Room: 306
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Chris Woodward, AFRL-RX; John Miller, TBD

**2:00 PM Invited**
**Advances in the Solidification of Single Crystal Superalloys**: *Tresa Pollock*[1]; Clinique Brundidge[1]; Jonathan Madison[1]; Jonathan Miller[2]; [1]University of Michigan; [2]Air Force Research Laboratory

Recent challenges in the solidification of superalloy single crystals include new creep resistant alloys, the need for physically large single crystals for power generation turbines and the linkage of solidification process models to mechanical property models. Recent developments in high gradient processing and techniques for quantifying solidification structure and defects will be discussed. Key features of the high gradient liquid metal cooling (LMC) process for solidifying superalloy single crystals will be quantified. New insights to defect formation that arise from fluid flow studies performed on 3-D dendritic structures obtained by serial sectioning will be presented. Finally, the aspects of solidification structure that influence creep and fatigue will be discussed along with the needs for linkage of process models to property models.

**2:30 PM**
**Direct Digital Manufacturing of Airfoils**: *Suman Das*[1]; John Halloran[2]; Wil Baker[3]; [1]Georgia Institute of Technology; [2]University of Michigan; [3]Honeywell Aerospace

Direct Digital Manufacturing (DDM) of airfoils is a concept that disrupts the current state–of–the–art investment casting process for manufacturing superalloy airfoils. DDM of airfoils will be achieved by processing photocurable ceramic resins through a new technology known as Large Area Maskless Photopolymerization (LAMP). LAMP combines layered manufacturing of complex three–dimensional objects with the fine–feature resolution and high throughput of massively parallel scanning maskless lithography to achieve a disruptive breakthrough in part build speed and feature definition. DDM of airfoils will eliminate nearly all the tooling, handling, and associated causes for scrap in the investment casting process. Thus, it will disrupt not only the cost

structure of conventional investment castings, but also the speed with which components can be fabricated. This presentation will highlight progress made to date on development of LAMP technology. This work is sponsored by DARPA grant HR0011–08–1–0075.

**2:50 PM**

**Experimental and Mathematical Modeling Progress on Scanning Laser Epitaxy: A Technique for Growing Single Crystal Superalloys**: Michael Kirka[1]; Rohan Bansal[1]; *Suman Das*[1]; [1]Woodruff School of Mechanical Engineering, Georgia Institute of Technology

We present recent progress on scanning laser epitaxy, a laser additive manufacturing technique under development for producing single crystal growth in nickel–based superalloys. Experimental investigations have been performed on the single-crystal capable nickel alloy CMSX-4. Additionally, mathematical models have been coupled with analytical information obtained from experiments to understand the scanning laser epitaxy process and the requirements to achieve monolithic deposits on like chemistry single–crystal nickel superalloy substrates. Presentation of microstructure development and need of process control in the context of the laser generated melt pool will be discussed from the view-point of repairing high–value single–crystal turbine engine components. This work is funded by the Office of Naval Research contract #N00173–07–1–G012.

**3:10 PM**

**Modeling of Grain Selection during Directional Solidification of Superalloy Single Crystal Turbine Blade Casting**: Dong Pan[1]; Qingyan Xu[1]; *Baicheng Liu*[1]; Jiarong Li[2]; Hailong Yuan[2]; Haipeng Jin[2]; [1]Tsinghua University; [2]Beijing Institute of Aeronautical Materials

Superalloy single crystal turbine blade are nowadays widely used as key parts in gas turbine engines. The single crystal turbine blade casting properties are quite sensitive to the grain orientation determined directly by the grain selection behavior of the grain selector.A mathematical model was proposed for the grain selection during directional solidification of turbine blade casting. Based on heat transfer model during directional withdrawing process, the competitive grain growth within the start block and the spiral part were simulated by using cellular automaton method (CA). Validation experiments were carried out, and the measured results were compared quantitatively with the predicted results. It is indicated that the model could be used to reproduce the grain morphology and the competitive grain evolution during solidification, together with the distribution of grain orientation of primary <001> dendrite growth direction, with respect to the longitudinal axis of the turbine blade casting.

**3:30 PM Break**

**3:50 PM**

**Prediction of As-Cast Grain Size Distribution from a Model of Equiaxed Solidification with Free Dendrite Transport**: *Wajira Mirihanage*[1]; David Browne[1]; [1]University College Dublin

As-cast microstructure is one of the essential engineering concerns for aerospace materials. Movement of equiaxed dendrites during solidification has a significant influence on the final microstructure of shape cast components. The authors have developed a predictive model of equiaxed solidification which treats dendrite transport in the solidifying alloy melt, at low computational cost. The non-equilibrium model considers nucleation from industrial inoculants, growth and movement of dendrites. The motion of free dendrites is computed as the combined effects of sedimentation settling of denser dendrites and transport of small dendrites by liquid flow due to natural convection. When free dendrites become coherent at the latter stage of solidification, the resultant equiaxed mush of coherent dendrites is assumed to have become a porous medium until it becomes completely solid. Simulations are used to establish the sensitivity of the as-cast structure of Al-Si shape castings to the magnitude of mould material heat transfer coefficients.

**4:10 PM**

**Damage Tolerant Cast Alloy Ti-5Al-5Mo-5V-3Cr for Aerospace Applications**: *Edward Chen*[1]; L.W. Weihmuller[2]; D.R. Bice[1]; G.D. Hall[2]; W.A. Thomas[2]; [1]Transition45 Technologies, Inc.; [2]Bell Helicopter Textron

It is well established that aerospace systems would benefit greatly from the development and application of high strength titanium alloys with improved durability and damage tolerance. Current and future commercial and military aircraft in particular have requirements for such alloys, particularly if near-net shape components can be manufactured as castings for affordability. The application of a higher strength titanium alloy versus the industry workhorse Ti-6Al-4V also allows direct conversion of forged titanium parts to titanium castings without sacrificing strength and durability or increasing weight. This presentation covers work performed to evaluate the emerging high strength titanium alloy Ti-5Al-5Mo-5V-3Cr-0.5Fe (Ti-5553) as castings. The microstructure-properties of this alloy including tensile strength, toughness, and damage tolerance under different thermo-mechanical processing conditions will be reviewed in depth. These results will be discussed in light of the potential applicability of cast Ti-5553 to aerospace structures. This work was supported by the Naval Air Warfare Center.

**4:30 PM**

**Coupling Computational Thermodynamics with Experimental Study for Accelerated Development of Mo-Si-B Based Alloys**: *Ying Yang*[1]; Hongbin Bei[2]; Easo George[2]; Jaimie Tiley[3]; Y. Chang[4]; [1]CompuTherm LLC; [2]Oak Ridge National Laboratory; [3]Air Force Research Laboratory; [4]University of Wisconsin-Madison

Recent advance in computational thermodynamics using the CALPHAD (CALculation of PHAse Diagram) approach has enabled us to rapidly obtain multi-component phase diagrams with significantly reduced experimental effort. In this presentation, the stable region of the three-phase (Mo)+Mo3Si+Mo5SiB2 equilibrium in the Mo-Si-B-X(X=Ti, Zr, Hf) system was rapidly established by coupling computational thermodynamics with guided experiments. Computational thermodynamics helps not only identifying critical alloy compositions for experimental study, but also understanding the complex microstructures in multi-component alloys. The results show that additions of Zr and Hf much limit the stable region of the three-phase (Mo)+Mo3Si+Mo5SiB2 equilibrium because of the formation of the ternary phases MoSiZr(Hf), while Ti addition leads to a much larger one. This work is an essential building block for developing high-performance Mo-Si-B based Refractory Metal Intermetallic Composites (RMICs) for ultra-high temperature applications (T>1200ºC).

**4:50 PM**

**Mechanical Properties of TiAl-Based Alloys for High Temperature Applications**: *Fereshteh Ebrahimi*[1]; Michael Kesler[1]; Sonalika Goyel[1]; Orlando Rios[1]; Damian Cupid[2]; Hans Seifert[2]; [1]University of Florida; [2]Freiberg University of Mining and Technology

There is a need to reduce the weight of the turbine engines used in aircrafts. Two-phase TiAl+Ti3Al alloys have recently been employed in the low temperature section of the turbine engines. In order to extend the applicability of TiAl-based alloys to higher temperatures we have designed alloys based on the TiAlNbCrMo system. These alloys solidify as solid solution beta phase which could be retained upon fast cooling to room temperature. Stable microstructures consisting of gamma (TiAl) phase with different volume fractions of the sigma (Nb2Al) phase were obtained by aging treatments. The results of this study elucidate that fracture at the prior beta grain boundaries limits the ductility of these alloys. In this paper the optimization of low temperature ductility and high temperature strength is discussed in terms of the volume fraction of the phases and their distribution. This work has been supported by NSF/AFOSR (DMR-0605702 and DMR-0856622).

**5:10 PM**

**Development of Ni-Mn-Based Braze Alloys for the Fast Epitaxial Braze Repair of Wide Cracks in Single-Crystalline Nickel-Base Superalloys**: *Britta Laux*[1]; Joachim Rösler[1]; [1]Technische Universität Braunschweig

Diffusion brazing is a widely-used technology for the repair of cracks in hot section turbine components, mostly fabricated from single-crystalline nickel-based superalloys. Typically, braze alloys similar to the base material, enhanced by fast diffusing melting point depressants like B, are used. In case of single-crystalline components, an epitaxial healing can be achieved, however, the filling of wide cracks in the range of 100-300 µm is difficult, due to the precipitation of brittle secondary phases. New Ni-Mn-based braze alloys enhanced by Al, Cr and Ti were developed, which allow a very fast epitaxial healing of wide cracks (300 µm). As B is replaced by Mn, the repair process can be significantly shortened since the epitaxial solidification is no longer completely diffusion controlled. Improved brazing cycles for the minimization of porosity, heat treatments producing a γ/γ'-microstructure very similar to the parent material as well as results from mechanical testing will be presented.

## Heterogeneous Nucleation and Initial Microstructure Evolution in Alloys and Colloids: Simulation II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS/ASM: Phase Transformations Committee
*Program Organizers:* Rainer Schmid-Fetzer, Clausthal University of Technology; Heike Emmerich, RWTH Aachen University; Frans Spaepen, Harvard University; Martin Glicksman, University of Florida; John Perepezko, University of Wisconsin, Madison

Monday PM Room: 614
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Dieter Herlach, DLR; Martin Glicksman, University of Florida



**2:00 PM Invited**
**Heterogeneous Nucleation as a Deterministic Process**: *A. Greer*[1]; [1]University of Cambridge

Heterogeneous nucleation in a wide variety of cases is likely to be deterministic, rather than stochastic. An example is the athermal heterogeneous nucleation of freezing, in which the number of nuclei generated is dependent on temperature and not on time, in contrast to many conventional analyses of nucleation rates. A correct analysis of the kinetics is essential for prediction of microstructure evolution. Accurate prediction may be facilitated when the nucleation is deterministic. Examples of such behaviour will be analysed and compared. They include not only cases from in materials science, but also from biological systems.

**2:25 PM**
**Heterogeneous Nucleation on Spherical and Flat Catalysing Surfaces**: *Ma Qian*[1]; [1]The University of Queensland

Fletcher's spherical substrate model is a basic model for understanding heterogeneous nucleation in nature. This work presents an analysis of this model using a novel analytical approach. It is revealed that when a special geometrical angle (pseudo-contact angle) were chosen for thermodynamic descriptions, the first derivatives of the free energy change with respect to embryo radius would be identical for nucleation on spherical and flat surfaces. It is further shown that there exists a local maximum in the difference between the equivalent contact angles for nucleation on spherical and flat surfaces. The negative spherical substrate size effect occurs primarily when $R < 5r^*$ (R: substrate radius) and diminishes rapidly when $R > 5r^*$. Ultrapure water that freezes at -39.1°C or below contains particles of $R < 5r^*$ while that freezes at -38.0°C or above includes particles of $R > 5r^*$. The freezing of liquid metals was discussed as well.

**2:45 PM**
**A Phase-Field Simulation Study on Heterogeneous Nucleation in Ti-Al-B Alloys**: Janin Eiken[1]; Victor Witusiewicz[1]; Ulrike Hecht[1]; *Markus Apel*[1]; [1]Access e. V.

During directional solidification of TiAl based alloys with Al contents around 45at.% the peritectic α-Ti phase usually nucleates on properitectic β-Ti dendrites. Adding boron to the melt may lead to the formation of $TiB_2$ particles. Depending on the Al and B content, $TiB_2$ appears in the melt prior to the nucleation of the α-Ti phase. In this case, $TiB_2$ particles may act as nucleation sites for α-Ti in the interdendritic melt. This scenario was observed in Bridgman experiments and being confirmed by thermodynamic calculations. Here, we present a phase-field simulation study which elucidates the effect of the Al content on the dendritic growth morphology and the effect of the boride particle size distribution on the heterogeneous nucleation of α-Ti. The simulation results agree with experimental observations and reveal the influence of the different parameters on grain refinement.

**3:05 PM**
**A Precipitate Growth Model Based on a Variational Approach**: *Qiang Du*[1]; Warren Poole[1]; Mary Wells[2]; [1]University of British Columbia; [2]University of Waterloo

In this paper, a new precipitate growth model is proposed for multi component alloys. The model is based on the approximate solution to the mass transfer diffusion equation by Ritz method (a variational approach) with a quadratic shape function. Using the model a number of case studies are investigated for binary systems with various extents of super-saturation as well as multi-component systems where the disparity in the diffusivities of the alloying elements exists. The proposed model appears to capture the precipitate growth kinetics well for these systems. It is concluded that the proposed model could replace the one widely used in a "conventional" Particle Size Distribution (PSD) model.

**3:25 PM**
**Effect of Local Stress on Nucleation and Variant Selection during Solid-State Transformation with Symmetry Reduction**: Rongpei Shi[1]; Ning Zhou[1]; *Yunzhi Wang*[1]; [1]Ohio State University

Microstructural features developed during solid-state reactions such as phase transformation, plastic deformation and fracture are often influenced by elastic strain fields associated with existing structural and compositional nonuniformities. A rigorous treatment of nucleation in solids requires a self-consistent description of the interactions between nuclei and an arbitrary pre-existing microstructure without any a priori assumptions. Taking advantage of the generality of phase-field total free energy functional and, in particular, its ability to describe arbitrary nonuniformities in the presence of long-range interactions, we study the effect of local stress and grain boundary in a polycrystalline aggregate on variant selection during nucleation and growth of a low-symmetry product phase from a high symmetry parent phase. The spatial distribution of different variants of the product phase is found to correlate strongly with the stress distribution in the polycrystalline sample. Under certain circumstances, certain variants may percolate through the entire sample, leading to micro-texturing.

**3:45 PM Break**

**4:05 PM Invited**
**Phase-Field Crystal Modeling of Nucleation, Patterning, and Early-Stage Growth in Colloidal Systems in Two and Three Dimensions**: *Laszlo Granasy*[1]; Gyorgy Tegze[1]; Gyula Toth[1]; Frigyes Podmaniczky[1]; Tamas Pusztai[1]; [1]Research Institute for Solid State Physics and Optics

Using a simple dynamical density functional approach (the phase-field crystal model), we address nucleation and morphology evolution during colloidal crystal aggregation. In single component systems, we observe a diffusion controlled growth mechanism at low supersaturations that switches to a diffusionless mechanism at high supersaturations, a behavior often seen in colloidal systems. We present a morphology map that contains transitions between compact, dendritic and fractallike structures, we investigate the change of growth anisotropy, polycrystalline aggregation, and study crystal aggregation in the presence of foreign matter represented by appropriate potentials. In 3D we use large scale-simulations of ~3 million particles to address various aspects of freezing the single component liquid into bcc, fcc and hcp structures, including dendritic solidification.

**4:30 PM**
**Nucleation and Successive Microstructure Evolution via Phasefield and Phasefield Crystal Method**: *Heike Emmerich*[1]; Ricardo Siquieri[1]; [1]RWTH Aachen

It is well known, that the mechanical material properties of a material sample after solidification are strongly tied to its microstructure structure. Nevertheless, the precise laws governing the initial stage of this structuring process, i.e. nucleation and the successive transient microstructure evolution scenarios, are still for from being fully understood. Here we show - after a thorough overview on the phasefield method and its relation to the phasefield crystal method - that the phase field method, which originally established itself to tackle the free boundary problem given by microstructure evolution, can also be employed to investigate the energetics of heterogenous nucleation in a solidifying sample. Moreover it is demonstrated, how the phasefield crystal method can shade more light in open questions regarding a quantitative formulation of nucleation statistics to thereby simulate the phase transition phenomena in solidification from nucleation to crystallization in larger domains thoroughly.

**4:50 PM**
**Ab Initio Determination of Phase-Field Parameters Needed for Scale-Bridging Studies of Nucleation and Microstructure Formation in the Ti-Fe Eutectic System**: *Martin Friak*[1]; Juergen Hubert[2]; Heike Emmerich[2]; Antje Schlieter[3]; Uta Kuehn[3]; Juergen Eckert[3]; Joerg Neugebauer[1]; [1]Max Planck Institute for Iron Research; [2]RWTH Aachen University; [3]Institute for Complex Materials at the Leibniz-Institute for Solid State and Materials Research

Ti-based alloys have been suggested for commercial applications with a great potential due to their high strength (~1000 MPa) and good corrosion resistance. The strength of these materials can be even further increased if bulk nano-structured eutectic alloys are produced. In our study we focus on the effects that local lattice strains in the nano-structured eutectics have on the thermodynamic phase stability, elastic properties, and kinetics during the initial stages of microstructure evolution. To achieve this goal a joint approach, where ab initio determined parameters are used in phase-field simulations and systematically cross-checked against experimental data, is adopted. In particular, the free energies of both stable and metastable phases, the elastic constants, local elastic strains due to lattice-constant mismatch between different inter-facing phases, and selected gamma-surface energies and kinetics energy-barriers in FeTi and beta-Ti phases obtained employing density functional calculations will be presented.

## Hume-Rothery Symposium: Configurational Thermodynamics of Materials: Session II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee, TMS: Chemistry and Physics of Materials Committee
*Program Organizers:* Chris Wolverton, Northwestern University; Mark Asta, University of California, Davis; Gerbrand Ceder, Massachusetts Institute of Technology (MIT)

Monday PM Room: 212
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* To Be Announced

**2:00 PM Invited**
**High Energy X-Ray Scattering Studies of Ordering and Phase Separation in Binary Metallic Alloys**: *Harald Reichert*[1]; [1]ESRF

Using high energy x-ray scattering we have developed experimental tools in order to reveal details in the energetics of binary alloys with very high accuracy. With this technique we have collected 2-dimensional scattering patterns for many binary alloy systems. In the time-resolved mode the technique can be used to monitor the evolution of diffuse scattering signals to Bragg-like peaks in the diffraction pattern. This allows one to follow phase transformations in-situ. Parallel to improvements in the data collection it is of equal importance to extract the maximum amount of information encoded in the diffuse scattering distribution. To this end we have developed a reciprocal space descriptiuon in a single unified model which allows one to analyze the diffuse scattering data in terms of a few physically motivated parameters. As examples, details of ordering and phase separation in Au-Ni, Ti-V, N-Pd, Cu-Au, Ni-W, and Mo-Ta alloys are presented.

**2:30 PM Invited**
**Phase Separation in Al(Sc)-Based Alloys on a Nanoscale**: *David Seidman*[1]; David Dunand[1]; [1]Norhwestern University

Phase separation is studied in Al(Sc)-based alloys utilizing atom-probe tomography and transmission electron microscopy, where the precipitating phase consists of coherent precipitates of Al3Sc(L12 structure). The effects of transition metal (TM) and/or rare earth (RE) and/or lithium additions on the temporal evolution of Al3(Sc1-xTMx or REx) is determined for a range of concentrations of TMs and/or REs. The time dependencies of the mean radius, number density, and supersaturations in the matrix and coherent precipitates are determined, during the coarsening regime, and used to extract alpha-Al/Al3(Sc1-xTMx or REx) interfacial free energies, solute diffusivities, solute solubilities in alpha-Al. The measured interfacial free energies are compared with first-principles calculations, using the Vienna ab initio simulation package (VASP), wherever possible. The relevance of this research to the use of these Al(Sc) based alloys for high temperature structural applications is also discussed. Research supported by DOE.

**3:00 PM**
**First-Principles Cluster Expansions for Predicting Surface Reconstructions**: Wei Chen[1]; *Chris Wolverton*[1]; William Schneider[2]; [1]Northwestern University; [2]University of Notre Dame

The cluster expansion (CE), pioneered by the work of Prof. de Fontaine, has become a remarkably ubiquitous and useful tool for studying problems involving states of configurational order/disorder. Here, we use a CE approach to study the "missing-row" reconstructions of (110) surfaces of transition metals. It is well-known that Au and Pt undergo a (1×2) missing-row reconstruction, while clean surfaces of Cu and Ag do not. We have used density functional calculations with a CE to study (110) surfaces as a 2D system of metal atoms and vacancies. The CE correctly demonstrates the (1×2) missing-row structure is stable for the (110) surface of Au and Pt, but not for Cu and Ag. The finite temperature properties of the missing-row surfaces were also studied by a CE+Monte Carlo approach, and we find transition temperatures, step energy anisotropy, and equilibrium shapes for the reconstructed surface in good agreement with experimental results.

**3:20 PM**
**Effects of Temperature and Chemical Order on Phonons in Fe-V Alloys**: *Jorge Munoz*[1]; Matthew Lucas[2]; Brent Fultz[1]; [1]California Institute of Technology; [2]Oak Ridge National Laboratory

Inelastic neutron scattering spectra were measured from 300 to 773 K on B2 ordered FeV, and on A2 disordered solid solutions of bcc Fe-V alloys of different compositions. A substantial softening of the phonons upon ordering was observed, so a higher vibrational entropy stabilizes the ordered phase. Generalized phonon density of states (GDOS) curves were obtained from these data and a general cluster expansion was employed to extract interaction generalized phonon DOS (IGDOS) functions from the disordered alloys. The long-range order parameter was obtained at each temperature. The reconstruction of the ordered FeV GDOS from the IGDOS functions is in qualitative agreement with the measurements, and predicts a change in vibrational entropy with ordering of about +0.2 kB/atom.

**3:40 PM Break**

**4:10 PM Invited**
**Modeling Ni-C Alloys to Study the Growth of Carbon Nanotubes and Graphene Sheets**: *François Ducastelle*[1]; Hakim Amara[1]; Christophe Bichara[2]; [1]LEM CNRS-ONERA; [2]CINaM-CNRS

We have developed a tight binding model for Ni-C alloys to study the catalytic nucleation and growth of graphene sheets or of single wall carbon nanotubes (1). The model describes the total energy of the system as a sum of atomic contributions, and is coupled to Monte Carlo simulations in different thermodynamic ensembles. Using grand canonical conditions, we calculate the adsorption isotherms and the corresponding configurations of C atoms on Ni surfaces as well as on crystalline or amorphous Ni clusters. At low concentration carbons atoms interact strongly with nickel. At higher concentration carbon segregates and self-organize to form sp2 graphitic structures which no longer interact with nickel atoms. This accounts for the catalytic properties of Ni but also of Fe and Co in the formation of these structures.(1) H. Amara, C. Bichara and F. Ducastelle, Phys. Rev. B 79, 014109 (2009).

**4:40 PM**
**Equilibria among $R_N Coin_{2+3n}$ Phases (R= La, Ce, Dy) Having $Ho_N Coga_{2+3n}$ Structures**: *Randal Newhouse*[1]; Gary Collins[1]; [1]Washington State University

Recent experiments showed high diffusional jump frequencies of Cd probes on the In-sublattice in $RIn_3$ phases of $L1_2$ structure (R= rare-earth) [Phys. Rev. Lett. 92, 225901 (2004); 102, 155901 (2009)]. Jump frequencies were determined from measurements of nuclear quadrupolar relaxation using perturbed angular correlation spectroscopy. Here, related ternary phases $R_n CoIn_{2+3n}$ (R= La,Ce,Dy) were studied that are formed by periodic replacement of mixed In-R layers with layers of transition-metal atoms (prototypes $Ho_n CoGa_{2+3n}$). Hyperfine interaction signals were observed that are consistent with Cd probes on In-sites in either type n=1 or n=2 phases or in a disordered mixture of type n=1 and 2 layers. Jumping was detected only at much higher temperatures than in $L1_2$ phases. The evolution of signals during annealing and also up to temperatures where the phases disappear will be presented and discussed.

**5:00 PM**
**First Principles Shape Memory Alloy Design: Ni-Ti-X (Pt, Pd) Ternary Systems**: *Nicholas Hatcher*[1]; Oleg Kontsevoi[1]; Arthur Freeman[1]; [1]Northwestern University

We investigate ternary additions on the martensitic behavior of NiTi by applying ab initio calculations using the highly precise FLAPW method to the Ni-Ti-X(Pt, Pd; 0-30%) system. We determine ternary site preferences, pair interaction energies, and the energy hierarchy among the phases, finding that Pd and Pt atoms replace Ni and decorate the lattice at second and third nearest neighbors from one another, respectively. We calculate the cleavage and planar generalized stacking fault energetics of the {001}, {011}, and {112} shear planes to determine shear and brittle/ductile behavior and identify a high resistance to {001} shear. Detailed elastic properties are calculated and analyzed in connection with the martensitic transformation, and we show that the C' elastic constant becomes unstable with increased alloying. Finally, we explain the effect of these additions on the NiTi transformation path. These results provide further inputs for multiscale approaches to SMA design. *Supported by AFOSR (FA9550-07-1-0174)

**5:20 PM**
**Gamma-Gamma' Interfacial Free Energy through In-Silico Nucleation Experiments**: *Stefano Angioletti-Uberti*[1]; Mark Asta[2]; Christopher Woodward[3]; Axel van de Walle[4]; Peter Lee[1]; Mike Finnis[1]; [1]Imperial College London; [2]University of California Davis; [3]Air Force Research Laboratory; [4]California Institute of Technology

By modeling the energetics of the system through a cluster expansion formalism, we perform in-silico nucleation experiments in a Ni-Al alloy at the gamma-gamma+gamma' phase boundary. From these simulations we obtain the probability of formation for clusters of different sizes and thus their free energy. We interpret our results using classical nucleation theory, and from these assumptions we derive the gamma/gamma' interfacial free energy. The results compare well with separate calculations of the interfacial free energy for planar interfaces in the same system, based on the same cluster expansion. Additionally, we obtain qualitative and quantitative information on the size dependence of the interfacial free energy, which is found to be an important correction to include when modelling the energetics of nanosized clusters

**5:40 PM**
**Multi-Scale Modeling of Martensite Formation in Fe-Based Solid Solutions**: *Alexander Udyansky*[1]; Johann von Pezold[1]; Jörg Neugebauer[1]; [1]Max-Planck-Institut für Eisenforschung GmbH

Martensitic phases refer to tetragonal states of interstitial Fe-based solid solutions containing carbon, nitrogen or oxygen. We study such dilute phases by combining atomistic modeling with the microscopic elasticity theory (MET), which allows us to account for long-ranged elastic interactions between impurities. The short-range chemical interactions, as well as the parameters entering the MET are obtained atomistically employing density functional theory (DFT). This approach allowed us to compute phase diagrams and provided a direct insight into the stability and formation of martensite: specifically, tetragonal states are predicted to be preferred also at low C concentrations due to a thermodynamically driven orientational ordering of carbon interstitials. The critical concentration for the cubic-tetragonal transition at room temperature is found in excellent agreement with experimental data. This methodology allows to study long-range elastic interactions even with rather modest supercell sizes making it an ideal tool in combination with modern DFT approaches.

## International Symposium on High-Temperature Metallurgical Processing: Ceramics and Intermetallics

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
*Program Organizers:* Jaroslaw Drelich, Michigan Technological University; Jiann-Yang Hwang, Michigan Technological University; Tao Jiang, Central South University; Jerome Downey, Montana Tech

Monday PM Room: 619
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Jerome Downey, Montana Tech

**2:00 PM Keynote**
**Process Development: Challenges and Driving Forces for Change**: *Karl Forwald*[1]; [1]Elkem AS Solar

The set-back in the global economy has left us in a stand-still in a large part of the high temperature processing industry. For many of us this may also be a time for long term thinking: What are the challenges for high temperature processing of materials? Do we need new materials and processes? Can we identify some main driving forces for new development? Who is in a position to decide on what development directions to follow: Shall we lean on international declarations, government regulations or board room and management decisions? Or will "the market" eventually give us the feed-back we need in order to set the course?These and other questions will be discussed from the authors point of view.

**2:40 PM**
**Glass Coating for Iron-Based Powder Metallurgy Components**: *Adele Garkida*[1]; Jiann-Yang Hwang[2]; Xiaodi Huang[2]; Allison Hein[2]; Zhiwei Peng[2]; [1]Ahmadu Bello University; [2]Michigan Technological University

Die-pressed Fe-1wt% C powder metallurgy compacts having theoretical densities ranging from 75%-85% were used as coating substrates. The glass slurries used for coating was prepared by stirring pulverized acustar untempered glass into butanol. Copper oxide, iron oxide and cobalt oxide were evaluated as transition metal oxide additives to promote adherence of the glass to the ferrous substrate. Likewise Borax and Boron oxide were evaluated as boron additives. Initially samples were coated by brushing the slurry onto the compact, later samples were coated by spraying the slurry onto the compact. These were sintered in vacuum at 1120ºC with I hour holding time. The coating composition adopted consisted of 70 wt% pulverized glass, 25 wt% boron oxide and 5 wt% cobalt oxide and its potential durability was characterized using an optical metallography. This can be employed as a wear-resistant coating for iron-based PM components used in automotive applications.

**3:00 PM**
**Thermodynamic Measurement of Al2O3-B2O3 System by Double Knudsen Cell Mass Spectrometry**: *Takashi Nagai*[1]; Masafumi Maeda[1]; [1]The University of Tokyo

Thermodynamic information on Al2O3-B2O3 system is important for various high temperature processes in ceramics, glass, and metallurgical industries, but the information is extremely limited. Recently, a process to produce solar cell grade Si from metallurgical grade Si by slag refreshing has been studied. In previous study, the possibility of the removal of B from Si using flux containing Al2O3 was reported although B is one of the most difficult elements to remove from Si.In this study, vapor pressure of B2O3 in equilibrium with Al2O3-B2O3 compounds or melts in Al2O3-B2O3 system were measured by double Knudsen cell mass spectrometry. The Gibbs energy of formation of Al18B4O33 was estimated from the vapor pressure in equilibrium with a mixture of Al18B4O33 and Al2O3 at 1573 to 1673 K. And activities of B2O3 in two phase region Al18B4O33 and B2O3-rich liquid, and Al2O3-B2O3 melts were obtained at 1373 to 1423 K.

**3:20 PM**
**Bonite - A New Raw Material Alternative for Silica-Free High Strength Aluminum Metal Refractories**: *Dale Zacherl*[1]; Andreas Buhr[2]; Dagmar Schmidtmeier[2]; Robert McConnell[1]; [1]Almatis, Inc; [2]Almatis GmbH

Refractory castables in the aluminum industry as well as chemical and petrochemical industries typically see much lower temperatures than in the steel industry. The maximum temperature is very often in the range of 800 – 1200 °C. Nevertheless these applications can also be very demanding regarding mechanical strength, abrasion resistance, and chemical stability. Mechanical strength of refractory linings is of particular interest at the intermediate temperatures, which do not providesufficient energy for strong sintering reactions. This paper discusses raw material concepts for high purity silica-free castables for demanding aluminum or petrochemical applications. Bonite, a new dense calcium hexaluminate (CA6) refractory aggregate is introduced for low wettability, high temperature stability and low thermal conductivity of aluminum refractories.

**3:40 PM Break**

**3:55 PM**
**On Line Monitoring and Process Parameters Estimation of Multiple Passes Laser Phase Transformation Hardening by Using High-Power Direct Diode Laser**: *Soundarapandian Santhanakrishnan*[1]; Radovan Kovacevic[1]; [1]Southern Methodist University

A component treated by a laser beam especially during multiple passes needs on line monitoring and process parameter optimization to achieve the required surface properties and mechanical properties. In this work, a coupled non-contact monitoring and estimation technique is developed. This technique is based on a laser-assisted infrared pyrometer and an infrared camera to monitor the laser treated surface temperature in real time. A laser-assisted infrared pyrometer is used to measure the emissivity and surface temperature of the laser treated zone. The measured emissivity is used as an input required for the infrared camera. An infrared camera is used to measure the temperature distributions across the laser treated surface in real time. A polynomial fitting method is used to obtain a relationship between the process parameters and the surface hardness for multiple passes laser hardening.

**4:15 PM**
**Phase Transformation of Andalusite-Mullite and Its Fiber Reinforcement to Refractory Ceramics**: Bowen Li[1]; *Jiann-Yang Hwang*[1]; Wayne Bell[1]; [1]Michigan Technological University

Andalusite (Al2SiO5) is a widely distributed industrial mineral. When heated to 1200-1400°C, andalusite will be rapidly decomposed into mullite and vitreous silica, a phase transformation process usually referred as mullitization. The phase transformation from andalusite to mullite can be different from a common crystal transition from a parent crystal to a product crystal. An individual andalusite crystal can be divided into thousands of fiber-shaped crystals of mullite. The finer the particle of andalusite is, the smaller the mullite fibers are produced. In this study, it was found that when fine particles of andalusite were dispersed among refractory aggregates in green body, cross-linked networks of mullite fibers were produced in sintered refractory bricks. This can increase the mechanical strength of refractory ceramics significantly.

**4:35 PM**
**Thermodynamic Measurement of Rare Earth Metal Systems by Knudsen Cell Mass Spectrometry**: *Sho Shirai*[1]; Takashi Nagai[1]; Masafumi Maeda[1]; [1]Institute of Industrial Science, University of Tokyo

RE metals are used as various functional materials, such as Nd-Fe-B magnet, electrode materials of batteries, hydrogen storing alloy and so on. Thermodynamic properties of RE metal system are very important to produce and recycle the materials. The properties can not been, however, estimated easily by traditional methods, because chemical affinities of RE metal with oxygen are very strong. Using double Knudsen cell mass spectrometry, vapor pressure of RE metals in equilibrium with the alloys can be measured without effect of oxygen, because this measurement is run under high vacuum condition. In this study, we investigated thermodynamic properties of Nd-Fe systems by this method. Vapor pressure of Nd in Nd-Fe was measured and the activities of Nd and Fe in Nd-Fe at 1373 to 1523K were revealed in complete composition domain. Gibbs free energy of formation of Nd2Fe17, intermetallic compound of Nd-Fe, was estimated.

**4:55 PM**
**A Technique to Measure Heat of Reaction in TiB2 Reinforced Intermetallic Matrix Composites**: *Andrew Baker*[1]; S.L. Kampe[1]; Tony Zahrah[2]; [1]Michigan Tech; [2]Matsys, Inc

Reaction synthesis is a technique to produce ceramics, intermetallics, and in-situ composite materials. The technique, and the resulting microstructures of the product, rely upon a large enthalpy reduction during the reaction, and also the relatively high temperatures that are characteristic of the process. This research focuses on a technique to measure the heat of reaction of a series of TiB2-reinforced titanium aluminide composites. To overcome the kinetic constraints and achieve ignition in these formulations, a boron (B)/potassium nitrate (KNO3) initiation aid was incorporated within the blended reactant compact and placed in an unmodified bomb calorimeter. A design of experiments matrix was created to determine the enthalpy of reaction and the associated sensitivity to a variety of process variables including volume relative proportions of product phases, reactant particle size, B/KNO3 fraction, gas environment and initial bomb pressure. This research was sponsored by the Office of Naval Research under grant N0014-07-1-1055.

**5:15 PM**
**Microwave Synthesis of Nano-Boron Carbide Powder**: *Liang Hu*[1]; Huimin Lu[1]; Yi Liu[1]; [1]Beihang University

Boron Carbide (B4C) is a light weight material and has many fine performances, such as high melt point, high hardness, low density, and good abilities of neutron absorption and anti-chemical corrosion, so widely applied in some domains such as refractory, project ceramics, nuclear industry, and astronavigation and so on. This paper describes the synthesis process of the nano-boron carbide powder with the microwave carbothermal reduction method. The experiments showed that the microwave synthesis temperatures of nano-boron carbide were controlled in the 1600~1700ºC range and the time in 60min. The energy consumption of this method has been reduced by about 70% as compared with traditional heating methods. The prepared powder has high purity and particle size in 30~90nm range, which is a very good raw material for sintering boron carbide products with high performances. In the meantime, the thermodynamics and kinetics of the microwave synthesizing nano-boron carbide were systematically studied.

## Jim Evans Honorary Symposium: Cast Shop Aluminum Production Joint Session: Flow and Solidification Phenomena in Nonferrous Casting

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division
*Program Organizers:* Ben Li, University of Michigan; Brian G. Thomas, University of Illinois at Urbana-Champaign; Lifeng Zhang, Missouri University of Science and Technology; Fiona Doyle, University of California, Berkeley; Andrew Campbell, WorleyParsons

Monday PM Room: 620
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Lifeng Zhang, Missouri University of Science and Technology

**2:00 PM Introductory Comments**

**2:10 PM**
**Coupled Multi-Physics Modeling of Continuous Casting of Steel and DC Casting of Aluminum**: *Philippe Thevoz*[1]; Olivier Ludwig[1]; Marco Aloe[1]; [1]Calcom ESI

As the product quality requirements are ever increasing and the gains in productivity are a must to sustain the competition, numerical modeling is now an essential tool to reach these targets. Due to the complexity of continuous and DC casting processes, a coupled multi-physics approach is required to capture all the important phenomena. This paper will present a fully coupled thermal-flow-transport-stress 3D modeling of continuous casting processes, with illustrations on continuous casting of steel and DC casting of aluminum. The influence of fluid flow, using different nozzle designs, on the first solidified shell will be investigated, together with the stress build-up and the air gap formation. Such coupling is achieved using a novel technique recently developed based upon the Mixed Lagrangian-Eulerian method (MiLE) implemented in the Finite Element

software ProCAST.Finally, results of microporosity, hot tearing, grain structure and solid state transformations, based upon further multi-physics coupling, will be presented.

**2:35 PM**

**Metal Flow and Heat Transfer in Wagstaff® Rapidfill™ Metal Distribution Systems for Billet Dc Casting**: *Bin Zhang*[1]; [1]Wagstaff Inc

Modelling of fluid flow and heat transfer in Wagstaff® Classic and RapidFill™ metal distribution systems for billet DC casting were conducted during the start-up and casting phases. Results clearly show the effect of different designs in the two systems on metal fill/flow and heat loss. The Wagstaff® RapidFill™ system allows metal to fill casting positions almost simultaneously. Total metal fill time is greatly reduced compared to the Classic system. The hold time difference between casting positions is also significantly decreased in the RapidFill™ system. It is shown that the metal heat loss in a RapidFill™ system is ~3-10°C less than that of a Classic system during the start-up phase and ~ 3°C less when casting approaches steady state. Details on metal flow and temperature variation during the start-up phase are presented for comparison at strategic locations. Results were verified through field measurements.

**3:00 PM**

**Thermal-Fluid-Compositional Model of Electron Beam Casting of Ti-6Al-4V**: Riley Shuster[1]; Daan Maijer[1]; *Steven Cockcroft*[1]; Tao Meng[1]; Denis Favez[1]; David Tripp[2]; Stephen Fox[3]; [1]The University of British Columbia; [2]TIMET Morgantown; [3]TIMET Henderson

Two of the principle concerns during the final stage of the semi-continuous ingot casting process associated with Electron Beam Cold Hearth Remelting/ Melting of titanium alloys are the formation of shrinkage voids and the evaporation of volatile species. The presence of voids or loss of chemistry control requires ingot cropping before further downstream processing. Raising the shrinkage void location can be achieved through hot topping, where the top surface is heated after steady state casting is complete. However, hot topping increases selective evaporation. A mathematical model of the final transient stage has been developed to assist in reducing the associated production losses during casting of Ti-6Al-4V ingots and slabs. The model, developed within a commercial software platform, solves the coupled thermal-fluid flow problem including solute conservation and evaporation. Plant trial measurements including sump depth, pool profile, and compositional analysis are used to validate the model predictions under various hot topping conditions.

**3:25 PM**

**The Effect of SF6 on the Surface Tension of AZ91D Magnesium Alloy**: Steven Roach[1]; *Hani Henein*[2]; [1]Vale INCO Ltd.; [2]University of Alberta

A new draining crucible technique was used to measure the effect of $SF_6$ on the surface tension of magnesium. This technique is based on the relationship between the height of metal in a crucible and the outgoing flowrate. This relationship is obtained from a model where the surface tension, viscosity and density of a melt are simultaneously determined. Experiments performed with molten magnesium at temperatures from 923 to 1173K indicate under Argon the surface tension (N/m) and density (kg/m$^3$) are [0.62 – 2.13 e-4 (T-$T_m$)] and [1651 – 0.16 (T-$T_m$)], which is within 6.5pct. and 2.5pct., respectively, of values reported in the literature. The viscosity (Nsm$^{-2}$) has been determined to range from 8.46e-4 at 8K superheat to 2.93e-4 at 250K superheat. $SF_6$ reduces the surface tension (N/m) of AZ91D to [0.491 – 7.44e-4 (T-$T_m$)].

**3:50 PM Break**

**4:05 PM**

**Application of Computational Fluid Flow and Experimentations to Improve Horizontal Casting Process Performance at Rio Tinto Alcan**: *André Larouche*[1]; [1]Rio Tinto Alcan

Over the last decade computational fluid dynamic has been increasingly used in the aluminum industry in order to understand, analyze and improve various processes in reduction, raw material and casting. Improved computer performances and user friendliness of CFD software has allowed reducing design cycle time, hence improving applicability of CFD in an industrial context. This paper describes the successful application of numerical modeling capabilities in conjunction with experimental approaches to understand and improve the horizontal direct chilled (HDC) casting of T-ingot. Several examples of fluid flow and thermal analysis such as metal feeding, casting speed increase, and alloy transfer are brought and described to highlight the contribution of CFD to the development and successful implementation of practical solutions in plants.

**4:30 PM**

**DC Casting of Aluminum Alloys — Importance of Mold Boundary Conditions**: *Amir Baserinia*[1]; Harry Ng[1]; David Weckman[1]; Mary Wells[1]; [1]University of Waterloo

Accurate thermofluids models of DC casting of aluminum ingots must be capable of solving the heat transfer equation within the ingot with realistic external thermal boundary conditions. These boundary conditions are typically separated into two zones: primary cooling, which occurs inside the water-cooled mold, and secondary cooling, where a film of water directly contacts the ingot surface. In this study, a simple yet comprehensive model is developed for the primary cooling region of the steady-state DC casting process. First the mold and its water-cooling were modeled using a commercial CFD package and the heat transfer coefficient was determined. A simple density-based model was then used to predict the added effect on the primary cooling of ingot shrinkage and the formation of a gap between the ingot and mold. Simulations using this model suggest that remelting of the primary shell and liquid metal exudation can be predicted.

**4:55 PM**

**Measurement of As-Cast Residual Stresses in an Aluminium Alloy AA6063 Billet Using Neutron Diffraction**: *Jean-Marie Drezet*[1]; Alexander Evans[2]; Christophe Jaquerod[3]; André Phillion[1]; [1]Ecole Polytechnique Federale Lausanne; [2]Paul Scherrer Institut, Villigen; [3]Alcan Aluminium Valais, Sierre

Machining and sawing Aluminium DC cast products without a stress relief treatment can lead to uncontrolled distortion, crack formation, and significant safety concerns due to the development of thermally-induced residual stresses within the casting. Numerical models have been developed to compute these residual stresses and only validated against measured surface distortions.In the present contribution, the variation in residual strains and stresses as a function of radius has been measured using neutron diffraction in an AA6053 grain-refined cylindrical billet. A thermomechanical finite element model was used to determine the minimum section-length which can be sawed from the billet without significantly relaxing the residual stresses while conforming to the requirements of the neutron diffractometer. The results of these measurements, particularly the depth at which the axial and hoop stresses change sign, can be used for validation of the numerical models and provide insight into the development of residual stresses within castings.

## Magnesium Technology 2010: ICME I (Integrated Computational Materials Engineering)

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
*Program Organizers:* Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Monday PM Room: 612
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* John Allison, Ford Motor Company; Alan Luo, General Motors Corporation

**2:00 PM**

**Integrated Computational Materials Engineering (ICME) for Magnesium: An International Pilot Project**: *John Allison*[1]; Baicheng Liu[2]; Kevin Boyle[3]; Lou Hector[4]; Robert McCune[5]; [1]Ford Motor Company; [2]Tsinghua University; [3]CanMET Materials Technology Laboratory; [4]General Motors R&D Center; [5]Robert C. McCune and Associates

This talk will provide an overview and progress report for an international collaborative project developing an ICME infrastructure for magnesium for use in automotive body applications. Quantitative processing-microstructure-property relationships are being developed for extruded magnesium alloys, sheet-formed magnesium alloys and high pressure die cast magnesium alloys. These relationships are captured in computational models which are then linked with manufacturing process simulation and used to provide constitutive models for component performance analysis. The long term goal is to capture

this information in efficient computational models and in a web-centered knowledge base. The work is being conducted at leading universities, national labs and industrial research facilities in the US, China and Canada. This project is sponsored by the U.S. Department of Energy, the U.S. Automotive Materials Partnership (USAMP), Chinese Ministry of Science and Technology (China) and Natural Resources Canada (Canada).

**2:20 PM Keynote**

**Thermodynamic and Elastic Properties of La-X (X=Al,Mg) Intermetallic Compounds from First Principles Calculations**: *Louis Hector Jr*[1]; Jan Wrobel[2]; Krzysztof Kurzydlowski[2]; [1]GM R&D Center; [2]Warsaw University of Technology

The La-X (X=Al,Mg) intermetallic compounds are a scientifically interesting class of materials with broad technological relevance. Some occur as precipitates in certain magnesium alloys while others are considered to be potential hydrogen storage materials. Using first principles density functional theory, we computed thermodynamic and elastic properties of LaAl, LaAl2, LaAl3, LaAl4, La3Al11, La3Al, La16Al13, LaMg, LaMg2, LaMg3, La2Mg17 and La5Mg41. Specifically, lattice parameters, thermodynamic functions from phonon calculations based upon the direct method for lattice dynamics, and enthalpies of formation at 0 and 298K, were calculated for each compound with the generalized gradient approximation. Components of the elasticity tensor, Cij, were computed with a least squares fitting method using a set of sequential strains to improve the accuracy of each calculation. Polycrystalline moduli based on the Hill criteria were calculated from the Cij. A new La3Al structure was identified from a soft mode in the phonon dispersion curves.

**2:50 PM**

**Numerical Simulation of Direct Extrusion of Magnesium Alloys**: *Wojciech Misiolek*[1]; Luigi DePari[1]; [1]Lehigh University

An existing flow stress model was examined with as-cast AZ31 compression test data from literature for a range of temperatures (250-450 deg C) and strain rates (0.001-30 s-1) to evaluate it's applicability to direct extrusion of AZ31. The model was successful in predicting the hardening region of the flow stress curve but was unable to simulate the sizable recovery portion of the flow stress curve that Mg alloys tend to exhibit before failure. In order to correct for this shortcoming, an empirical recovery expression was developed for the model with the same range of temperatures and strain rates. This modified flow stress model has then been implemented into the finite element software package DEFORMTM 3-D to predict the state variables of hot-direct extrusion of Mg alloy automobile structural components manufactured with an port-hole extrusion die.

**3:10 PM Keynote**

**On Modeling the Extrusion Process of Magnesium Alloys**: *Esteban Marin*[1]; Stephen Horstemeyer[1]; Clemence Bouvard[1]; Douglas Bammann[1]; Haitham El Kadiri[1]; Paul Wang[1]; [1]Mississippi State University

The work presents the lab-scale extrusion experiments being performed on magnesium alloy AZ61, the numerical modeling of the process using an Eulerian / ALE approach, and the constitutive modeling framework characterizing the material's mechanical response. The lab-scale experiments have been planned to learn about the extrusion process (material microstructure, material flow, processing parameters) as well as to be used as a benchmark case to validate the modeling and simulation tools. The experiments have been modeled using HyperXtrude, an Eulerian finite element code suitable to represent deformation processes where severe plastic deformation occurs, as in the case of extrusion. Two particular material modeling frameworks have been employed to model the process: a macroscopic internal state variable (ISV) model and a crystal plasticity model. Predictions from the numerical tools (finite element code and material models) are compared with experiments to show the predictive capability of the modeling approach.

**3:40 PM Break**

**4:00 PM**

**Transmutation and Accommodation Effects by Glide Twinning**: *Andrew Oppedal*[1]; Haitham El Kadiri[1]; [1]Mississippi State University

Magnesium is being intensively integrated in large mechanical structures for energy savings due to its favorable strength / stiffness to weight ratio. This industrial renaissance is reviving attention to the complex fundamentals of deformation twinning in metallic double-lattice structures. Significant advances were made to integrate deformation twinning in polycrystal plasticity simulations, but constitutive models are still unable to capture significant hardening mechanisms unique to deformation twinning indentified and studied in the late 1950s through the 1970s. This spurs the need to recall and analyze major mechanisms relevant to hardening in double lattice metals. In this work, we emphasize the effect of parent dislocation "transmutation" upon their incorporation within a twin, and effects related to slip and kinking accommodations of twinning deformation. A model routine is formulated for these mechanisms within the framework of a dislocation-density based constitutive model put forward by Beyerlein and Tomé (2008).

**4:20 PM**

**Plasticity in a Rod-Textured Extruded Mg AM30 Alloy**: *Q. Ma*[1]; H. El Kadiri[1]; [1]Mississippi State University

When a sharp-textured hcp metal is loaded under an orientation whereupon twinning or detwinning is profuse, the stress-strain curve is sigmoidal with a critical inflection point. Authors unarguably attributed the dramatic stress increase in the lower-bound vicinity of the inflection point to a combined effect of i) Hall-Petch mechanism correlated to grain refinement by twinning, and ii) twinning-induced reorientation requiring activation of hard slip modes. Accordingly, these two mechanisms cast the driving approach for determination of appropriate strain hardening model parameters in polycrystal plasticity simulations, namely the Visco-Plastic Self Consistent (VPSC) model developed by Molinari (1987) and implemented by Lebensohn and Tome (1993). Predominantly, the Hall-Petch extent needed to fit the sigmoidal curve corresponded to a latent hardening rate of twinning on other active slip modes 400% to 3500% higher than the slip-slip self-hardening rates. In this paper, we experimentally and numerically demonstrate that these two mechanisms alone cannot consistently explain the effect of profuse twinning on the hardening rate in textured hcp metals. We argue based on adopting various mechanistic approaches in hardening model correlations. These correlations are motivated by available literature and carefully analyzed twinning-induced texture evolution upon strain. We mainly focus on the extended formulation of Voce hardening model most commonly used in the polycrystal VPSC simulations. The hcp material is exemplified by a rod-textured AM30 alloy whereupon twinning readily impinges the entire structure under a suitable compression orientation. We suggest the parent dislocation transmutation mechanism upon incorporation within the twin lamella as to play a major role in the increasing hardening rate induced by profuse twinning in the lower-bound range of the inflection point.

**4:40 PM**

**Effect of Grain-Matrix Interaction Stiffness on Slip System Hardening Parameters of a Viscoplastic Self-Consistent Polycrystal Model**: *Babak Raeisinia*[1]; Sean Agnew[1]; [1]University of Virginia

The flow curves and Lankford coefficients of a rolled AZ31 magnesium alloy deformed under different uniaxial strain paths are simulated using a viscoplastic self-consistent polycrystal model. The experiments are modeled while the rigidity of the interaction between the individual grains and the surrounding polycrystalline aggregate is systematically varied from a stiff (nearly iso-strain) to a compliant (nearly iso-stress) interaction. The simulated critical resolved shear stress (CRSS) for the prismatic slip system is insensitive to the type of interaction used, consistent with the notion that the flow stress in magnesium alloys is controlled by the strength of the prismatic slip mode. This is in contrast to the trend observed for the basal slip system where the model CRSS rises appreciably as more compliant interactions are used. Simulated CRSS ratios of non-basal slip to basal slip are closest to those reported in the literature for single-crystals in the stiff interaction regime.

**5:00 PM**

**Extracting Post-Uniform Constitutive Behavior from High Temperature Tensile Test Data**: *Cyrus Dreyer*[1]; Louis Hector[1]; Sean Agnew[1]; [1]University of Virgina

Constitutive model development for the simulation of hot forming operations is traditionally conducted using hot compression or torsion data, yet neither is possible for thin sheet metal due to sample buckling. It is widely recognized to be difficult to extract constitutive behavior from tensile test data at strain levels adequate for simulating metal forming. However, at the temperatures required for warm forming of magnesium alloys, the strain rate sensitivity can be sufficient to delay the onset of catastrophic localized necking instability to significant strain levels. This permits the extraction of true stress-true strain data even in

cases where modest strain softening, e.g., due to dynamic recrystallization has occurred. A protocol involving on-sample measurements of the strain within a localized region and invoking the Marciniak-Kucinski approach for describing the development of plastic instability is used to constrain the parameters of an internal state variable model due to Bammann, Chelsea, and Johnson.

**5:20 PM**

**Strain Field Measurement during Bending of Extruded Magnesium Alloys**: *Adi Ben-Artzy*[1]; Louis Hector[2]; Paul Krajewski[2]; [1]Rotem Ind. ; [2]GM

Tensile and compressive deformation of magnesium alloys AZ31, AZ61, AM50 and ZM21 were investigated with a digital image correlation (DIC) algorithm. Three and four-point bending tests of rectangular bars at selected orientations relative to the extrusion direction were conducted in a miniature screw-driven stage. Four-point bending was designed to give a plane strain state between the fixture pivots. Displacement and load data were acquired with external controller electronics and custom data acquisition software. High resolution digital images of one surface of each bar were recorded during deformation with a variable framing rate high speed digital camera. Post-processing of the image data with the DIC algorithm resulted in strain contour maps from which the evolution of tensile and compressive strains in both bending schemes was quantified. Bending strains were found to be sensitive to Mg alloy composition and orientation, and tensile bending data was in good agreement with uniaxial tensile tests.

**5:40 PM**

**Cyberinfrastructure for Integrated Computational Material Engineering**: *Tomasz Haupt*[1]; [1]Mississippi State University

The goal of the cyberinfrastructure for ICME is to exploit the recent transformative research in material science involving multiscale physics-based predictive modeling, multiscale experiments and design. More specifically, the creation of the cyberinfrastructure will result in the development of "community of practice" portal that allows development and integration of multiscale physics-based materials models for selected properties and processes. This presentation demonstrates the use of the modern information infrastructure based on AJAX-based rich user interfaces, Service Oriented Architecture (SOA), Web Services and Grid computing streamlining of the process of gathering experimental results, and deriving the material properties (using online model calibration tools) for a particular material model (e.g., Damage Fit or Multistage Fatigue Fit) and employing the material model in finite element analysis in the process of building validated metamodels and design optimizations to support the Magnesium Front-End Three Nation Pilot Program.

## Magnesium Technology 2010: Primary Production and Flammability Issues

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
*Program Organizers:* Sean Agnew, University of Virginia; Eric Nyberg, Pacific Northwest National Laboratory; Wim Sillekens, TNO; Neale Neelameggham, US Magnesium LLC

Monday PM Room: 613
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Neale Neelameggham, US Magnesium LLC; Adam Powell, Opennovation

**2:00 PM**

**The Magnesium Industry Today: A Global Perspective**: *Greg Patzer*[1]; [1]International Magnesium Association

This document/presentation will present the current situation in the global magnesium industry in 2009 and Ytd. 2010. World demand for magnesium continues to remain subdued, succumbing to the global recessionary period as well as intra-industry pressures. The industry has seen marked changes in the sources of supply for primary and alloyed magnesium in recent years. The current state and trends in the production side of the magnesium industry will be examined. The immediate past industry changes will be assessed as to the impact that is being realized today, while also examining those developing factors, which will impact the industry's future. Particular attention will be focused upon current production, capacity utilization and potential capacity coming on line. Historical pricing as compared to production cost factors will be examined. Factors affecting future demand will be discussed as well as potential trends.

**2:20 PM**

**Magnesium: Bridging Diverse Metal Markets**: *Susan Slade*[1]; [1]US Magnesium LLC

Magnesium consumption crosses a multitude of metal industries as both a structural material and a chemical unit. Magnesium's superior strength to weight ratio and ductility enhances aluminum alloys and provides lightweighting opportunities in automotive, electronic and aerospace components. Its diverse chemical properties make magnesium important in the production of low-sulfur steel and ferroalloys, as a reducing agent for transition metals and organic chemistry, in electrochemical applications and finally pyrotechnic applications for the military. Growing environmental pressures and the global economic recession impact each of these consuming industries differently. The same pressures impart a long-term bearing on the supply side of the magnesium industry as well. The future impact of the convergence of the unique pressures on both the supply and demand of magnesium will be described.

**2:40 PM**

**Development of Recyclable Mg-Based Alloys**: Nathan Reade[1]; Jerry Sokolowski[1]; Adam Gesing[2]; Carsten Blawert[3]; Daniel Fechner[3]; *Norbert Hort*[3]; [1]University of Windsor; [2]GCI; [3]GKSS

The formation of phases during solidification and their disappearance during melting of Mg alloys with recyclable composition was studied with the help of macro thermal analysis using UMSA. Solidification rates were chosen to simulate the conditions encountered in conventional commercial magnesium shape casting processes. Distribution, quantity and composition of phases in the resultant microstructure were used to understand the measured mechanical and corrosion properties of these recyclable alloys.

**3:00 PM**

**Preparation of Al-Mg Alloys from MgO by Molten Salt Electrolysis Method**: Sh Yang[1]; Fl Yang[1]; *Xianwei Hu*[2]; Zhaowen Wang[2]; Zhongning Shi[2]; Bingliang Gao[2]; [1]Jiangxi University of Science and Technology; [2]Northeastern University

Aluminum-magnesium alloys were prepared from magnesium oxide by molten salt electrolysis method. MgF2-BaF2-LiF was taken as electrolysis. Current efficiency was more than 80%, the maximum was 89.4%. Effect of temperature on electrolysis was great. The process of electrolysis was controlled together by electrochemical polarization and concentration polarization at 850º,but at 870º and 890º, the process was controlled only by electrochemical polarization. The results showed that electrolytic temperature had little effect on electrolytic process if about 7% KCl as additive was in electrolyte, and the electrolytic process was controlled by electrochemical polarization.

**3:20 PM**

**Effect of KCl on Conductivity of BaF2-LiF-MgF2 Molten Salts**: Sh Yang[1]; Fl Yang[1]; Guocheng Wang[1]; Xianwei Hu[2]; Zhaowen Wang[2]; *Zhongning Shi*[2]; Bingliang Gao[2]; [1]Jiangxi University of Science and Technology; [2]Northeastern University

Conductivity of molten salt was studied by continuously varying cell constant method. It was proved that the method was reliable and accurate by experiments. Cell impedance was measured by electrochemical instrument of a PGSTAT 30 and a BOOSTER 20A. The results were shown that effect of KCl on conductivity of molten salt was great for MgF2-BaF2-LiF electrolyte. Electrical conductivity was increased with content of KCl in electrolyte. Electrical conductivity could be increased 0.58s/cm with content of KCl in electrolyte from 0wt% to 11wt%. Also, Electrical conductivity could be increased 0.2-0.3s/cm if temperature was increased 40º. Activation energy of conductance was obtained by the experimental results.

**3:40 PM**

**Powder Metallurgy of Magnesium: Is it Feasible?**: *Paul Burke*[1]; Georges Kipouros[1]; [1]Dalhousie University

Magnesium and its alloys are attractive materials for use in automotive and aerospace applications because of the low density and good mechanical properties. However, difficulty in forming magnesium and the limited number of available commercial alloys limit their use. Powder Metallurgy (P/M) can be used to alleviate the formability problem through near-net-shape processing,

and also allows unique chemical compositions that can lead to new alloys with novel properties. A surface layer forms on Mg powders through reactions with the atmosphere, and this layer acts as a barrier to diffusion making traditional press and sinter processing problematic. Previously, attempts have been made to circumvent the issue with novel approaches to powder production, powder consolidation and sintering techniques. Discussion of the performance and applicability of these methods is provided. This work also presents an in-situ layer disruption mechanism by the use of Ca, which can utilize traditional press and sinter P/M technology.

**4:00 PM Break**

**4:20 PM**

**Fireproof Evaluation of CaO Added Mg-3Al, Mg-6Al, and Mg-9Al Mg Cast Products**: *Jin-Kyu Lee*[1]; Shae K. Kim[1]; [1]Korea Institute of Industrial Technology

Mg products are easily oxidized and burned when they are exposed to high temperature or fire by accident. The fireproof solution has been developed by adding CaO in Mg alloys. The fireproof was evaluated by three methods: quantitative DTA for small sphere specimen, furnace ignition test for burrs and machined chips, and torch ignition test for products. DTA was carried out for obtaining quantitative ignition temperature data with respect to specimen geometry and test environment; the furnace test for burr and chip ignition temperature data; and the torch test for ignition temperature data about manufactured products. This paper will discuss the results of fireproof properties of 1wt.% and 1.5wt.% CaO added Mg-3Al, Mg-6Al and Mg-9Al Mg alloys compared with other high temperature Mg alloys. Fireproof Eco-Mg alloy will be low-cost Mg alloys for airplane and train applications in terms of preventing poisonous gas generation, inhalation burn, and ignition generation.

**4:40 PM**

**Effect of Ca(OH)2 on Oxidation and Ignition Resistances of Pure Mg**: *Dong-In Jang*[1]; Shae K. Kim[1]; [1]Korea Institute of Industrial Technology

CaO added Eco-Mg alloy has the potential to maximize the environmental benefits provided by lightweight, unlimited, and recyclable Mg alloy by eliminating global warming SF6 or other protective gases as well as Be addition. It is possible to ensure the safety during manufacturing and application, especially without sacrificing process abilities and mechanical properties and increasing the cost of Mg alloy. However, only one problem of CaO is prone to moisture absorption during storage. Instead of CaO, it is attempted to use Ca(OH)2, which does not absorb moisture during storage, for Eco-Mg alloy. This paper will discuss the effect of Ca(OH)2 on oxidation and ignition resistances of pure Mg and to compare the results with them of CaO addition. Pure Mg was used instead of Mg alloys to minimize the effects of other elements.

**5:00 PM**

**Research on the Oxidation Behavior of AZ91D-Based Magnesium Alloys**: *Hongjie Luo*[1]; [1]Northeastern University

Magnesium alloys are easily to be ignited and oxidized during heating and melting, especially in high temperature, so their applications are limited by these problems. In this study, some anti-ignition magnesium alloys were prepared by adding Ca, Ce mischmetal and Sb into molten AZ91D magnesium alloy and their oxidation behavior at high temperature were also measured. Meanwhile, the phase compositions of oxidation film were analyzed and the microstructure of these alloys was observed. The results showed that the antioxidant capacity of AZ91D-based alloys has been improved significantly after the elements of Ca, Ce mischmetal and Sb were added into them. The phenomenon of oxidation weight increase on the alloy's surface happens nearly at 700º when Ca, Ce mischmetal and Sb content are 1wt. %, 1 wt. % and 0.4 wt.%, respectively. The curves of oxidation weight increase of the above alloy are coincident closely when heated at 500º or 600º.

**5:20 PM**

**Low-Cost Zero-Emission Primary Magnesium Production by Solid Oxide Membrane (SOM) Electrolysis**: *Adam Powell*[1]; Uday Pal[2]; Steve Derezinski[1]; [1]Metal Oxygen Separation Technologies, Inc.; [2]Boston University

Solid Oxide Membrane (SOM) Electrolysis is a new low-cost process for direct extraction of magnesium oxide to pure magnesium and oxygen gas. It is a high-temperature molten salt process with a solid oxide ion-conducting membrane separating the liquid electrolyte from the anode. This both protects the anode from the molten salt, significantly expanding the range of anode material candidates, and also leads to high purity oxygen production. Unlike the Hall Cell, operating above the boiling point of magnesium effectively adds in-line distillation, making the product much purer than the raw material. The absence of chlorine and carbon anywhere in the process makes it emission-free. A Department of Energy cost modeling study concluded that this process has lower cost than any other existing or proposed primary magnesium process. This talk will discuss challenges and opportunities involved in scaling SOM Electrolysis to meet the growing magnesium needs of the automotive industry.

**5:40 PM**

**Corrosion Resistance of Graphite Anode for Magnesium Electrolyzers**: *Bing Li*[1]; Jingwei Lou[1]; Can Zhan[1]; Jianguo Yu[1]; [1]East China University of Science and Technology

Graphite is used as a inert anode material due to its stability in a molten chloride salt electrolyte. But during the magnesium electrolysis, there is a gradual wearing of the anodes, which increases cell resistance and hinders hydrodynamics of cell.In this paper, several graphite electrodes, made of different ingredients such as a needle coke, a composite of needle coke and petroleum coke, a fine petroleum coke and a high-purity petroleum coke were employed as anodes for magnesium electrolysers. The electrolysis experiments were conducted from a molten chloride salt electrolyte with current density of 1A/cm2 at 750º under air to accelerate the corrosion of graphite anode. After electrolysis, SEM wasused to examine the morphology of graphite anode and analyze the corrosion resistance of graphite anode. Results showed the corrosion resistance of graphite anode was related to the graphite purity, grain size, apparent porosity and oxidative stability in air.

## Materials in Clean Power Systems V: Clean Coal-, Hydrogen Based-Technologies, Fuel Cells, and Materials for Energy Storage: Materials for Clean Coal Power and CCS Systems II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Conversion and Storage Committee
*Program Organizers:* Xingbo Liu, West Virginia University; Zhenguo Yang, Pacific Northwest National Lab; K. Weil, Pacific Northwest National Lab; Mike Brady, Oak Ridge National Lab; Jay Whitacre, Carnegie Mellon University; Ayyakkannu Manivannan, National Energy Technology Laboratory; Zi-Kui Liu, Penn State University

Monday PM
February 15, 2010
Room: 211
Location: Washington State Convention Center

*Session Chairs:* David Alman, National Energy Technology Laboratory; Bruce Pint, Oak Ridge National Laboratory

**2:00 PM Invited**

**Competing Fatigue Failure Modes in Structural Alloys and the Implications for Life-Management Approaches**: *Michael Caton*[1]; S. K. Jha[2]; J. M. Larsen[1]; [1]US Air Force Research Laboratory; [2]Universal Technology Corporation

Recent fatigue studies of numerous aerospace alloys have revealed competing failure modes under relevant loading conditions contributing to dual-fatigue lifetime distributions. It has been observed that inherent fatigue life variability is often composed of a population of life-limited specimens that experience immediate crack initiation and a population of long-life specimens that demonstrate a significant crack initiation period. Recognizing the competition of these distinctly different mechanisms enables reduced uncertainty in life prediction methods and has significant implications for damage prognosis and life-management practices for fracture-critical components. Alloys demonstrating this phenomenon include Ni-base superalloys, Ti alloys, Al alloys, and γ-TiAl. While these studies have focused on aerospace applications, the observations have relevance for the design and life management of any fracture-critical structure, including power generation systems. A generic framework for applying probabilistic life prediction methods will be presented.

**2:40 PM Invited**
**Addressing Materials Processing Issues for USC Steam Turbines: Cast Versions of Wrought Ni-Based Superalloys**: *Paul Jablonski*[1]; Christopher Cowen[1]; Phillip Maziasz[2]; Neal Evans[2]; Yuki Yamamoto[2]; [1]National Energy Technology Laboratory; [2]Oak Ridge National Laboratory

The proposed steam inlet temperature to the Advanced Ultra Supercritical (A-USC) steam turbine is high enough (760C) that traditional turbine casing and valve body materials such as ferritic/martensitic steels will not work due to temperature limitations of this class of materials. In this presentation we will explore cast versions of seven traditionally wrought Ni-based superalloys in order to evaluate their application as casing or valve components for the next generation of Industrial Steam Turbines. The full size castings are quite substantial: ~4in thick, several feet in diameter and weigh 5-10,000lb each half. Our castings were quite a bit smaller, but section size was retained and cooling rate controlled in order to produce relevant microstructures. We developed a multi-step homogenization heat treatment in order to better deploy the alloy constituents. These castings were subsequently evaluated by characterizing their microstructure as well as their mechanical performance (tensile and creep).

**3:20 PM**
**Assessing Cast Alloys for Use in Advanced Ultra-Supercritical Steam Turbines**: *Neal Evans*[1]; Yukinori Yamamoto[2]; Philip Maziasz[2]; Paul Jablonski[3]; [1]University of Tennessee, Knoxville; [2]Oak Ridge National Laboratory; [3]National Energy Technology Laboratory

Cast forms of traditionally wrought Ni-base precipitation-strengthened superalloys are being considered for service in the ultra-supercritical conditions of next-generation steam turbines (760°C, 35MPa). In this study, small (~10 kg) castings of Nimonic 105, Nimonic 263, Haynes 282, and Inconel 740 were produced under conditions designed to mimic the casting conditions of full-size components, where slow cooling rates and segregation may impact materials properties. A multi-step homogenizing, solutionizing and heat treating schedule was used to eliminate segregation and develop the strengthening γ' phase. For comparison, wrought forms of these alloys were obtained and heat treated to develop the strengthening γ' phase. Microstructures developed in both forms of these alloys were examined via SEM; phase identification and chemistry are being confirmed by TEM. This presentation will discuss microstructures observed in both forms of these alloys and assess the suitability of using these traditionally wrought alloys in cast form.

**3:40 PM Break**

**3:50 PM Invited**
**High Temperature Corrosion of Fe-Cr, Fe-Al, Fe-Si and Fe-Si-Al Alloys in CO2-H2O Gases**: *David Young*[1]; Jianqiang Zhang[1]; Thomas Gheno[1]; Huan Li[1]; [1]University of New South Wales

Iron and model alloys containing 2.25, 9, and 20 wt% Cr, 2, 4 and 6 wt% Al, 1, 2 and 3 wt% Si, and dilute Fe-Si-Al ternaries were reacted in dry and wet CO2 gases at 800°C. External chromia scales grew on Fe-20Cr according to slow, linear kinetics in dry CO2. Additions of H2O accelerated the reaction until steady-state parabolic kinetics were achieved. Lower Cr alloys developed thicker iron oxide scales, featuring cavities, cracks and poor adherence, and sustained internal oxidation. The presence of H2O led to even higher oxidation rates. Aluminium additions to iron of up to 4 wt% had no effect, but 6 wt% significantly slowed oxidation. Silicon additions had little effect. However, simultaneous alloying with aluminium and silicon strongly depressed corrosion rates. The effectiveness of different alloy additions is discussed, along with the effects of water vapour, in the context of oxyfuel combustion technology.

**4:30 PM**
**Phase Stability of Cast and Wrought IN 740 at Ultra Supercritical Boiler Temperatures**: *Christopher Cowen*[1]; Paul Jablonski[1]; Xingbo Liu[2]; [1]United States Department of Energy; [2]West Virginia University

The nickel-based superalloy IN 740 has gained much attention recently as a candidate material for use as tubing in Ultra Super Critical (USC) power plant applications. The ultimate USC goal is a coal-fired power plant that operates utilizing steam at pressures up to 35 MPa and temperatures up to 760°C. The microstructural stability of IN 740 at USC temperatures has been shown to be an issue through observations of gamma prime coagulation, transformation of gamma prime to eta phase, and also through the formation of G phase. In this work, we evaluate the effect of nominal Al content on the microstructural stability of wrought IN 740 at USC temperatures for times up to 2000 hours. We also provide a comparison of the microstructural stability of both wrought and cast versions of IN 740 evaluated under these conditions.

**4:50 PM**
**Development of Friction Stir Welding Technology for Coal and Nuclear Power Applications**: *K. Scott Weil*[1]; Glenn Grant[1]; Yuri Hovanski[1]; Curt Lavender[1]; Jens Darsell[1]; [1]Pacific Northwest National Lab

Most ferritic/martensitic steels used in coal and nuclear plant applications are limited to operating temperatures of <600°C due to degradations in tensile and creep strength. While the addition of insoluble, nanoscale oxide dispersoids greatly improves their high-temperature mechanical properties of these alloys, it can also make them more difficult to join into large-scale componentry. Liquid phase methods of joining such as brazing and fusion welding lead to regions within the joints that are devoid of the dispersoids and the associated strengthening effects, making the joined components susceptible to failure by creep. Friction stir welding is a solid-state joining technique that has shown promise in joining hard-to-weld materials such as ODS alloys in coupon-size specimens. Our work is focused on translating the technology to larger scale plate and pipe joining, evaluating possible tools for thick section welds, and developing methods of weld qualification based on in-situ measurements of weld tool force.

**5:10 PM**
**Interaction of Mechanical Performance and Environmental Compatibility**: *Sebastien Dryepondt*[1]; Bruce Pint[1]; [1]Oak Ridge National Laboratory

Several preliminary studies are being conducted to study the effect of environment on high temperature mechanical properties, including the effect of environmentally-resistant coatings. Earlier work suggested that in-situ measurements would be superior to measuring mechanical properties of specimens after exposure to a simulated environment such as steam or exhaust gas. Examples include Fe- and Ni-base alloys for boiler applications, Ni-base alloys for valves and Al-rich coatings for these alloys. The effect of internal oxidation on mechanical properties is an initial objective. One concern about oxidation-resistant coatings is the potential for detrimental effects on substrate mechanical properties including creep strength and fatigue resistance. In component design, the coating layer is commonly assumed to have no creep strength. However, a coating that reduces component fatigue life is not a practical solution. The coating ductile-to-brittle transition temperature may determine its applicability for some applications.

## Materials Processing Fundamentals: Deformation Processing and Heat Treatment

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Process Technology and Modeling Committee
*Program Organizer:* Prince Anyalebechi, Grand Valley State University

Monday PM Room: 601
February 15, 2010 Location: Washington State Convention Center

*Session Chair:* Prince Anyalebechi, Grand Valley State University

**2:00 PM**
**Mechanical Properties and Their Dependence on Microstructure in Hot-Rolled 3rd Generation Advanced High Strength Steels**: *Meghan McGrath*[1]; Dave Van Aken[1]; Von Richards[1]; [1]Missouri University of Science and Technology

A new third generation advanced high strength steel is based on duplex microstructures of acicular ferrite and retained austenite. Good combinations of strength and ductility can be achieved in these duplex steels by suppressing cementite formation and transformation induced plasticity from the retained austenite. This paper compared the mechanical properties of two hot-rolled alloys with duplex microstructures. The two compositions used were: (1)0.14%C, 13.87%Mn, 1.42%Si, and 3.51%Al; and (2) 0.06%C, 14.2%Mn, 1.85%Si, and 2.38%Al. Addition of aluminum reduces density along with inhibiting formation of cementite. These steels were cast, homogenized, and hot rolled at 900ºC to obtain a refined microstructure with ultimate tensile strengths of 865 MPa and 1,217 MPa with elongations of 39% and 28%, respectively.

The amount of metastable austenite in both compositions decreased after tensile testing from 58% and 28% to 6% and 0%, respectively. Microstructures were characterized using optical microscopy, scanning electron microscopy, and x-ray diffraction.

**2:20 PM**

**Influence of Direct Aged Treatment on Creep Behaviors of Hot Continuous Rolling GH4169 Superalloy**: *Sugui Tian*[1]; Zhenrong Li[1]; Zhonggang Zhao[1]; Liqing Chen[2]; Xianghua Liu[2]; [1]Shenyang University of Technology; [2]Northeast University

By direct aged treatment, creep properties measurement and microstructure observation, the influences of the direct aged treatment on the creep behaviors of Hot Continuous Rolling GH4169 Superalloy are investigated. Results show that the creep resistance of Hot Continuous Rolling GH4169 Superalloy may be obviously improved by directly aged treatment. Under the applied stress of 700MPa at 660º, the creep life of the alloy is enhanced to 126h from 60h. During hot rolling, the deformation features of the alloy are the twinning and the double orientations slipping of the dislocation activated within the twinning regions. And the alloy displays a smaller grain size. After direct aged treatment, the finer γ ‘‘phase is dispersedly precipitated in the matrix of the alloy, which is a main reason of enhancing creep life of the alloy.

**2:40 PM**

**The Effect of Carburizing on the Fatigue Life of 4130 Steel**: *Roselita Fragoudakis*[1]; Anil Saigal[1]; [1]Tufts University

AISI 4130 (25Cr-4Mo) steel is a medium carbon Cr-Mo steel used in automotive, aeronautic and other general-purpose industries. In this study, 4130 steel fatigue specimens were carburized at 1700ºF to enhance the carbon content in the steel, raising it from approximately 0.3% wt (non-carburized) to 0.9% wt (carburized, oil quenched and tempered). Rotating bending fatigue tests were performed on the R. R. Moore Rotating Beam Fatigue Testing System, in order to determine the high cycle fatigue (HCF) life of 4130 steel and plot the corresponding S-N diagrams. A comparison of the results of carburized and non-carburized specimens shows that carburizing of steel: a) doubles the surface hardness and b) improves the fatigue strength of the steel by at least an order of magnitude.

**3:00 PM**

**Cast and Wrought Tensile Properties of a 2nd Generation Advanced High Strength Steel (AHSS)**: *Tracy Frick*[1]; Dave Van Aken[1]; Ryan Howell[1]; [1]Missouri University of Science and Technology

Second generation AHSS include an age hardening austenitic alloy known as TRIPLEX. These steels are duplex, austenite and ferrite, when solution treated and are hardened by aging at 550°C to obtain ultimate tensile strengths greater than 1,500 MPa. An austenitic grade with composition Fe-30Mn-9Al-1Si-0.9C-0.5Mo has been developed for lightweight, cast military armor. The addition of 9 wt.% aluminum reduces the density by 13% over traditional steel chemistries. The current study examines the same alloy after hot rolling and recrystallization at 900°C. Specimens were water quenched to produce an equiaxed, 10 to 20 µm grain diameter with a hardness of Rockwell C27 after recrystallization. The rolled microstructure contained less than 1% ferrite, which appeared as small discrete islands aligned parallel to the principal rolling direction. Aging at 530°C for 10 and 30 hours produced a hardness of Rockwell C30 and Rockwell C37, respectively.

**3:20 PM Break**

**3:40 PM**

**Microstructure and Mechanical Properties of Multiphase Steel after Quenching and Partitioning**: *Thomas Rieger*[1]; Oliver Buelters[2]; Jian Bian[3]; [1]Department of Ferrous Metallurgy, RWTH Aachen University; [2]Institute for Metal Forming, RWTH Aachen University; [3]ThyssenKruppSteel

The ‘quenching and partitioning’ process has been developed to produce high strength steel. After austenitisation and interrupted quenching the austenite transforms partly to martensite. The remaining austenite is stabilised by carbon partitioning from martensite. After final quench the tempered martensite gives a high strength level. The TRIP-assisted local strain hardening assures satisfying ductility. The process design is limited by reactions competing against the carbon partitioning, i. e. carbide precipitation and isothermal bainite transformation. For a given chemical composition the vast scope of adjustable mechanical properties is investigated as function of the process parameters. The corresponding microstructure is characterised by light and electron microscopy and XRD measurements. Supplementary information on the process kinetics is obtained by dilatometry. Silicon shows more effective in retarding cementite precipitation than aluminium. Retained austenite occurs in filmy constitution between bcc laths as well as in blocky grains. The authors are grateful for support by ThyssenKruppSteel.

**4:00 PM**

**Ag Exudation during Internal Oxidation in Various Contact Materials**: *Gunther Schimmel*[1]; Bernd Kempf[2]; Markus Rettenmayr[1]; [1]Friedrich-Schiller-University Jena; [2]Umicore AG & Co. KG

Internally oxidized Ag alloys are frequently used as contact materials in high-energy switching operations. During internal oxidation, diffusion of Ag atoms towards the sample surface is commonly observed. This leads to the formation of external and internal layers that consist of essentially pure Ag. In the present work, results of high temperature bending tests of already oxidized specimens and targeted oxidation experiments accompanied by FEM simulation calculations are presented. There is no correlation between internal stresses and the amount of Ag in the exuded layers. The diffusion direction of Ag atoms is shown to be dependent on the direction of oxygen diffusion. It is concluded that exudation is dependent on the oxygen concentration gradient that generates a difference in the chemical potential of the Ag atoms. The gradient in oxygen concentration between sample surface and internal oxidation front is identified as main driving force for the Ag diffusion.

**4:20 PM**

**Effects of Phosphorous on the Precipitation Kinetics of κ-carbides in the Fe-30%Mn-9%Al-1%Si-0.9%C-0.5%Mo Alloy System**: *Laura Bartlett*[1]; David Van Aken[1]; Kent Peaslee[1]; Ryan Howell[2]; [1]Missouri University of Science and Technology; [2]Army Research Lab

Recent studies on the age-hardenable Fe-30%Mn-9%Al-1%Si-0.9%C-0.5%Mo alloy have shown that small increases in the amount of phosphorous, 0.001 to 0.043 wt%, corresponded to an increase in hardness for all aging times tested in the temperature range of 530 to 600°C. As phosphorous increased, the time to achieve peak hardness was also found to decrease by 50% for specimens aged at 530°C and 60% for specimens aged at 600°C. Avrami kinetic analysis suggests that phosphorous lowers the activation energy for the precipitation of fine κ-carbide, $(Fe,Mn)_3AlC$, from 301 kJ/mol to 204 kJ/mol as the phosphorous level is increased from 0.001 to 0.043 wt%. Microstructural changes as a function of aging time, temperature, and phosphorous content were characterized by optical microscopy as well as scanning electron microscopy (SEM). Phosphorous was found to segregate to interdendritic grain boundaries and promote both the precipitation and size of the κ-carbide on the grain boundaries.

**4:40 PM**

**A Study on Heat Transfer Coefficient Distribution in High Pressure Hydrogen Quenching**: *Bowang Xiao*[1]; Gang Wang[1]; Yiming Rong[1]; [1]Worcester Polytechnic Institute (WPI)

Recently more and more attentions are paid to High pressure hydrogen quenching (HPHQ) for its advantages such as environmental friendliness, low distortion, etc, compared to oil and water quenching. In practice, heat transfer coefficient (HTC) usually varies from part surface to surface because of the variation of gas velocity around the part. This article studies the effect of HTC distribution on part distortion and microstructure after HPHQ and shows that Finite Element simulations considering HTC distribution predict different distortion from these without considering HTC distribution. It is also pointed out in this article that HTC distribution can be optimized by adjusting part orientation and HPHQ condition so that part distortion is minimized.

**5:00 PM**

**Evolution of Graphite Phase Morphology during Graphitization Process in Hypereutectoid Steels**: *Amin Rounaghi*[1]; Payam Shayesteh[1]; Ali-Reza Kiani-Rashid[1]; [1]Ferdowsi University

Graphitization process in steels is referred to the decomposition of cementite phase to graphite and ferrite during prolonged time. Graphite presence in the final microstructure is identified as one of the best solid lubricants that significantly improves wear resistance, machinability and self-lubricating of ferrous alloys. In addition, investigations show effect of graphite shape and morphology on mentioned properties. In this matter, we can referred to magnesium edition in molten iron for production ductile cast iron with spherical graphites and its

better strength rather than other similar kinds such as gray cast iron with flake graphites. In present research, effect of graphite stabilizer alloying elements on morphology, size and distribution of graphite particles has been studied during graphitization process in hypereutectoid steels.

## Mechanical Performance for Current and Next-Generation Nuclear Reactors: Ensuring Lifetime and Reliability

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS/ASM: Nuclear Materials Committee
*Program Organizers:* Dylan Morris, NIST; Greg Oberson, Nuclear Regulatory Commission; Nicholas Barbosa, National Institute of Standards and Technology; Wolfgang Hoffelner, Paul Scherrer Institute

Monday PM Room: 201
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Greg Oberson, U.S. Nuclear Regulatory Commission; Matthew Kerr, US Nuclear Regulatory Commission

**2:00 PM Invited**
**Ensuring the Performance of Nuclear Reactor Pressure Vessels for Long Time Service**: *Randy Nanstad*[1]; [1]Oak Ridge National Laboratory

Structural integrity of the reactor pressure vessel is a critical element in demonstrating the capability of light water reactors for operation to at least 80 y. The Light Water Reactor Sustainability Program is a collaborative program between the U.S. Department of Energy and the private sector directed at extending the life of the present generation of nuclear power plants to enable such long-time operation. Given that the current generation of light water reactors were intended to operate for 40 y, there are significant issues that need to be addressed to reduce the uncertainties in regulatory application. The neutron dose to the vessel will at least double, and the database for such high dose levels under the low flux conditions in the vessel is nonexistent. Associated with this factor are uncertainties regarding flux effects, effects of relatively high nickel content, and uncertainties regarding application of fracture mechanics.

**2:30 PM Invited**
**Experience of the Fossil Industry with the Creep-Strength Enhanced Ferritic Steels**: *Jeffrey Henry*[1]; [1]Energy Solutions Group

The Creep Strength Enhanced Ferritic Steels, such as Grade 91, have been selected by design engineers for use in the fossil power industry because of their superior elevated temperature properties when compared to more "traditional" alloys, such as Grade 22. In many cases the selections have been made without regard to the ability of OEMs and constructors to satisfy the more demanding process requirements of these steels. In this presentation we will review the numerous problems that were encountered in the fossil industry when Grade 91 was introduced on a large scale and will discuss the reason for those problems. In addition, we will look at the steps that were taken by the ASME Code to address some of the more critical issues that were encountered in an effort to minimize the risk of component failure.

**3:00 PM**
**Comparative Plant Performance of Stabilized and Non-Stabilized Austenitic Stainless Steels**: *Raul Rebak*[1]; [1]GE Global Research

Austenitic stainless steels core internals components in light water reactors may be susceptible to stress corrosion cracking and irradiation assisted stress corrosion cracking. One of the effects of irradiation is the hardening of these materials and a change in the dislocation distribution in the alloy. Irradiation also alters the local chemistry of the alloys, for example in the vicinity of grain boundaries which may increase their susceptibility to environmentally assisted cracking. In the United States the nuclear power plants internals are mostly made using 304/304L, in Japan the nuclear industry prefers 316L and in Europe (e.g.; Germany) mostly the stabilized 347/321 versions of stainless steels. An open literature review has been conducted to compare the relative performance of these austenitic stainless steels under operation conditions. The review will cover, among other things, the sensitization behavior during welding, the irradiation embrittlement and the susceptibility to environmentally assisted cracking.

**3:20 PM**
**Effects of Common Alloying Additions on Solidification Cracking of Zirconium Alloys**: *Micah Hackett*[1]; George Young[1]; [1]KAPL

Zirconium alloys are commonly welded for nuclear applications, but they can be susceptible to solidification cracking. In this work the role of minor alloying additions on the weldability and solidification cracking behavior of zirconium alloys is explored using transverse varestraint testing with gas tungsten arc welding (GTAW). The alloy compositions vary in the content of Fe, Cr, Sn and Nb in order to measure the effect of composition on solidification cracking susceptibility. Solidification cracking resistance was assessed as a function of welding speed and heat input. Gleeble testing was used to determine the solidus temperature to better understand the effects of alloying elements on solidification range.

**3:40 PM**
**Effect of Neutron Radiation Exposure on Low Cycle Fatigue of 304SS**: *Korukonda Murty*[1]; Indrajit Charit[2]; [1]North Carolina State University; [2]University of Idaho

We review here the tensile and fatigue characteristics of Type 304 SS from hexagonal cans of EBR-II guide tubes before and after irradiation to a fast fluence of ~8x1026 n/m2. The mechanical properties were evaluated at room temperature and 598K. Typical radiation hardening is noted along with ductility loss while the work hardening parameter decreased following radiation exposure. Symmetrical strain reversal fatigue tests at 0.1 cps were performed at varied strains from ~1% to 2.4% with number of cycles varying from 500 to 40,000. While a slight decrease in fatigue life is noted at high strains, the data clearly revealed improved fatigue life at low strains or high cycles. Predictive model equation developed based on Universal Slopes concept using tensile data is noted to predict the fatigue behavior of both unirradiated and irradiated materials at room temperature and 598K. This work is supported by the Department of Energy grant #DE-AC07-05ID14517.

**4:00 PM Break**

**4:15 PM Invited**
**When the Turtle Can't Get There and the Rabbit Gets Lost: Predicting Low Flux High Fluence RPV Embrittlement**: *G. Robert Odette*[1]; Takuya Yamamoto[1]; [1]University of California, Santa Barbara

Nuclear plant life extension will require accurate predictions of irradiation-induced ductile-brittle transition temperature shifts (TTS) in RPV steels at high fluence, far outside the existing surveillance database. Progress in mechanistic understanding of irradiation embrittlement has led to physically motivated TTS correlation models that provide excellent statistical fits to the surveillance database. However, these models cannot reliably extrapolate TTS predictions, since 99% of the existing surveillance data is less than half the maximum extended life fluence. High flux can access high fluence, but such TTS data is under-predicted by current models that do not properly account for dose rate effects. We describes a systematic effort to develop a mechanism database on dose rate effects in support building physically based models to reliably predict TTS for low flux high fluence conditions, that also account for possible embrittlment by delayed formation of "late blooming phases" in low copper steels.

**4:45 PM Invited**
**Materials Issues Potentially Impacting Long-Term Safe Operations**: *C. E. Carpenter*[1]; [1]U.S. Nuclear Regulatory Commission

Existing nuclear power plants (NPPs), which were originally designed for operating lives of 30 to 40 years, are being considered for extended operating periods, of perhaps 80 or more years. The U.S. Nuclear Regulatory Commission (NRC) has enacted a process for plant safety assessments to enable license renewal from 40 to 60 years, and 54 such license extensions has been granted to date; and, it is expected that more than 90 percent of current plants will seek at least an initial license renewal period (e.g., 40 to 60 years). The NRC is initiating research to determine what are the key technical and regulatory issues that require attention by the nuclear industry to enable a second, and subsequent, renewal period, to 80 years and potentially longer. This presentation will address the key material aging-related degradation issues that are under consideration by the NRC at this time.

**5:15 PM Invited**

**Safety Evaluation Challenges for NGNP VHTR Materials of Construction and Components**: *Makuteswara Srinivasan*[1]; Amy Hull[1]; Shah Malik[1]; [1]U.S. Nuclear Regulatory Commission

A variety of specialty metallic alloys, nuclear graphite, ceramic insulation, and ceramic matrix composites, will be used in the design and construction of components for the very high temperature gas cooled reactor (VHTR) next generation nuclear plant (NGNP). Several challenges exist in the safety evaluation of these components for design certification and licensing review. The component performance reliability estimate depends on the robustness of several models which provide technical bases, data, and analyses methods. These models address environment-assisted material degradation, component inspection, structural integrity estimation, and risk assessment. Aspects of these models are inter-connected and influence risk information. A technical decision exercise has provided prioritization guidance on research areas needed for construction of the above models. Consensus design codes and standards need to address performance acceptance criteria, surveillance and inservice inspection requirements, and component degradation management program and procedure to assess its efficacy.

**5:45 PM**

**Gen IV Materials (ASME-DOE Project)**: *James Ramirez*[1]; [1]ASME

To facilitate the commercialization (licensing, construction, operations and maintenance, etc) of Generation IV reactors, ASME Standards Technology, LLC (ASME ST-LLC) has been working with major stakeholders from industry, government, and academia to develop and compile material research required to update and build the next set of ASME codes and standards that will directly support commercialization of Gen IV reactors. This paper will focus on providing a summary of the projects results currently being developed under the ASME ST-LLC and DOE materials project and more specifically will address the following areas: operating conditions for allowable stress values, ASME Code considerations for IHX, creep and creep-fatigue crack growths at structural discontinuities and welds, improving ASME Subsection NH, and new materials such as Inconel 617, Haynes 230, and Hastelloy XR.ASME ST-LLC utilizes and "early involvement approach" where stakeholders review and comment on the projects conclusions early on.

## Modeling, Simulation, and Theory of Nanomechanical Materials Behavior: Plasticity and Strength of Nanostructured and Nanoscale Materials II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS: Nanomechanical Materials Behavior Committee

*Program Organizers:* Thomas Buchheit, Sandia National Laboratories; Sergey Medyanik, Washington State Univ.; Douglas Spearot, University of Arkansas; Lawerence Friedman, Penn State University; Edmund Webb, Sandia National Laboratories

Monday PM Room: 304
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Jian Wang, LANL; Edmund Webb, Sandia National Laboratories

**2:00 PM Invited**

**Discrete Dislocation Modeling of the Relaxation of Intrinsic Stress in Thin Films**: Can Ayas[1]; *Erik Van der Giessen*[1]; [1]University of Groningen

Deposition techniques for (sub-)micron thick metallic films generally leave intrinsic stresses. They arise as a consequence of the Volmer--Weber mechanism. Island growth and coalescence lead to a tensile stress in the film, which can be relaxed by the diffusion of material into the grain boundary, or by plasticity inside the grains, or by a combination of both.We present a framework to analyze these relaxation processes --during or after growth-- based on discrete dislocations. The approach builds on an existing method to describe plastic deformation at (sub-)micron size scales in terms of dislocations treated as line singularities in an elastic solid. Within the same framework, grain boundary diffusion is represented by the 'climb' motion of edge dislocations along grain boundaries, with a mobility that is determined by the diffusion coefficient. In addition to the separate relaxation mechanisms, we analyze the coupling between diffusion and dislocation plasticity.

**2:30 PM**

**A Further Step in Understanding the Plastisity Size-Dependency: 3D Modelling of Solid and Annular Micropillars**: *Jaafar El-Awady*[1]; Satish Rao[2]; Christopher Woodward[3]; Dennis Dimiduk[3]; [1]AFRL/UTC; [2]AFRL/UES; [3]AFRL

In this work, we conduct three-dimensional dislocation dynamics simulations of solid and annular cylindrical Ni single-crystals under compression. The simulations are performed using the parametric dislocation dynamics coupled with the boundary element method. We investigate various boundary conditions and loading techniques as well as the trapping of dislocations within the micropillar due to the presence of FIB damage or due to a coating with a higher strength material. It is observed that the the FIB damage as well as the coatings will raise the stored dislocation density by a couple of orders of magnitude. Annular micropillars are oserved to exhibit a size effect similar to solid micropillars. The flow strength obays a power law relationship with the effective radius of the annular pillar (i.e. Rout - Rin). Finally, the results of the annular micropillar simulations are compared to the plastic deformation of freestanding thin films.

**2:50 PM**

**Modeling the Statistics of Yield Behavior in Nanopillar Compression and Nanoindentation**: *James Morris*[1]; Hongbin Bei[1]; George Pharr[2]; Easo George[1]; [1]Oak Ridge National Laboratory; [2]University of Tennessee

Strengths of materials at small length scales approach theoretical values, due to the absence of defects in the probed volumes. These strengths may be an order of magnitude larger than those measured in the bulk. When the probed volume contains few dislocations or other defects, the mechanical properties are inherently statistical, with a wide variation in the yield and flow behavior. A statistical model is presented to capture the change in behavior from the defect-free case to the bulk limit, with a focus on capturing the wide variability of the intermediate regime. The model is compared to both nanoindentation and pillar experiments. Both the average potency and the distribution of dislocations and pinning points are included. We also present molecular dynamics simulations examining the effects of pre-existing dislocation networks on subsequent plastic behavior. This research was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy.

**3:10 PM Invited**

**Modeling the Mechanical Properties of Gum Metal**: *Daryl Chrzan*[1]; Matthew Sherburne[1]; Yuranan Hanlumyuang[1]; Tianshu Li[2]; J. W. Morris, Jr.[1]; [1]University of California, Berkeley; [2]University of California, Davis

The remarkable mechanical properties of Gum Metal, a Ti-Nb based alloy developed recently by Toyota, are considered within the context of ideal strength and dislocation core structures. The hypothesis that these alloys deform at stresses approaching their ideal strength is examined. It is suggested that the elastic anisotropy of these materials enables the dislocations to be pinned easily by nanoscale defects even at stresses approaching the ideal shear strength of the bulk alloy. Further, ab initio electronic structure total energy calculations indicate that the dislocation core structures are spread significantly, a feature that may also lead to pinning of dislocations. This work is supported by the National Science Foundation, and Toyota Motor Corporation.

**3:40 PM Break**

**4:00 PM**

**Tensile Deformation of Gold Nanowires: Structural Transitions during Elongation and Breaking Mechanisms**: *Francesca Tavazza*[1]; Lyle Levine[1]; Anne Chaka[1]; [1]National Institute of Standards and Technology

Semistatic density functional theory is used to explore the evolution of gold nanowires during tensile deformation under a wide range of conditions. Large structural changes are observed, resulting in the formation of locally ordered intermediate structures with interesting electronic properties. A rich diversity of deformation pathways is also uncovered, that converge to only two final local configurations with reproducible breaking strengths, in agreement with experimental results

**4:20 PM**
**Atomic-Scale Analysis of the Mechanical Behavior of Gold Nanofoams**: *Kedarnath Kolluri*[1]; Michael Demkowicz[1]; [1]Massachusetts Institute of Technology

We present atomic-scale analysis of a model Au nanofoam (nf-Au) using an EAM potential. A model nf-Au structure forms spontaneously upon relaxing a random distribution of atoms with a density of 20% of perfect crystalline FCC Au. The relaxed microstructure is polycrystalline and contains numerous defects in both the nanofoam ligaments and nodes. Annealing using Molecular Dynamics (MD) at 300 K for ~ 1 ns causes the nanofoam to coarsen. During volume conserving uniaxial compression of the nf-Au, ligaments are in a combined state of tension, shear, bending, and torsion. Peaks and valleys of the stress-strain curves after initial elastic loading correlate with the necking and pinch-off, respectively, of nanofoam ligaments. Similar mechanisms are found to operate during nanofoam coarsening.

**4:40 PM**
**Molecular Dynamics Simulations of Uniaxial Compression of Silicon Nanoparticles**: *Lucas Hale*[1]; William Gerberich[1]; Roberto Ballarini[1]; Neville Moody[2]; Xiaowang Zhou[2]; Jonathan Zimmerman[2]; [1]University of Minnesota; [2]Sandia National Laboratories

Molecular dynamics has been used to study the uniaxial compression of silicon nanospheres based upon both Stillinger-Weber and Tersoff potentials. The mechanical behavior of the particles is found to be sensitive to sphere size, temperature, crystallographic orientation, and the interatomic potential. Plastic deformation is found to be accompanied by nucleation and migration of dislocation when the particle size and temperature exceed some critical values. The critical particle size for the dislocation deformation mechanism is larger for the Tersoff potential than for the Stillinger-Weber potential. During compression along the [100] crystallographic direction using the Tersoff potential, a diamond-cubic to β-Sn phase transformation occurs. Similar transformation is not found for the Stillinger-Weber potential. An alternate yielding and hardening mechanism is also observed. These results provide comprehensive understanding of the unique mechanical properties of silicon nanoparticles.

**5:00 PM**
**Reaction Rate Theory Prediction of Dislocation Nucleation in Aluminum at Room Temperature**: *Linh Nguyen*[1]; Derek Warner[1]; [1]Cornell University

The limited time domain directly accessible to atomistic simulations of plasticity has long hindered their predictive capability. For nano-structured and/or nano-dimensioned metals, this shortcoming is exacerbated due to the high strain rate sensitivity of the controlling deformation mechanisms, such as dislocation nucleation. One viable means of overcoming this challenge is to view plasticity within a thermally activated reaction rate context. In this work, we use the finite temperature string method to calculate the most probable path for dislocation nucleation in a nano-wire and nano-void at room temperature. By parallelizing the samples trajectory along this path we can calculate directly the rate of nucleation at experimental loads. Sampling along the reaction path yields the free energy of activation, and thus, allows for a systematic assessment of the transition state theory prediction and the validity of other simplifying approximations common to recent efforts.

## Neutron and X-Ray Studies of Advanced Materials III: 2D Materials Science from Diffraction

*Sponsored by:* The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Titanium Committee
*Program Organizers:* Rozaliya Barabash, Oak Ridge National Laboratory; Jaimie Tiley, Air Force Research Laboratory; Erica Lilleodden, GKSS Research Center; Peter Liaw, University of Tennessee; Yandong Wang, Northeastern University

Monday PM
February 15, 2010
Room: 303
Location: Washington State Convention Center

*Session Chairs:* Karen Pantleon, Denmark Technical University; Carol Thomson, NIU

**2:00 PM Keynote**
**In Situ X-Ray Scattering Studies of the Polarization Structure of Ultrathin Ferroelectric Films**: *Gregory Stephenson*[1]; Matthew Highland[1]; Dillon Fong[1]; Timothy Fister[1]; Paul Fuoss[1]; Jeffrey Eastman[1]; Stephen Streiffer[1]; Carol Thompson[2]; [1]Argonne National Laboratory; [2]Northern Illinois University

Ferroelectric thin films are both fascinating and useful because their polarization structure depends sensitively on the nature of the charge compensation at their interfaces. We have been using in situ surface x-ray scattering to monitor film growth and observe the polarization structure of ultrathin epitaxial PbTiO3 films as a function of thickness and environmental conditions. Recently we discovered that in coherent PbTiO3 / SrRuO3 heterostructures on SrTiO3 (001) substrates, the direction of polarization can be switched by changing the oxygen partial pressure (pO2) in equilibrium with the surface. Here we present results on the equilibrium polarization phase diagram and stability limits as a function of temperature, pO2, and film thickness. Results will be compared with a model for the interaction of surface chemistry with the ferroelectric phase transition. Work supported under contract DE-AC02-06CH11357 between UChicago Argonne LLC and the Dept. of Energy.

**2:30 PM Invited**
**Nucleation, Coarsening, and Coalescence during Layer-by Layer Growth via Pulsed Laser Deposition: Time-Resolved, Diffuse X-Ray Scattering Studies**: *Joel Brock*[1]; John Ferguson[1]; Hui-Qiong Wang[1]; Arthur Woll[1]; [1]Cornell University

We report real-time x-ray diffuse scattering studies of the structure of the growth surface of complex oxide thin films during pulsed laser deposition in the layer-by-layer growth mode. These measurements provide detailed information on the in-plane growth kinetics on the relevant time and length scales. Empirically, it is necessary to monitor both the in-plane length scale, $L$, and the characteristic relaxation time, $\tau$, as a function of temperature, to obtain the diffusivity, $D$. For both homo- and hetero-eptitaxy, island nucleation and coalescence under supersaturated conditions followed by coarsening of the resulting island distribution are key fundamental growth processes. The time-dependent pair distribution function, $G(r,t)$, obtained by Hankel transforming the experimental data, is used to characterize both the island size distribution and the near neighbor separations as a function of time and coverage.

**2:50 PM Invited**
**The Growth and Formation of Nanostructures at Surfaces: In Situ X-Ray Scattering Studies**: *Paul Miceli*[1]; Shawn Hayden[1]; Michael Gramlich[1]; Chinkyo Kim[2]; Edward Conrad[3]; [1]University of Missouri-Columbia; [2]Kyunghee University; [3]Georgia Institute of Technology

The surfaces of metal crystals are a useful laboratory for studying atomic-scale processes that control the growth and formation of nanostructures. Many of these processes depend on structure that lies below the surface. Because it is sensitive to both the surface and the subsurface of a sample, x-ray scattering possesses unique capabilities for investigating buried defects and subsurface strains that are not observed by conventional "surface-only" tools. This talk will discuss recent results from our ongoing research program that explores vacancy clusters, surface strains and quantum size effects in the epitaxial crystal growth of metals. These are in-situ studies which utilize the surface scattering

facility that we developed at Sector 6 at the Advanced Photon Source. Financial support is gratefully acknowledged from NSF and DOE.

**3:10 PM Invited**

**Time-Resolved X-Ray Studies of Materials Processing at Surfaces and Interfaces - Present and Future**: *Carol Thompson*[1]; [1]Northern Illinois University

The development of surface and interface x-ray scattering and spectroscopy techniques has led to seminal in-situ measurements of materials as they are synthesized and processed or interact with their target environments. Researchers have applied these techniques to study fundamentals of advanced materials, e.g., deposition, catalysis, electrochemistry, phase transitions, oxidation, corrosion, and biological activity. At the x-ray synchrotrons, several new user facilities have been proposed for in-situ and real-time materials processing. As these dedicated facilities come into operation within the next few years, they will provide increased user access and offer flexible designs for experiments. This enhances the opportunities for new communities of materials researchers to exploit these techniques successfully for their materials systems and questions. Using examples from the current literature and from our own research, we describe the strengths and weaknesses of these x-ray techniques for studying materials synthesis and processing in real-time at surfaces and interfaces.

**3:30 PM Invited**

**Two-Dimensional X-Ray Diffraction for Advanced Materials Analysis**: *Bob He*[1]; [1]Bruker AXS

Two-dimensional x-ray diffraction is the ideal, non-destructive, analytical method for examining samples of all kinds, such as metals, polymers, ceramics, semiconductors, and biomaterials, especially materials with anisotropic, nanoscale, composite or heterogeneous structures. This presentation introduces the geometry convention, data collection strategy, data interpretation, and data analysis in two-dimensional X-ray diffraction for various applications, including phase identification, crystal size, stress and texture measurements. Based on the matrix transformation between diffraction space, detector space and sample space, the unit diffraction vector can be expressed in the sample space corresponding to all the geometric parameters and Bragg conditions. The fundamental equations for crystal size, stress and texture measurement are developed from the unit diffraction vector expressed in sample space. The experimental examples in crystal size, stress and texture analysis for various samples, including friction stir weldment, magnetron sputter-deposited Cu films and micro-arc oxidation generated TiO2 films, are given in this presentation.

**3:50 PM Invited**

**In-Situ X-Ray Diffraction Studies of Microstructure Evolution in Electrodeposits at Room Temperature and Elevated Temperatures**: *Karen Pantleon*[1]; [1]Technical University of Denmark

The thermodynamic non-equilibrium state of electrodeposited layers causes changes of the microstructure as a function of time and/or temperature. For Cu- and Ag-layers, self-annealing occurs already at room temperature, whereas Ni-layers evolve only at elevated temperatures. (Self-)annealing affects the functionality and reliability of electrodeposited components, which can be beneficial, e.g. increased electrical conductivity of interconnect lines, or detrimental, e.g. reduced strength in MEMS applications. Applying X-ray diffraction with conventional and synchrotron radiation, the kinetics of microstructure evolution has been investigated in-situ as a function of time at room temperature and during isothermal and isochronal annealing up to temperatures of 800 K. Time-resolved line profile analysis and crystallographic texture analysis during the course of (self-)annealing have revealed orientation dependent growth of the as-deposited nanocrystalline grains and drastic changes of preferred grain orientations. Quantification of the kinetics allowed conclusions on the mechanisms of microstructure evolution.

**4:10 PM Break**

**4:20 PM Invited**

**Strain Profiling in Group-III-Nitride Based Multilayer Systems on Silicon**: *Alois Krost*[1]; Rainer Clos[1]; Juergen Blaesing[1]; [1]Otto-von-Guericke University Magdeburg

The epitaxial growth of crack free group-III Nitrides on large size silicon substrates is a promising way towards large scale production of, e.g. high-brightness LEDs for general lighting. However, the growth of thick layers is hindered by a huge thermal mismatch between GaN and Si inducing tensile stress. This problem can be overcome by a proper strain management during growth by the insertion of low-temperature AlN interlayers allowing for the growth of group-III Nitride layers exceeding 7 micrometer in thickness on 6 inch substrates. The impact of strain compensating (Al,Ga)N is evaluated by in situ curvature measurements and ex situ x-ray measurements. The strains and stresses of such multilayers are calculated analytically and compared to experimental results.

**4:40 PM Invited**

**Real Time Reciprocal Space Mapping of Nano-Islands Induced by Quantum Confinement**: *Hawoong Hong*[1]; Aaron Gray[2]; T.-C. Chiannng[2]; [1]Argonne National Lab; [2]University of Illinois at Urbana-Champaign

The effects of the quantum confinement have been observed pronouncedly in the island morphology in the Pb thin films. The evolution of these nano-islands on Si (111)-(7x7) and sapphire (001) surfaces has been studied. A CCD camera was used to collect the 2/3-D maps of the x-ray reflectivity in real time. Large ranges of the reflectivity curves, and the rocking curves at every point on the reflectivity curves could be continuously measured. The abundance of information from 2-D k-space maps reveals clear changes in growth modes of thin Pb films. With the 3-D extension of this method, it was possible to observe the ordering of the islands and their coarsening. The islands coarsen and order, maintaining a nearly uniform inter-island distance but without angular correlation. Over a wide temperature range, the inter-island ordering is well correlated with the development of "magic" island heights caused by quantum confinement.

**5:00 PM**

**Dependence of the Preferred Growth Directions of GaN Nanorods on Polytypism**: Sanghwa Lee[1]; Yuri Sohn[1]; *Chinkyo Kim*[1]; Dong Ryeol Lee[2]; Hyun-Hwi Lee[3]; [1]Kyung Hee University; [2]Soongsil University; [3]Pohang Accelerator Lab

Reciprocal-space-mapping with a 2-dimensional (2-D) area detector in a grazing incidence geometry is applied to determine crystallographic orientations of GaN nanostructures epitaxially grown on a sapphire substrate. By using both unprojected and projected reciprocal space mapping with a proper coordinate transformation, the crystallographic orientations as well as the preferred growth directions of GaN nanostructures with respect to that of a substrate were unambiguously determined. In particular, polytypism is found to play an important role in determining the preferred growth directions and preferred orientations of GaN nanorods.

**5:15 PM**

**Roentgenograhic Determination of Residual Stresses in Carbonitrided Layers**: Angel Zumbilev[1]; *Iliya Zumbilev*[1]; [1]Technical University of Sofia, Plovdiv Branch

The aim of the present investigation is to examine the influence of carbonitriding in low temperature plasma over forming macro-residual stresses on the surface of the materials.Particular modes of ion carbonitriding are considered, in which layers of different depth and different surface micro-hardness are obtained. The residual stresses in the carbonitride layers are determined by the method of sin2Y. The results show that at different modes of ion carbonitriding residual macro-stresses with different sizes are obtained and they depend on the mode of treatment (ammonia pressure, temperature of carbonitriding, duration of treatment) and the depth of the carbonitride zone.

**5:25 PM Invited**

**High-Energy X-Ray Measurements of Layered Systems for Energy Applications**: *Jonathan Almer*[1]; Di-Jia Liu[2]; B. Harder[3]; K. Faber[3]; [1]Argonne National Laboratory, X-Ray Science Division; [2]Argonne National Laboratory, Chemical Technology Division; [3]Northwestern University, Materials Science and Engineering Department

Microfocused high-energy x-rays are used to investigate two layered systems relevant for energy applications: solid-oxide fuel cells and thermal barrier coatings. The combination of an undulator source, brilliance preserving optics and focusing lenses at Sector 1 of the Advanced Photon Source provides high-energy x-rays (E~80 keV) with transverse beamsizes on the micron-level. In addition to their high penetration power, a key feature of high-energy x-rays is their forward scattering geometry, which when combined with area detectors allows for efficient mapping of microstructural gradients. Both x-ray diffraction and radiography are used to map the areal distribution of phases, internal strain, lattice parameter and integrated porosity of solid-oxide fuel cell (SOFC) stacks.

This information is used to better understand SOFC degradation mechanisms in the presence of reactive Cr, as well as further general understanding of SOFC processing-structure relationships. Figure 1 illustrates the lattice parameter distribution of the $La_xSr_{1-x}MnO_{3-\delta}$ phase from a deactivated SOFC, obtained from GSAS-refinement of diffraction data. The changes in lattice parameter in the vicinity of the electrolyte/anode and electrode/cathode interfaces are discussed with respect to processing, electro-chemical activity and related Cr-species accumulation. High-energy diffraction is also used to study phase and strain distributions of thermal barrier coatings (TBCs). When applied to turbine blades, TBCs permit higher engine operating temperatures, with concomitant increases in energy efficiency. However, the high temperatures are often combined with harsh operating environments, which have led to challenges for these coating systems. Accordingly, we examine the microstructural evolution of TBCs upon exposure to high temperatures and relevant reactive materials, to improve understanding of potential degradation mechanisms.



## Pb-Free Solders and Emerging Interconnect and Packaging Technologies: Mechanical Behavior, Failure Mode

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* Kwang-Lung Lin, National Cheng Kung University; Sung Kang, IBM; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Iver Anderson, Iowa State University; Fu Guo, Beijing University of Technology; Thomas Bieler, Michigan State University; Andre Lee, Michigan State University; Rajen Sidhu, Intel Corporation

Monday PM
February 15, 2010
Room: 204
Location: Washington State Convention Center

*Session Chairs:* Laura Turbini, Research In Motion; Jenq-Gong Duh, National Tsing Hua University

**2:00 PM**
**Effects of POSS on Impact Behavior of Thermomechanically Fatigued Lead-Free Solder Joints**: *Takayuki Kobayashi*[1]; Andre Lee[1]; K.N. Subramanian[1]; [1]Michigan State University

Polyhedral oligomeric silsesquioxanes (POSS) addition to lead-free electronic solders have shown to considerably increase the residual shear strength and reliability of thermomechanically fatigued solder joints. In this study, impact tests on joints made with SAC305 and SAC105 solders, with and without POSS, were carried out. Realistic studies to evaluate the reliability of electronic devices after impact should be carried out after they have experienced different extents of thermal influences during service. Hence, impact studies on these solder joints were performed after subjecting them to different extents of thermomechanical fatigue (TMF) in (-15ºC to 150ºC), and (-55ºC to 125ºC) temperature ranges. Roles of the microstructural features and IMC morphologies that result from the imposed TMF treatments and their influences on the impact responses of SAC305 and SAC105 solder joints, with and without POSS, will be discussed.

**2:15 PM**
**Influence of Interface Reaction on Out–of–Plane Displacements during Thermal Cycling of Copper–Silicon Bond with an Indium Interlayer**: Daniel Gruber[1]; *Nagaraj Chelliah Machavallavan*[1]; Jia Liu[2]; Indranath Dutta[2]; Rishi Raj[1]; [1]University of Colorado; [2]Washington State University

Out of plane displacement field on the surface of a silicon wafer bonded to a thick copper substrate with a thin indium layer is measured as a function of the shape of the heating and cooling cycle. The largest bending strains are measured when cooling down from the elevated temperature. The shear lag model is employed to understand the relaxation of strains induced by the difference in the thermal expansion of copper and silicon. The time dependent propagation of the shear relaxation from the edge of the silicon wafer is analyzed and correlated with the experiments. The influence of the a reaction between copper and indium, which forms intermetallics, on the stress relaxation behavior is reported.

**2:30 PM**
**The Fracture Behavior of Aged Sn-Ag-Cu Solder Joints during High-Strain Rate Loading**: *Taehoon You*[1]; Heylim Choi[1]; Eunsik Kim[2]; Jungtak Moon[2]; Heeman Choi[1]; [1]Kookmin University; [2]MK Electron

Lead-free solders, particularly Sn-Ag-Cu, are becoming more and more popular in the electronic packaging industry, due to the environmental concerns associated with lead. However, the fact that they are stiffer and more brittle also makes them more prone to brittle failure during impact loading, which is frequently encountered in microelectronic packages for handheld electronic devices. This study assesses the reliability of eutectic Sn-Pb, Sn-1.0Ag-0.5Cu, Sn-3.0Ag-0.5Cu and Sn-4.0Ag-0.5Cu solder balls with and without an aging treatment at 150ºC for various times; this study focuses primarily on how the pad surface finish and solder alloy composition affects the reliability of solder joints using a high-speed ball pull test method. Also investigated is the effect of selected rare-earth dopants on the pull strength of a Sn-1.0Ag-0.5Cu solder joint under a high-strain rate condition with and without aging treatment. The fracture forces and failure mechanisms are also examined.

**2:45 PM**
**Synchrotron Microdiffraction Study of Localized Stress and Surface Evolution in Sn-Cu System**: Nitin Jadhav[1]; Eric Buchovecky[1]; Wenjun Liu[2]; Jon Tischler[3]; Gene Ice[3]; Allan Bower[1]; *Eric Chason*[1]; [1]Brown University; [2]Argonne National Laboratory; [3]Oak Ridge National Lab

It is generally understood that whiskers are the result of compressive stress build up in Sn layers on Cu, but the stress distribution within the Sn layer and how it relaxes due to whisker formation are not clear. We have done in-situ studies on the Sn-Cu system using synchrotron X-ray microdiffraction to develop an atomic-level understand of the relationship among the IMC growth, stress and whisker formation. Spatially-resolved Sn and Cu fluorescence maps quantify the local rate of IMC formation and whisker development. By overlaying the IMC and Sn stress maps we can see the effect of IMC formation on localized stress in the Sn layer and the dependence of whisker nucleation on Sn grain orientation and localized stress. Based on insight developed, we present results of finite-element simulations that include stress relaxation in the Sn due to both plastic deformation and through Sn diffusion along Sn grain boundary.

**3:00 PM**
**Effect of Zn-Containing Flux on the Joint Strength and Microstructure of Sn-3.5Ag Soldering on an Electroless Ni-Au Surface Finish**: *Hitoshi Sakurai*[1]; Youichi Kukimoto[2]; Katsuaki Suganuma[1]; [1]Osaka University; [2]Harima Chemicals, Inc.

The joint strength and microstructure for Sn-3.5Ag soldering on an electroless Ni-Au pad by using the new flux containing Zn(II) stearate were investigated. The content of the zinc compound in a flux varies from 0 wt.% to 50 wt.%. The results of a bump pull test showed that the Zn-containing flux gave higher joint strength than did a conventional flux without Zn compound. The study of the interfacial microstructure revealed that the thickness of the interfacial intermetallics layer became thinner with increasing Zn content in a flux. In addition to this, the new flux gave a thinner P-rich layer at the interface than that seen with the conventional flux. It is assumed that Zn derived from a flux affects an interfacial reaction which correlates with the suppression of P-rich layer, and this may contribute to the improvement of the joint strength obtained by using the Zn-containing flux.

**3:15 PM Break**

**3:30 PM**
**Electrochemical Corrosion Behavior of HighTemperature Pb-Free Solders**: *Chi-Hang Tsai*[1]; Jenn-Ming Song[1]; [1]National Dong Hwa University

This study aims to investigate the electrochemical corrosion behavior of the potential Pb-free solders, Bi-11wt%Ag and Zn-40wt%Sn, in 3.5% NaCl solution using the potentiodynamic polarization method with the scanning range from -2000mV to +2000mV. Pb-5wt%Sn alloy is also examined for comparison. Experimental results show that the corrosion potential (Ecorr) decreases in turns from Bi-11Ag, Pb-5Sn, Zn-40Sn to Zn-40Sn-0.5Al. Bi-11Ag exhibits the highest current density (Icorr), and that for Pb-5Sn is the lowest. The Pb-5Sn samples have a much extended passive region compared to Bi-11Ag, while the Zn-40Sn samples show no passive behavior. According to the XPS data, the corrosion products are PbCl2 and PbO for Pb-5Sn, BiOCl, AgCl2, and Bi2O3 for Bi-11Ag, and ZnCl2, ZnO, SnO, SnO2 for Zn-40Sn. Furthermore, it is found that a small amount of Al addition improved the corrosion resistance of Zn-40Sn.

**3:45 PM**

**Joint Strength Enhancement by CNT Inserted Sn3.5Ag Solder Balls**: *Young-Ki Ko*[1]; Yoon-Ki Sa[1]; Jung-Hwan Bang[1]; Jeong-Han Kim[1]; Chang-Woo Lee[1]; Sehoon Yoo[1]; [1]KITECH/Micro-Joining Center

CNTs were inserted onto the surface of Sn-3.5Ag solder balls by using simple ball milling process. Sn-3.5Ag solder balls with diameter of 450 um were mixed in the CNT dispersed solution. Ball mill times were varied with 6, 12, 24, and 36 hours. Even after 24hrs of ball milling, the shape and size of Sn-3.5Ag solder balls were not changed. The amount of surface-inserted CNTs increased as the ball mill time increased. CNT-inserted Sn-3.5Ag solder balls were attached on ENIG finished copper pads by reflow process. Intermetallic compound layer thickness of CNT-inserted solder balls were not different from that of non-mixed solder balls. Shear strength of CNT inserted Sn3.5Ag solder balls increased by 10 % after 24 hour-ball mill process. From the fracture surface observation after the shear test, fracture occurred at solder region, not at the intermetallic layer.

**4:00 PM**

**Intermetallic Compounds and Mechanical Properties of Sn-3Ag-0.5Cu and Sn-1Ag-0.5Cu-0.06Ni-0.01Ge Solder Ball Grid Array Packages with ENIG Surface Finish**: *Chin-Liang Chen*[1]; Jim Wang[2]; Tung-Han Chuang[1]; [1]National Taiwan University; [2]SHENMAO Technology Inc.

Due to concerns about the material costs of Sn-3Ag-0.5Cu (SAC305) solder, the Ag content of this alloy should be decreased; thus, a Sn-1Ag-0.5Cu-0.06Ni-0.01Ge (SAC105) has been suggested. Tensile tests show that the SAC105 alloy possesses lower ultimate strength and greater elongation in comparison to SAC305 alloy. Further studies on the reflowed and aged solder joints in BGA packages with Au/Ni/Cu pads using both alloys indicate that the $(Cu, Ni)_6Sn_5$ interfacial intermetallics layers in SAC105 specimens are twice as thick as those in SAC305, and that the morphology of the latter is continuous, in contrast to the coarse scallop-shape of the former. For evaluation of the bonding strength of solder joints with this solder, the ball shear test (BS), high speed ball shear test (HS-BS), and ball pull test (BP) were employed. The fractography of specimens after BS and BP tests revealed ductile fractures through the solder balls and the HS-BS tests resulted in brittle fractures along the solder/pad interfaces, and the bonding strengths of SAC105 solder joints in all cases were lower than those of SAC305.

**4:15 PM**

**Internal Strain Evolution during Thermal Cycling in a Row of Lead-Free Solder Joints in a Flip Chip Ball Grid Array Package**: *Bite Zhou*[1]; Thomas Bieler[1]; Guilin Wu[2]; Stefan Zaefferer[2]; Tae-Kyu Lee[3]; Kuo-Chuan Liu[3]; [1]Michigan State University; [2]Max-Planck Institut für Eisenforschung; [3]Cisco Systems, Inc.

In-situ measurement of transmission diffraction patterns during thermal cycling of a single row from a 44x44 flip chip ball grid array package were analyzed to characterize internal strain evolution. The analyzed joints were in the ball grid array with a 1 mm pitch and an 18x18 mm die. The as-assembled package on its printed circuit board was sliced to provide a row that went through the center of the package. A thermal cycling stage was placed in a synchrotron beamline so that patterns could be measured periodically during a thermal cycle, in about 20% single, 40% bi-, and 40% tri-crystals (the method was confirmed with serial sectioning on other samples). Due to the different orientations in each joint, the effect of the anisotropic expansion coefficient on strain history in each in each of the crystal orientations is analyzed to identify how crystal orientations affect strain history during a thermal cycle.

**4:30 PM**

**Mechanical Shock of Environmentally-Benign Pb-Free Solders**: *Kyle Yazzie*[1]; Huiyang Fei[1]; Jason Williams[1]; Dallas Kingsbury[1]; Hanqing Jiang[1]; Pedro Peralta[1]; Nik Chawla[1]; [1]Arizona State University, School of Mechanical, Aerospace, Chemical, and Materials Engineering

Pb-free solder alloys are subjected to mechanical shock and drop conditions in service. A fundamental understanding of intermediate strain rate behavior (10-50 s-1) is lacking. Existing methods developed to probe this strain-rate regime do not provide a direct and local measurement of strain in the solder. Quantifying the contributions of intermetallic thickness and solder microstructure to the mechanical shock behavior of the solder specimen is extremely important and needs to be studied. In this study the mechanical behavior of pure Sn and Sn-3.5Ag-0.7Cu solders was systematically quantified at strain-rates of 0.001 s-1 to 30 s-1. Digital image correlation was used in conjunction with high-speed video to measure global and local strain fields. A novel mirror configuration was used to measure the instantaneous cross-sectional area for the measurement of true stress-true strain. Multiscale modeling of the complex stress state experienced by solder during mechanical shock was conducted and will be presented.

**4:45 PM**

**Fatigue-Creep Interaction Damage Theory Based Thermal Fatigue Life Prediction Model for SnAgCu Solder Joints**: Yan Chang[1]; *Li Yan*[1]; Liu Na[1]; [1]Beijing University of Technology

SnAgCu solder is fast becoming a reality in electronic manufacturingduring to marketing and legislative pressure. This paper studies thermal fatigue life prediction model for SnAgCu solder on basis of classic continuous damage mechanism (CDM). In order to imitate the service condition of chip, the single point model coupling temperature and creep is adopted. The value of resistance is defined as the damage variable. As the value of stress or strain is difficult to be measured, FEM is applied to calculate the value of stress and strain. Then the constant in damage model is fitted by the experiment data. Base on the damage evolution equation, the life of solder joint is calculated and the result is compared to actual life of solder joint. It can be seen that difference between the prediction life and tests is no more than 10.2%.

## Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials IX: Session II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Alloy Phases Committee

*Program Organizers:* Chih-ming Chen, National Chung Hsing University; Srinivas Chada, Medtronic; Sinn-wen Chen, National Tsing-Hua University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-ho Lee, Hongik University; Kejun Zeng, Texas Instruments; Yee-wen Yen, National Taiwan University of Science and Technology; Wojciech Gierlotka, AGH University of Science and Technology; Chao-hong Wang, National Chung Cheng University

Monday PM — Room: 203
February 15, 2010 — Location: Washington State Convention Center

*Session Chairs:* Jae-Ho Lee, Hongik University; Chao-hong Wang, National Chung Cheng University

**2:00 PM Invited**

**Interfacial Reactions in the Sn-Co/Ni and Sn-Cu-Co/Ni Couples**: Yu-kai Chen[1]; *Sinn-wen Chen*[1]; [1]National Tsing Hua University

Sn-Cu-Co alloys are promising Pb-free solders, and Ni is the most commonly used barrier layer. Interfacial reactions in the Sn-Co/Ni and Sn-Cu-Co/Ni couples are examined. Ni3Sn4 and (Ni, Co)Sn4 phases are formed in the early stage in the Sn-yCo/Ni couples reacted at 250oC where y varies from 0.06wt% to 0.4wt%. The Sn concentrations in the (Ni,Co)Sn4 phase decrease with longer reaction time, and it becomes (Ni,Co)Sn4-x. With even longer reaction time, the (Ni,Co)Sn4-x phase cracks and detaches from the interface, and the Ni3Sn4 phase becomes the stable reaction product. There are at least two different competing reactions in the Sn-Cu-Co/Ni couples. For example, the interfacial reactions in the Sn-0.7wt%Cu-0.05wt%Co/Ni couples are similar to those in the Sn-0.7wt%Cu/Ni couples, and the η-Cu6Sn5 is the reaction phase. The reaction phases in the Sn-0.3wt%Cu-0.05wt%Co/Ni are Ni3Sn4 and (Ni, Co)Sn4-x, and the results are similar to those in the Sn-Co/Ni couples.

**2:25 PM**

**Interfacial Reactions of Co/Sn/Cu Sandwich Structure**: *Chao-hong Wang*[1]; Chun-yi Kuo[1]; [1]National Chung Cheng University

In flip-chip packaging, Sn-based solder bumps are used to connect integrated circuit (I.C.) chip and Cu pad substrate. Co and Co-based alloys are the potential materials for under bump metallurgy (UBM) of I.C. chip. Thus, the designed experiments are conducted in this study to investigate the interfacial reactions of Co/Sn/Cu sandwich structure. The Co/Sn/Cu couples were electroplated on Cu substrates at various Sn layer thicknesses of 75μm to 600μm. The reactions

were carried out at various temperatures, 150°C and 200°C, respectively. For the Sn/Cu interface, $Cu_6Sn_5$ and $Cu_3Sn$ are formed, which is similar to those of Sn/Cu interfacial reactions without Co layer. For the Co/Sn interface, two reaction phases, $Co_3Sn$ and $(Cu,Co)_6Sn_5$, are simultaneously observed. Remarkably, the $Co_3Sn$ reaction layer is much thinner than that of Sn/Co couple without Cu layer. The results in the present study indicate the $Co_3Sn$ growth is significantly inhibited in Co/Sn/Cu sandwich couple.

**2:45 PM**

**Effects of Mechanically Applied Stress on Solid-State Sn/Cu Interfacial Reaction**: *Chi-pu Lin*[1]; Chih-ming Chen[1]; [1]National Chung-Hsing University

Plastic boards have been widely employed as the substrates for flexible printed-circuit board (PCB) due to their flexibility. The flexible PCB is usually bended in order to reduce the packaging volume, which makes the solder joints subjecting to tensile or compressive stress. Sn is the primary element of Pb-containing and Pb-free solders and Cu is commonly used as a metallization layer for flexible PCB. In this study, Sn layer was electrodeposited on a thin Cu foil and solid-state interfacial reactions of the Sn/Cu joints subjected to tensile or compressive stress were investigated. The samples were bended upward or downward to apply tensile or compressive stress on the joint interface, respectively. Formation and growth behavior of the reaction products at the Sn/Cu interface were investigated and the correlation with the mechanically applied stresses was also discussed.

**3:05 PM**

**Stress Effect Study on Sn-Cu Intermetallic Formation by Four Point Bending Method**: Chuo-Cheng Yang[1]; *Ya-Chi Cheng*[1]; Chi-Jia Tong[1]; Ming Tzer Lin[1]; [1]National Chung Hsing University

In microelectronics, the fabrication of a reliable solder joint plays a very important role. In order to obtain better understanding on the effect of stress state related to interfacial IMC layer growth, custom design four point bend experiments had been conduct. Double polished silicon wafers were cut into strips with 27mm in length and 5mm in width as test samples. Electroplate 10um copper layer on silicon and deposit 30um tin layer on top of it. During the experiment, a set of samples were put into furnace for 168 hours at 200°X and relatively low levels of in-plane bending stress were applied on the samples under tensile, compressive stress and without stress. The results on intermetallic formation affected by different stress levels (10MPa, 20MPa and 50MPa) were presented. Both tensile and compressive stresses effects on the Cu-Sn intermetallic formation were observed and increased with increased the amplitude of the stress.

**3:25 PM**

**Correlation between Solder Wettability and Surface Properties of Deformed Cu Foils**: *Yu-Hsiang Hsiao*[1]; Chengyi Liu[1]; [1]National Central University

Typically, a so-called hardening surface layer would form on the Cu foil after mechanical process. This thin surface layer has very different properties from the bulk Cu foil. With different degrees of mechanical process, such as, cold-roll, the surface layers exhibit various properties. Nano-indentor and GIXRD were used to analyze the properties on the surface of Cu foils with different cold-roll percentages. We found that the hardening surface layer thicken with the cold-roll percentage. With the aid of the nano-indentor and GIXRD analysis, we can correlate the results of the surface properties of the cold-rolled Cu substrates with the solder wettability. According to the preliminary results, we found that the mechanical property of the very surface layer of the cold-rolled Cu foil has a strong influence on the solder wettability. In this talk, the detail relationship between solder wettability and Cu surface properties will be presented.

**3:45 PM Break**

**4:05 PM Invited**

**Suppressing the Sn-Patch Growth in Ti/Ni(V)/Cu under Bump Metallization with Snagcu Solder after Aging**: Kai-Jheng Wang[1]; *Jenq-Gong Duh*[1]; [1]National Tsing Hua University

The sputtered Ti/Ni(V)/Cu under bump metallization (UBM) is widely used in flip chip technology, owing to the non-magnetic metals and low consumption rate. It was noted that V did not react with solders and intermetallic compounds (IMC) during reflow and aging, yet a Sn-patch would form in the Ni(V) layer. However, the Sn-patch growth may cause the IMCs detaching from the interface of solder joints. In this study, the Sn3.0Ag0.5Cu solder was reflowed on the Ti/Ni(V)/Cu UBM with various Cu thickness at 250 C for 60 s, and then aged at 150 C for various periods of duration. It was revealed that the Sn-patch growth could be controlled by Cu thickness in the Ti/Ni(V)/Cu UBM. Therefore, a feasible approach to suppress the Sn-patch formation was discussed and proposed.

**4:30 PM**

**Interfacial Reactions of Sn-0.7Cu,Sn-9Zn and Sn-58Bi Lead-Free Solders with the Au/Ni/SUS304 Substrate**: *Yee-wen Yen*[1]; Kuen-da Chen[1]; Wei-kai Liou[1]; [1]National Taiwan University of Science and Technology

This study investigates the interfacial reactions between three kinds of lead-free solders, Sn-0.7 wt%Cu (SC), Sn-58 wt%Bi (SB) and Sn-9 wt%Zn (SZ), and Au/Ni/SUS 304 substrates. According to experimental results, only the (Cu, Ni)6Sn5 phase with a hexagonal-type structure was formed in the SC/Au/Ni/SUS 304 couples. It should be the. When the Ni layer was completely consumed, the massive spalling of the (Cu, Ni)6Sn5 phase was found in the solder. When the (Cu, Ni)6Sn5 phase was spread over the interface, the SUS304 substrate surface could directly contact the SC solder and then the FeSn2 phase with a platy-type layer was formed at the interface. In the SB/Au//Ni/SUS304 couple, only the Ni3Sn4 phase with a needle type layer was found at the SB/Au/Ni/SUS 304 interface. The Ni5Zn21 phase with a layered structure was formed at the SZ/Au//Ni/SUS 304 interface.

**4:50 PM**

**Influence of Palladium Thickness on the Solderability between Sn3Ag0.5Cu and Au/Pd/Ni(P) Surface Finish**: *Wei-Hsiang Wu*[1]; S. P. Peng[1]; C. H. Lin[1]; C. E. Ho[1]; [1]Yuan Ze University

Electroless nickel/immersion gold (ENIG) process has been widely accepted for depositing a viable surface finish over the Cu, providing a reliable soldering and wire-bonding pads in the high-end applications. However, the galvanic hyper-corrosion of the electroless nickel induced by the gold plating process will cause the so-called "black pad", which might embrittle the interfacial strength and deteriorate the overall reliability of one package. In order to solve this problem completely, modifications to ENIG by plating one additional Pd layer in-between, preventing the gold plating bath to attack Ni, are being considered and adopted by industry. The present study was conducted to evaluate the solderability of Sn3Ag0.5Cu with Au/Pd/Ni(P) having various Pd thicknesses (0-0.3 microns). The reliability of the joints was examined using a high-speed ball shear test with a shear speed of 2 m/s. The correlation between the interfacial strength and various Pd thicknesses will be presented in this study.

**5:10 PM**

**Electroless Nickel Plating on Porous Medium**: So-Young Chun[1]; Young-Mok Rhym[2]; *Jae-Ho Lee*[1]; [1]Hongik University; [2]Korea Institute of Materials Science

Electroless nickel plating is widely used technique in solder and packaging industries. In most cases, electroless nickel plating was applied on the open metallic surfaces and the rate of deposition was controlled with temperature and time. However, when the electroless plating is applied on the porous medium, the rate of deposition is also dependent on the activation process and mass flow of electrolyte prior to the electroless plating. In this research, electroless nickel plating on the porous medium was investigated. The carbon/polymer was selected as the substrate. Hydrophobic surface prevented the penetration of solution into the porous medium. Wettability of the substrate was improved by surface treatment prior to the activation process. The concentration of activating solution was varied to give the uniform palladium seeds. The optimum concentration of activating solution was also investigated. The rate of deposition in the porous medium was investigated with time and reducing agent concentration.

## Processing Materials for Properties: Agglomerates and Composite Materials Processing

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division
*Program Organizers:* Brajendra Mishra, Colorado School of Mines; Akio Fuwa, Waseda University; Paritud Bhandhubanyong, National Metal and Materials Technology Center

Monday PM Room: 617
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* David Olson, Colorado School of Mines; Greg Oberson, U.S. Nuclear Regulatory Commission

**2:00 PM Keynote**
**Innovations in Processing of Lightweight Metal Matrix Composites**: *Ramana Reddy*[1]; [1]The University of Alabama

Newly emerging chemical in situ process is economical and energy efficient for large scale manufacturing of particulate metal matrix composites compared to the conventional (physical ex situ) processes. This presentation concentrates on some of the key examples from the author's experience and others, application of engineering fundamentals on the in situ processing of nanoparticulates of discontinuously reinforced Al and Mg alloys metal matrix composites. Aluminum and Magnesium alloys were reinforced with AlN nanoparticles using chemical in situ process. Thermodynamic analyses were made to identify the conditions for the in situ formation of the AlN in Al and Mg alloys. Effect of experimental processing parameters (i.e. time of gas injection, concentration of ammonia, and temperature of the melt) on the formation of AlN nanoparticles in alloy was determined. The AlN quantities in the composites varying from 5 to 51 wt % were produced. Increase in either injection time or flow rate of the ammonia gas increased the nitride content. AlN particles with an average size of 400 nm were produced. The measured Vickers hardness of the composites formed increased with increasing AlN content. The amount of AlN experimentally formed is in good agreement with the thermodynamically predicted data. A kinetic rate equation of in situ formation of AlN nanoparticulates in lightweight metal matrix composites was developed. A possible reaction mechanism of AlN formation was proposed. The industrial applications of this in situ manufacturing of composites by molten metal technology were discussed.

**2:30 PM**
**Surface Peening Morphology Evaluation in Anodized Aluminum Alloy Rotors**: *M. Bilal Khan*[1]; [1]SCME NUST

Correspondence between photoelastic stress and stress bearing capacity by shot peening on the tip of a light weight turbine rotor. SEM investigates the treated tip and the bulk of the rotor. EDS reveals the coarse features of silicon shots reinforced Al-alloy at the tip with anodized Al sealed with hot water in the bulk of the rear profile. AFM measures 3D flake surface topography of the treated tip. The hole-drill method predicts the distribution of stresses at the profile. Measurements are performed on an aerodynamic guide module under static pressure. High tensile residual stress in pressure die cast LM24 alloy convert to compressive mode by similar treatment. Pro Cast predicts the stress across the fin portion of the cast part. Radiography shows that Level 4 porosity exists in the thicker portion of the cast part due to the steep temperature gradient accompanied by the drastic dimensional changes, (~3.0-10 mm).

**2:50 PM**
**Processing of Mullite and Mullite-Zirconia Composites Via Microwave Sintering**: *Subhadip Bodhak*[1]; Susmita Bose[1]; Amit Bandyopadhyay[1]; [1]Washington State University

The objective of this research is to evaluate microwave sintering as a viable option to process high strength mullite and mullite-zirconia composites utilizing reduced time and energy. Mullite samples were sintered using a 3 KW, 2.45 GHz microwave furnace. With an increase in sintering temperature from 1400°C to 1500°C, the porosity decreased from 30 to 13% and the compressive strength increased from 128±18 MPa to 387±21MPa when 1wt% MgO was added as a sintering aid to mullite. Furthermore, yttria stabilized tetragonal zirconia (YTZP) was incorporated (up to 20wt%) to prepare zirconia toughened mullite composites. A maximum hardness of 10.24±0.61 GPa, compressive strength of 740±38 MPa and indentation fracture toughness of 3.65±0.43 MPam1/2 was obtained for Mullite with 1wt% MgO and 10wt% ZrO2 composites sintered at 1500°C in microwave furnace. Microwave sintering data, when compared with those of conventional sintering, reveal that localized heating of microwaves significantly enhanced the mechanical properties.

**3:10 PM**
**SHS Synthesized Aluminum-Titanium Carbide MMC Die Casting Alloys**: William Garrett[1]; Cosan Unuvar[1]; *John Moore*[1]; [1]Colorado School of Mines

Self-propagating High-temperature Synthesis utilizes the local heat of an energetic solid-state reaction to propagate the reaction through a mixed chemical body. For aluminum-titanium carbide composites the SHS process forms titanium carbide from elemental titanium and carbon powders as they are added to an aluminum melt which is then die cast. The SHS synthesis of TiC in aluminum increases the workable volume fraction of TiC (30-60 Vol%) in the aluminum alloy and reduces oxide impurities at the Al/TiC interface. The engineered properties design target is to match the wear, hardness, and compression strength properties of cast iron with the Al/TiC composite. Reducing the ignition temperature of the titanium/carbon reaction is done by adding low ignition temperature, chemical oven reactants to the elemental Ti and C powders and is important to reducing the cost of the manufacturing process and equipment and adapting the process for magnesium/TiC composites.

**3:30 PM**
**Compaction Behavior of Aggregated and Agglomerated Nano-Powder Using Discrete Element Method**: *Avinash Balakrishnan*[1]; Christophe Martin[1]; [1]Grenoble-INP

Why are powders made of nano-particles so difficult to compact? We attempt to answer this question using 3-D discrete simulations in which the adhesive forces linked to van der Waals interactions are explicitly taken into account at the particle length scale. Our simulations show that indeed, there is a significant effect of particle size and interparticle friction on the macroscopic stress necessary to attain a given relative density. The homogeneity of the compacted microstructure is also greatly affected by size effects. We show that aggregates exhibit a brittle to plastic transition in their crushing behavior as the particle size decreases. We demonstrate that both normal and tangential interparticle contact forces are affected by adhesive effects and that their coupling is responsible for the difficulty to compact these powders. Finally, we investigate the effect of compaction pressure and particle size on the tensile strength of green compacts made of sub-micronic particles.

**3:50 PM**
**Effects of Particle Size, Deformation and Heat Treatment Processing on Strength and Ductility of Aluminum-Iron Composite**: *Samson Adeosun*[1]; Sanmbo Balogun[1]; Fidelia Ochulor[1]; Wasiu Ayoola[1]; Olatunde Sekunowo[1]; [1]University of Lagos, Akoka

This paper presents new evidence for enhancement of strength and ductility of aluminum 1200 alloy through the addition of steel particles. Iron particles of sizes 106,181,256,362.5 and 512.5 µm were added to the aluminum alloy. The samples produced were homogenized at 4200C for 10 hours and further processed through upset forging at 320C and annealed at 4700C for 8 hours. Tensile strength of 280MPa and ductility of 1.75 were achieved in the aluminum alloy with coarse particles of Fe (106µm) at 10% reduction when forged and subsequently annealed. With 50% reduction and finer-sized particles of Fe, strength dropped to 160MPa without significant decrease in ductility (1.7). The presence of fine Fe particles at grain boundaries after annealing with most of the particles in solid solution is responsible for the drop in strength.Keywords: Iron particle, forging, annealing, strength and ductility, aluminum alloy.

**4:10 PM**
**Features of a Structure and Properties of the Agglomerates Obtained from Rich Ores**: *Sereda Borys*[1]; Irina Kruglyak[1]; Aleksandr Zherebtsov[1]; [1]ZSEA

Structural - textural features of agglomerate structure obtained from rich ores deposits have been described in this work. The formation and cross-feeding of different structures (breciated, porphyry, eutectic type) of sintered agglomerates have been described. The mechanism of structures changes in the block of fluxed agglomerate from periphery to the center at different fuel content has been considered. Influence of original sintering composition on a structure and properties of obtained agglomerates has been established. It is determined that durability and reducibility is depended from good crystallization, structural

porosity homogeneousness at basicity rate 1.8 – 2.0. At low basicity and large fuel content in ore mixture the high durability of agglomerates is observed too.

**4:30 PM**

*In situ* Neutron Diffraction Study of Solvent-Free Fabrication of Ferromagnetic Core-Shell $Fe_3O_4$-Carbon Nanocomposite: *Sven Vogel*[1]; Vilas Pol[2]; Luke Daemen[1]; George Chertkov[1]; [1]Los Alamos National Laboratory; [2]Argonne National Laboratory

The synthesis of nanomaterials is the subject of intense current research since the nanoparticles of interest to applications are only prepared in the laboratory and are difficult to produce in a monodisperse form. Most of the current processes are complex and synthesize nanomaterials to varying degrees of commercially-viable quantities. Pol et al. developed a completely new facile synthesis approach called RAPET [*R*eaction under *A*utogenic *P*ressure at *E*levated *T*emperature] for the nano-synthesis of metals, oxides, carbides, phosphides, sulfides etc. However, it has always remained a challenge to understand the exact dissociation stage of the chemical precursor under autogenic pressure at elevated temperature and the exact arena of segregation or formation of nanostructures. Using neutron diffraction, we could study the thermolysis of a single deuterated precursor, $Fe_3(CH_3COO)_6(OH)_2CH_3COO$, in a closed reactor in situ at temperatures up to 700\176C. The reaction produced ferromagnetic core-shell $Fe_3O_4$-carbon nano-composite without need for a solvent or catalyst.

**4:50 PM**

**Thermal Properties of Advanced Diamond - Metal Matrix Composites**: *Vikas Sinha*[1]; Jessica Remmert[2]; Sabyasachi Ganguli[3]; Robert Wheeler[1]; Jonathan Spowart[4]; [1]UES, Inc.; [2]UTC; [3]UDRI; [4]Air Force Research Laboratory

A reliable operation as well as further improvements in performance of advanced electronic components requires a credible thermal management strategy. A high thermal conductivity and a tailorable coefficient of thermal expansion are important attributes of candidate substrate/ packaging materials for high power density electronic components. Several diamond containing metal matrix composites have been developed to generate this combination of properties. Diamond-metal matrix composites were fabricated and variables included metal matrix chemistry as well as volume % of diamond. The thermal conductivity for different material conditions was measured via laser flash techniques. Attempts were also made to directly measure the interface thermal conductance via time-domain thermoreflectance techniques. To aid in improved understanding of interfacial thermal transport, microstructural characterization of interface region was also carried out. The chemistry changes near the interface were evaluated via energy dispersive spectroscopy and/ or electron energy loss spectroscopy techniques in a transmission electron microscope.

**5:10 PM**

**Effect of Proess Parameters on Powering Characteristics of Galavnneald Materials**: *Ram Janam Singh*[1]; Khursid Khan[2]; Shantanu Chakrabarti[2]; [1]NIT Jamshedpur; [2]Tata Steel

Globally, the use of metallic coated steel sheets in the automobile industry is increasing. The sheet should have excellent formability, weldability, paintability and corrosion resistance. Since bare cold rolled steel sheets have certain limitations, application of galvanised steel sheets is steadily expanding. Galvannealed steel sheet fulfills all requirments and has become of major interest in the automobile industry. The success of Galvannealed (GA) steels in the body-in-white depends upon material design and parameters of various processes involved, like cold rolling, continuous annealing, hot dipping and galvannealing. In the present work, powdering resistance, one of the important product characteristics of GA, has been studied and a relation is established between the process parameters and the powdering value. The product obtained through optimisation of the process parameters has been successfully used by major automotive producers without any powdering problem, for internal as well as external automotive applications.

**5:30 PM**

**Porous Superelastic NiTi Produced by Sintering with NaCl Space-Holders**: *Ampika Bansiddhi*[1]; David Dunand[2]; [1]Kasetsart University; [2]Northwestern University

Porous NiTi with ~30-40% porosity was fabricated by densification of NiTi powder with temporary NaCl space-holders. The porosity and pore characteristics (pore size, shape and connectivity) were controllable through the initial volume fraction and geometry of the space holders. The NiTi foams exhibited shape recovery behavior at room temperature and at body temperature. High compressive yield strength with low stiffness and large recovery strain were observed. Simple, cost-effective processing and post-processing procedures were demonstrated to achieve a desired porous structure, and hence a desired mechanical and shape recovery behavior. By combining the unique properties of NiTi with an adjustable foam architecture, NiTi foams can be custom-designed for multi-functional applications such as impedance-matching connectors between structural parts, energy-absorbing structures, actuators, and bone implants.

**5:50 PM**

**Experimental Investigations of the Ti-Fe- Eutectic System Needed for the Further Understanding of the Microstructural Evolution in an Eutectic Alloy at Different Cooling Rates**: *Antje Schlieter*[1]; Uta Kühn[1]; Martin Friak[2]; Juergen Hubert[3]; Heike Emmerich[3]; Joerg Neugebauer[2]; Juergen Eckert[1]; [1]IFW Dresden; [2]MPI Düsseldorf; [3]RWTH AAchen

Ti-based alloys have been suggested for commercial applications with a great potential due to their high strength (~1000 MPa) and good corrosion resistance. The strength of these alloys can be further increased by producing a nano/ ultrafine grained and lamella structured Ti-eutectic. In this paper we focused on the influence of the different cooling rates due to the several casting techniques (arc melting, cold crucible, tilt casting, injection casting and Bridgeman technic) on the Ti-Fe-eutectic alloy. The evolution of the microstructure, the phase constituents, the chemical composition linked to the mechanical properties and elastic constants depending on lamellar growth with multiple orientation will be presented.To achieve this goal a joint approach, where the detected experimental data are systematically cross-checked against the phase-field simulations with parameters determined from ab initio calculations, is adopted.

## Refractory Metals 2010: Processing and Properties II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Refractory Metals Committee
*Program Organizers:* Brian Cockeram, Bechtel-Bettis; Gary Rozak, H.C. Stark

Monday PM Room: 2A
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Brian Cockeram, Bechtel Marine Propulsion Corporation; Gary Rozak, H. C. Starck, Inc.

**2:00 PM**

**Tantalum Plates with Controlled Texture**: *Dincer Bozkaya*[1]; Peter Jepson[1]; [1]H.C. Starck Inc.

Texture control of tantalum plates used as sputtering targets is crucial for the uniformity of thin films as the sputtering rate at a given location is a strong function of grain orientation. In addition, texture of plates and sheets may also be critical in other applications such as forming. In this paper, a new grade of tantalum plate with improved uniformity of texture radially and through thickness is introduced. The thermomechanical processes utilized for breaking up the ingot structure of electron-beam melted tantalum are described. A novel rolling process for obtaining the desired, uniform texture in final plates and sheets is explained.

**2:25 PM**

**Analysis of Tantalum Taylor Impact Specimens**: *Joel House*[1]; John Bingert[2]; Philip Flater[1]; James O'Brien[3]; William Hosford[4]; Robert DeAngelis[5]; Richard Harris[1]; [1]Air Force Research Laboratory; [2]Los Alamos National Laboratories; [3]O'Brien and Associates; [4]University of Michigan; [5]University of Florida

Taylor impact experiments were conducted on commercially pure Ta subjected to thermo-mechanical processing including Equal Channel Angular Processing (ECAP), forging and annealing. The microstructures were characterized by optical microscopy and Electron-Backscattered Diffraction (EBSD) before and after the impact experiments. Specimens of the material were tested in the cold worked and recrystallized conditions. The final geometry of recovered specimens was obtained to characterize the plastic anisotropy. The EBSD data sets from the recovered impact specimens revealed the texture evolution in the specimen as measured along the axial length. These data show the evolution of the <111>, and <100> texture components as they rotate toward the compression

axis of the sample. The final specimen geometry, optical microscopy and EBSD characterization reveal the affect of processing history on the high strain rate deformation in the Taylor impact experiment.

**2:50 PM**

**Comparison of Optimized Finite Element Crystal Plasticity Model and Tensile Tests of Niobium Single Crystals**: *Derek Baars*[1]; Payam Darbandi[1]; Chris Compton[2]; Wenjun Liu[3]; Rozaliya Barabash[4]; Thomas Bieler[1]; [1]Michigan State University; [2]National Superconducting Cyclotron Laboratory; [3]Advanced Photon Source Argonne National Laboratory; [4]Oak Ridge National Laboratory

Manufacturing superconducting radio frequency (SRF) cavities from single or multi-crystal niobium is being investigated as an alternative to using polycrystalline niobium, as single crystal sheets may be cut directly from a purified ingot, eliminating the cost of forging and rolling ingots into polycrystalline sheet. Simulation of deformation of single or multi-crystal parts is desirable, but the data needed to develop single crystal constitutive models are lacking. Selected crystal orientations to resolve shear stress on specific slip systems during uniaxial tension are used to identify orientation dependent critical resolved shear stresses and work hardening parameters. Active slip systems are identified using orientation imaging microscopy, electron channeling contrast imaging, and depth resolved 3-D x-ray measurements. These specimens develop shape changes that reflect activated slip system and work hardening phenomena, so optimized constitutive models can be developed to predict shape changes and observed activated slip systems.

**3:15 PM**

**Deformation Mechanism for Polycrystal Niobium at Cryogenic Temperature**: Payam Darbandi[1]; *Derek Baars*[2]; Saravan Chandrasekaran[3]; Farhang Pourboghrat[3]; Tom Bieler[2]; Chris Compton[4]; [1]Michigan State University; [2]Department of Chemical Engineering and Materials Science, Michigan State University; [3]Department of Mechanical Engineering, Michigan State University; [4]National Superconducting Cyclotron Laboratory, Michigan State University

Niobium has been used in superconducting cavities for several years. The cavities operate at 4.2K, as niobium becomes superconducting at temperatures below 9.2 K. Since the deformation mechanisms of niobium at 4.2 K are not completely understood, tensile testing was done at 4.2 K in liquid helium. Serrated flow of the material was observed in the stress-strain curve which literature of similar tests suggests that it is caused by twinning and not by dislocation slip alone. Orientation imaging microscopy of the polished cross-section of the specimen revealed evidence of twinning. Tensile tests were also done at 77 K in liquid nitrogen, with no serration in the stress-strain curve. Serration has been reported in similar literature, though at lower strain rates than the present test. Strain rate affects the deformation mechanism, affecting the serration behavior. The effect of strain rate on serrated flow will be discussed.

**3:40 PM Break**

**3:55 PM**

**Mechanical Properties and Constitutive Modeling of Some Refractory Metals**: *Shuh Rong Chen*[1]; G.T. (Rusty) Gray III[1]; [1]Los Alamos National Laboratory

The high density and high melting temperature of several refractory metals make them attractive candidates for defense applications. The mechanical properties of these metals are very sensitive to deformation temperature, strain rate, and the impurity contents due to its more open crystallographic structure (bcc) with a higher intrinsic barrier (Peierls stress) to dislocation movement. We will present constitutive properties on Ta, Mo, W, V, Nb, and Re obtained by compressing samples as a function of temperature from 77 to 1273K, and strain rate from 0.001/s to 3000/s. We will also present the development of more physically-based constitutive models to describe the mechanical responses of these metals to large strains. And finally we will validate the models and their corresponding parameters using high velocity Taylor cylinder impact tests by comparing results from the post-mortem plastic deformation profiles and the finite element calculations.*Work supported jointly by the U.S. Department of Energy and Department of Defense

**4:20 PM**

**Chromium Alloys for More Efficient Fossil Energy Conversion Technologies**: *Omer Dogan*[1]; Michael Gao[1]; Paul King[1]; [1]DOE National Energy Technology Laboratory

In order to improve efficiency and reduce environmental emissions in fossil energy conversion systems, new technologies such as oxy-fuel gas turbines, hydrogen turbines, and syngas turbines are being developed. These technologies will require new materials that can withstand higher temperatures. Even the state-of-the-art nickel based superalloys cannot meet these demands as they are unsuitable for use above 1150°C. Accordingly, alloys based on refractory metals such as Nb, Mo, Cr and W are being investigated as potential solutions. The materials community has been interested in Cr based alloys since 1950's due to (i) their relative low cost, (ii) relative low density and (iii) good high temperature strength. However, low ductility and fracture toughness of Cr and its alloys around room temperature kept them from a major structural application. We will review the historical data on mechanical properties and take a close look at the recent research on the Cr alloys.

**4:45 PM**

**Strength and Oxidation Resistance of Mo-Si-B Alloys Produced by Reaction Synthesis**: *Michael Middlemas*[1]; Joe Cochran[1]; P. Jain[2]; K. S. Kumar[2]; [1]Georgia Institute of Technology; [2]Brown University

Mo-Si-B alloys are a leading candidate for next generation of jet turbine engine blades and have the potential to increase the service temperature of the base metal 200-300°C higher than nickel-based superalloys. The alloys form a composite microstructure of the molybdenum solid solution ($Mo_{ss}$) and two intermetallic phases, $Mo_3Si$ and $Mo_5SiB_2$, where the $Mo_{ss}$ phase increases ductility and the intermetallic phases enhance strength and provide oxidation resistance. The properties of the alloys are highly dependent on the morphology of the microstructure. A powder processing approach has been developed in which the three-phase alloys are produced through the reaction of molybdenum, $Si_3N_4$ and BN powders. The resulting microstructures are a fine dispersion of the intermetallic phases in a $Mo_{ss}$ matrix. The strengths of the alloys produced by this method have been examined by tensile testing between 1000 and 1300°C and the oxidization resistance was examined using cyclic oxidation tests.

**5:10 PM**

**Cyclic Deformation Behavior of Commercially Pure Mo and a Mo-Si-B Solid Solution Alloy**: *X Yu*[1]; K.S. Kumar[1]; [1]Brown University

Monotonic tensile deformation behavior of Mo and a Mo-Si-B alloy has been previously examined and it was shown that in both these materials, dynamic strain aging(DSA)occurs for specific strain rate-temperature combinations. Therefore,it was thought necessary to understand the cyclic deformation behavior of these materials,particularly in the regime where DSA is prevalent. In this presentation, we will discuss our results on the cyclic deformation response(tension-tension, R=0.1)of commercially pure Mo in the fully recrystallized condition in the temperature range 300K-873K using various levels of maximum stress.The resulting fracture surfaces have been analyzed and will be compared to those obtained from monotonic loading. A similar parallel study is currently underway on a Mo-Si-B solid solution alloy to understand the contribution of the Si in solid solution(since B is thought to have negligible solubility)to the cyclic loading response. Results from these studies will be presented and compared, and the underlying deformation mechanism will be discussed.

**5:35 PM**

**Tensile Creep of Mo-Si-B Alloys**: P. Jain[1]; *Sharvan Kumar*[1]; [1]Brown University

The elevated temperature uniaxial tensile response at a nominal strain rate of $10^{-4}$ $s^{-1}$ and the tensile creep response at constant load between 1000ºC and 1300ºC of a (i) single-phase solid solution (Mo-3.0Si-1.3B in at.%), (ii) two-phase alloy containing ~35 vol.% of the T2 phase (Mo-6Si-8B in at.%), and (iii) three-phase alloy with ~50 vol.% of T2 + $Mo_3Si$ phases (Mo-8.6Si-8.7B in at.%) were evaluated. The results confirm that Si in solid solution significantly enhances both the yield strength and the creep resistance of these materials. A Larson-Miller plot of the creep data showed improved creep resistance of the two-and three-phase alloys in comparison to Ni-based superalloys. A stress exponent of ~5 for the solid solution alloy and ~7 at 1200ºC for the two multiphase alloys suggests dislocation climb to be the controlling mechanism. Grain boundary precipitation of the T2 phase during creep deformation is

observed and the precipitation kinetics appears to be affected by the test temperature and applied stress.

## Solid-State Interfaces: Toward an Atomistic-Scale Understanding of Structure, Properties, and Behavior through Theory and Experiment: Thermodynamics and Morphological Stability

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee

*Program Organizers:* Michael Demkowicz, Massachusetts Institute of Technology; Douglas Medlin, Sandia National Laboratories; Emmanuelle Marquis, University of Oxford

Monday PM Room: 602
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Douglas Medlin, Sandia National Labs; Wayne Kaplan, Technion - Israel Institute of Technology



**2:00 PM Invited**
**Gamma/Gamma Prime Interfacial Free Energies in Nickel-Based Alloys: Atom-Probe Tomographic Experiments and First-Principles Calculations**: *David Seidman*[1]; [1]Northwestern University

We determine gamma(f.c.c.)/gamma-prime(L12) interfacial free energies in Ni-Al-Cr and Ni-Al alloys by studying the coarsening kinetics of gamma-prime precipitates, which permits us to determine the rate constants associated with the temporal evolution of the mean radius and number density of the precipitates, and the matrix supersaturation, employing 3D atom-probe tomography. The interfacial free energies for the Ni-Al-Cr alloys are extracted employing a continuum formalism for ternary alloys, Kuehmann-Voorhees, where all requisite quantities are experimentally measured except for the second derivatives of the free-energy surfaces w.r.t. concentration, which come from thermodynamic data bases. For the Ni-Al alloys we use an approach developed by Ardell that does not require a value of the diffusivity. In parallel with the experiments we employ first-principles calculations (VASP) to calculate interfacial energies, which are compared with the experimental values. It is concluded that the dominant quantity affecting the interfacial free energy is chemical (electronic) in origin.

**2:30 PM**
**Atomic Scale Characterization of Deformation Induced Interfacial Mixing in a Nanostructured Cu/V Composite Wire**: *Xavier Sauvage*[1]; Cécile Genevois[1]; Gérald Da Costa[1]; Victor Pantsyrny[2]; [1]University of Rouen, CNRS; [2]Bochvar Institute of Inorganic Materials

The nanoscaled structure of a Cu/V composite wire was investigated by HR-STEM (HAADF) and Atom Probe Tomography (APT). Some deformation induced interfacial mixing was clearly observed. Mixed layers are typically 2nm wide vanadium gradients in the fcc Cu phase. This mechanical mixing leads to the local fragmentation and dissolution of the filaments where their thickness is less than 5nm. Thus, the material contains locally a significant amount of vanadium super saturated solid solutions in fcc Cu.

**2:50 PM**
**Heterogeneous Nucleation of Nisi2 Epitaxial Growth in Si Nanowires**: *Yi-Chia Chou*[1]; Wen-Wei Wu[2]; Lih J. Chen[3]; K. N. Tu[1]; [1]University of California Los Angeles; [2]National Chiao Tung University; [3]National Tsing Hua University

We report the heterogeneous nucleation of NiSi2 epitaxial growth in Si nanowires observed by in situ high resolution TEM. Heterogeneous nucleation of NiSi2 with Si nanowires was achieved by forming steps in the interface of NiSi2 and Si, which resulted in two silicide/Si interfaces in TEM observation. The lattices between the two interfaces are partially transformed to NiSi2. Moreover, the third interface was observed to move repeatedly from the silicide-rich interface to the Si-rich interface. Line scan EDX analysis indicated the stepwise distribution of Ni and Si concentration profile between the two interfaces. The steps served as kinks for heterogeneous nucleation which required lower supersaturation than homogeneous nucleation. Incubation time of forming one NiSi2 nucleus was measured shorter, and thus the growth rates were faster by heterogeneous nucleation than by homogeneous nucleation.

**3:10 PM**
**Metastability and Competition in Grain Boundary Complexion Transitions**: *Shen Dillon*[1]; Gregory Rohrer[2]; [1]University of Illinois at Urbana-Champaign; [2]Carnegie Mellon University

Grain boundaries may undergo structural and compositional transitions that are analogous to grain boundary 'phase' transitions. These transitions are termed complexion transitions and their behavior significantly affects microstructural evolution and materials properties. The relative energy of boundaries of different complexion is quantified to determine their stability. The results indicate the importance of an activation barrier and metastability. Like other processes subject to phase selection, both the equilibrium thermodynamics and the activation barrier will impact complexion transitions. This suggests that complexion transitions should compete with processes like precipitation for partitioning excess solute. Relative interfacial energies between the host and various precipitates are measured experimentally and correlated with the propensity for complexion transitions. The results indicate a new criterion for additive selection to control grain boundary complexions and microstructural evolution.

**3:30 PM**
**Influence of Grain Boundary Chemistries in Mix-Mobility Thin Film Growth**: *Bianzhu Fu*[1]; Gregory Thompson[1]; [1]University of Alabama

Thin films exhibit compressive-tensile-compressive stress states during the nucleation of islands, coalescence of islands and post-coalescence stages of growth. Using an in situ wafer curvature measurement technique, the stress evolution in Fe-Pt and Fe-Cu systems has been investigated. The stresses were shown to be compositionally dependent. In general, the tensile or compressive stress for the various binary compositions was associated with whichever element enriched the grain boundaries. Under specific growth conditions, a 'zero-stress' state could be achieved. The as-deposited alloy stress states do not show significant stress recovery upon ceasing the deposition. In contrast, in situ annealing resulted in significant changes in stress recovery which is believed to be the enhanced migration of atoms from the boundaries because of the phase state of the film. TEM, AFM and atom probe tomography have been employed to quantify the boundaries as they relate to the preferential segregation and thin film stress.

**3:50 PM Break**

**4:10 PM Invited**
**Thermodynamics of Solid-Fluid Interfaces with Non-Hydrostatically Stressed Solids**: T. Frolov[1]; *Y. Mishin*[1]; [1]George Mason University

While thermodynamics of solid-fluid interfaces is well understood for hydrostatic systems, the case of non-hydrostatically stressed solids remains less studied. We present thermodynamic relations for the free energy, interfaces stress, segregation and other excess properties of interfaces between fluids and non-hydrostatically stressed solid phases in multi-component systems. These relations lead to various thermodynamic integration schemes and are presented in forms convenient for atomistic simulations. These relations are illustrated by several examples of calculation of interface thermodynamic properties in solid-liquid coexistence systems using Monte Carlo and molecular dynamics methods with embedded-atom potentials.

**4:40 PM**
**Grain Boundary Misorientation Instabilities**: *W. Craig Carter*[1]; [1]MIT

Grain boundary energies are, among other things, a function of misorientation. Such misorientation-energy functions typically have cusps at particular misorientations. It is demonstrated that such functions, including some reported in the literature, possess instabilities with respect to the creation of an additional misorientation.

**5:00 PM**
**On the Thermodynamic Stabilization of Defects in the Framework of a Defactant Concept**: *Reiner Kirchheim*[1]; [1]University of Göttingen

Gibbs treatment of surfactants is extended to include other crystalline defects besides interfaces, because there is an analogy between surfactants in liquids stabilizing structures with large surface areas like foams or microemulsions, and components in a crystalline solid stabilizing grain boundaries, dislocations and vacancies. These components are called defactants (defect acting agents). Thus

solutes may reduce the defect formation energies given by Gibbs as $d\gamma=-\Gamma d\mu$, where $\mu$ is the chemical potential of solute, $\Gamma$ is the excess solute and $\gamma$ is now the defect energy. In this context conditions and consequences are discussed where defect energies become zero or negative leading to metastable equilibrium or instable crystalline phases. First principle calculations of (i) the surface energy of small alumina particles with excess hydrogen, (ii) the grain boundary energy of copper with bismuth excess and (iii) the vacancy formation energy in iron with excess hydrogen all yield negative defect formation energies.

**5:20 PM**

**Interfacial Structure and Morphological Evolution of Platinum Nano-Precipitates Embedded in Sapphire**: *Melissa Santala*[1]; Velimir Radmilovic[2]; Raquel Guilian[3]; Mark Ridgway[3]; Andreas Glaeser[1]; Ronald Gronsky[1]; [1]University of California; [2]National Center for Electron Microscopy; [3]Australian National University

Platinum nano-precipitates (<100nm diameter) were formed in sapphire by high-energy ion implantation followed by thermal annealing. The morphology of the precipitates depends on the orientation relationship of the precipitates within the sapphire and can be either partially or almost fully faceted. Atomically flat faceted interfaces coinciding with low index planes of sapphire were observed with high-resolution transmission electron microscopy (HRTEM). Prominent planar interfaces form for interfacial relationships with $(111)_{Pt}$ planes parallel to low-index planes of sapphire. Some specimens were annealed at 1600°C for 100h, a time and temperature expected to result in full morphological equilibration for precipitates <100nm in diameter, if the evolution were purely diffusion-limited. For precipitates with an orientation relationship $(0001)_{sapphire} \| (111)_{Pt}$;$[10\text{-}10]_{sapphire} \| [1\text{-}10]_{Pt}$ the presence of atomically flat interfaces parallel to the (0001) basal plane of sapphire frustrates the expected morphological equilibration.

## Surface Engineering for Amorphous-, Nanocrystalline-, and Bio-Materials: Session II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee

*Program Organizers:* Sandip Harimkar, Oklahoma State University; Arvind Agarwal, Florida International Univesity; Sudipta Seal, University of Central Florida; Narendra Dahotre, University of Tennessee

Monday PM Room: 604
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Arvind Agarwal, Florida International University; Roger Narayan, North Carolina State University; Narendra Dahotre, University of Tennessee

**2:00 PM Introductory Comments**

**2:05 PM Invited**

**Assessment of Microbial Biofilm Growth on Nanocrystalline Diamond Coatings Using a CDC Biofilm Reactor**: J. S. Lewis[1]; S. D. Gittard[1]; *Roger Narayan*[1]; C. J. Berry[2]; Robin Brigmon[2]; R. Ramamurti[3]; R. N. Singh[3]; [1]University of North Carolina & North Carolina State University; [2]Savannah River National Laboratory; [3]University of Cincinnati

Nanostructured materials may play a major role in next generation medical devices. In this work, atomic layer deposition was used to deposit titanium oxide coatings on all the surfaces of nanoporous alumina membranes in order to reduce pore size in a controlled manner. The titanium oxide coating serves several purposes, including: (1) maintaining a narrow pore size distribution, (2) reducing pore size in a controlled manner, (3) preventing aluminum ions from leaching into tissues, and (4) creating a biocompatible membrane/tissue interface. Staphylococcus aureus and Escherichia coli were used to evaluate the antimicrobial properties of TiO2-coated nanoporous alumina membranes. Nanostructured materials prepared using atomic layer deposition may be used in "smart" drug delivery devices or self-sterilizing medical devices.

**2:30 PM**

**Wetting Behavior of Laser Synthetic Surface Micro Textures on Ti-6Al-4V for Bioapplication**: *Sameer Paital*[1]; Wei He[1]; Claus Daniel[2]; Narendra Dahotre[1]; [1]The University of Tennessee; [2]Oak Ridge National Laboratory

Wettability at the surface of an implant material play a key role in its success as it modulate the protein adsorption and thereby influences cell attachment and tissue integration at the interface. Hence, surface engineering of implantable materials to enhance wettability to physiological fluid under in vivo conditions is an area of active research. In light of this, in the present work a laser based optical interference and direct melting techniques were used to develop synthetic micro textures on Ti-6Al-4V alloys and their effects on wettability were systematically studied. Improved wettability to simulated body fluid and distilled water was observed for the Ca-P coatings obtained by direct melting technique. The effect of physical texture and wetting on biocompatibility of laser processed Ca-P coating was evaluated in the preliminary efforts on culturing of mouse MC3T3-E1 osteoblast cells.

**2:50 PM**

**Corrosion of the Implant Alloy Ti-45wt.%Nb in Simulated Physical Media**: *Daniela Zander*[1]; [1]TU Dortmund

Titanium and titanium alloys, especially Ti-6Al-4V, are used for various medical applications, e.g. endoprosthesis. There is evidence that metallic dissolution into surrounding tissue can cause complications with the implant and the environment. This research focuses on investigating the electrochemical surface properties of Ti-45wt.%Nb in simulated physiological media. In order to verify the influence of the radical reaction by free oxygen and the interaction with hyaluronic acid by potentiodynamic polarization and electrochemical impedance spectroscopy were conducted in PBS solution without and with H2O2 and hyaluronic acid in comparison to titanium. The surface topography and passive layer was studied by X-ray diffraction, SEM and TEM.DC and AC measurements showed a strong influence of OH°-radicals and hyaluronic acid on the passive current and the passivation mechanisms. The results obtained by electrochemical measurements are correlated with microstructural investigations, e.g. the formation of a porous passive layer due to the influence of H2O2.

**3:10 PM**

**Corrosion Behaviour Evaluation of Fluoridated Hydroxyapatite/Niobium Filler-Matrix Composite Coating for Hard Tissue Implant**: *Ehsan Mohammadi Zahrani*[1]; M. H. Fathi[2]; Akram Alfantazi[1]; [1]The University of British Columbia; [2]Isfahan University of Technology

Need to reduce cost in public health services has compelled use of 316L stainless steel as the most economical alternative for hard tissue implants because of its relatively low cost and reasonable corrosion resistance. However, this material is prone to localized attack in long-term application due to the aggressive biological effects. In this study fluoridated hydroxyapatite/niobium (FHA/Nb) coatings with 0 and 100% degree of fluoridation was deposited on 316L SS substrate by using nanostructure FHA powders, produced by mechanical alloying technique, to improve the corrosion properties and biocompatibility of implant. XRD and SEM techniques were utilized to investigate the effect of fluoridation on morphology of the coatings. Electrochemical polarization tests were carried out in ringer and normal solution in order to determine the corrosion behaviour of coated specimen as an indication of biocompatibility. The results confirmed that fluoridation could improve the corrosion resistant and phase stability of the plasma-sprayed coating.

**3:30 PM**

**Surface Engineering of Titanium Alloy Modified by Plasma-Based Low-Energy Ion Implantation**: *M.K. Lei*[1]; Z.L. Wu[1]; Y.X. Ou[1]; T.K. Song[1]; Q. Zhou[1]; [1]Dalian University of Technology

Plasma-based low-energy ion implantation, including plasma source ion nitriding/carburizing and plasma source low-energy ion enhanced deposition, has emerged as a low-temperature surface engineering technique for metal and alloy. In this work, the biomedical titanium alloy samples were modified by plasma source ion nitriding at a low temperature ranging from 500ºC to 650ºC, and sequently by physical vapor deposition of the titanium nitride films. The TiN films deposited on the plasma source ion nitrided titanium alloy surface which consisted of TiN and Ti2N possessed an improvement in wear and corrosion properties. The tribological behavior of the modified titanium alloy samples were investigated on a ball-on-disc tribometer against AISI 52100

bearing steel. The electrochemical polarization measurement was measured in 1% NaCl solution using a standard three electrodes system. It can be found that plasma-based low-energy ion implantation of titanium alloy provides a potential application in biomedical devices.

**3:50 PM Break**

**4:05 PM**
**Surface Nitriding of Ti-6Al-4V for Bio-Implant Application**: *Jyotsna Dutta Majumdar*[1]; [1]Indian Institute of Technology Kharagpur

Ti-6Al-4V alloy is widely used as an implant material because of its high strength, superior corrosion resistance and bio-compatibility. However, a poor wear resistance causes restriction over prolonged use of the component as bio-implant. In the present study titanium nitride layer has been formed on Ti-6Al-4V substrate by cathodic arc evaporation and laser gas alloying process. The effect of surface nitriding on the characteristics and properties (microhardness, wear, corrosion resistance and bio-compatibility) of the surface has been studied in details. Titanium nitrided layer formed by physical vapor deposition is homogenous and defect-free with an improved hardness to 650 VHN as compared to 260 VHN of as-received Ti-6Al-4V. Wear resistance of the physical vapor deposited surface is also improved significantly. Corrosion resistance against simulated body fluid is marginally improved in titanium nitride coated surface as compared to as-polished Ti-6Al-4V substrate.

**4:25 PM**
**Nanostructured Bio-Scaffold for Bone Implants, Stents: A Biomedical Evolution**: *Shampa Aich*[1]; Chandra Shekhar[1]; Mrinal Mishra[1]; [1]Indian Institute of Technology

Multifunctional nanostructured bio-scaffolds were fabricated on biocompatible titanium and Nitinol surfaces through a simple, inexpensive, easy scale-up, one-step hydrothermal reaction of alkali (NaOH) with the metal and alloy surfaces without using any seeds, templates, $TiO_2$ powder, or stabilizers. The structure and morphologies of the bio-scaffold were investigated using electron microscopy and glancing angle X-ray diffraction techniques. The nano-scaffold seems to root firmly inside the Ti and NiTi substrates and grow on top to eventually self assemble into macroporous (~ 0.5-1.0 µm in diameter) scaffolds. The effects of alkali concentration, reaction time on the bio-scaffold morphologies were investigated. The maximum bio-scaffold thickness of 987 nm was observed at NaOH conc. 2.85 M for 3 hours treatment time. Thus-formed scaffolds on the metal implant or stent surfaces, mimicking the natural extracellular matrix in structure, can promote good cellular compatibility, mechanical toughness and proliferation and perform controlled on-site drug release and photocatalytic sterilization.

**4:45 PM**
**The Effect of Processing Parameters on Surface Properties of Ti6Al7Nb Alloys**: *Mert Günyüz*[1]; Murat Baydogan[1]; Huseyin Cimenoglu[1]; Eyup Sabri Kayali[1]; [1]Istanbul Technical University

In this study, the effect of micro arc oxidation process on surface morphology of Ti6Al7Nb alloy was investigated. Micro arc oxidation was performed by using an AC power supply operating in constant voltage mode between 450-600 V using two different electrolytes. Mechanical and physical properties were evaluated on the surface including hardness, contact angle and surface roughness of the oxide film. Rockwell C testing was used to compare the relative adhesion characteristics of the oxide film. The surface oxide layer was examined by an X-ray diffractometer. Mechanical and physical properties of the samples were discussed on the basis of micro arc oxidation parameters such as applied voltage and electrolyte composition.

**5:05 PM**
**Electrodeposition of Hydroxyapatite on Magnesium for Biodegradable Implant Applications**: *Satendra Kumar*[1]; M. Jamseh[1]; Sankara Narayanan TSN[1]; [1]National Metallurgical Laboratory, Madras Centre

Development of biodegradable Mg based implants and stent materials assumed significance in recent years. Higher corrosion rate, generation of large volume of hydrogen gas and, increase in local pH value of body fluid, are the major limiting factors in using Mg as a biodegradable implant material. The present work aims to engineer the surface of Mg by electrodeposition of hydroxyapatite to control the rate of corrosion and to solve the associated problems. The corrosion behaviour of hydroxyapatite coated Mg in Ringer's solution was evaluated by electrochemical impedance spectroscopy and its corrosion protective ability was compared with that of the untreated one. The study reveals that deposition of hydroxyapatite coating on Mg decreased the rate of corrosion. Being a bioactive coating, it will also enable bone in-growth. The study concludes that electrodeposition of hydroxyapatite coating is a useful surface engineering approach in the development of biodegradable Mg based biomaterials.

**5:25 PM**
**Fabrication and Characterization of $Tio_2$ Films on Ti-6Al-4V by Anodic Oxidation**: Maria Vera[1]; Alicia Ares[1]; Mario Rosenberger[1]; Diego Lamas[2]; *Carlos Schvezov*[1]; [1]CONICET-UNaM; [2]CONICET-CITEDEF

In contact with air, a protective titanium dioxide ($TiO_2$) film normally covers the surface of titanium and its alloys, which is 2 to 7 nm in thick. Artificial $TiO_2$ films can be produced on Ti-6Al-4V substrates by anodic oxidation. In this study, $TiO_2$ films were produced on Ti-6Al-4V substrates by anodic oxidation technique, using a $H_2SO_4$ solution as electrolyte at different voltages (from 10V to 100V). The morphology, thickness and phase composition of the films were determined by scanning electron microscopy, X-ray reflectometry and X-ray diffraction, respectively. $TiO_2$ films of different colors were obtained, with thickness ranging from 20 to 200 nm depending on the applied voltage. Relationships among voltage, color and thickness were confirmed. The film roughness is of the order of the substrate roughness. At voltages higher than 70V, two crystalline phases of $TiO_2$ were obtained, anatase and rutile. For lower voltages the coating is amorphous.

**5:45 PM**
**Effect of Laser Surface Treatment on Tribological Behavior and Bioactivity of ASTM F-75 Cobalt Base Alloy**: *J.L. Davila*[1]; F. Cepeda Rodriguez[1]; M.F. Trejo Aguirre[1]; [1]Corporación Mexicana De Investigación En Materiales

A biomedical ASTM F-75 cobalt base alloy specimen was subjected to laser surface treatment using pulsed Nd-YAG laser equipment. Laser surface treated and untreated discs specimens were tested on Pin-on-disk tribometer to evaluate the wear resistance following the ASTM G99 standard. The laser surface treatment was beneficial, the treated specimen present considerably minor wear than the untreated one, and also increased surface microhardness of 325 to 445 Vickers at remelted zone due to grain refinement and solid solution strengthening. For the Bioactivity evaluation, the laser surface treated and untreated samples were immersed in simulated body fluid (SBF) for 15 days, later the metallic surface were analyzed by Optical Microscopy (OM), Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray analysis (EDX) and Infrared Spectroscopy (IR). The results show presence of P-O stretching band, that indicating formation of apatite.

## Sustainable Materials Processing and Production: Motivating Sustainability I

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee, TMS: Education Committee
*Program Organizers:* Christina Meskers, Umicore; Randolph Kirchain, Massachusetts Institute of Technology; Diana A. Lados, Worcester Polytechnic Institute; Markus Reuter, Ausmelt Limited

Monday PM Room: 2B
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Markus Reuter, Ausmelt Ltd.; Iver Anderson, Ames Laboratory

**2:00 PM Introductory Comments by Iver Anderson**

**2:10 PM Plenary**
**A Framework and a New Paradigm for Sustainable Materials Development and Engineering: The Status Quo is not Sustainable!**: *Diran Apelian*[1]; [1]Worcester Polytechnic Institute

Though there is much discussion on the subject of sustainable development for the 21st Century, the Materials Science and Engineering (MSE) community has not responded nor provided a roadmap as to how we respond to the global issues regarding material resource scarcities. Inorganic materials are non-renewable; one would expect that appropriate design, life-cycle analysis, judicious material

selection as well as recovery and recycling be the keystones of a new paradigm. The status quo is not sustainable. A framework is presented and discussed in an effort to have material development and material engineering be sustainable in the 21st Century.

**2:35 PM Plenary**
**The Sustainable Organization: Fostering the Capacity for Change**: *Leith Sharp*[1]; [1]Harvard University

For the last 18 years Leith Sharp has worked with a wide variety of universities around the world. Most recently Leith founded and directed Harvard's Green Campus Initiative (now the Office for Sustainability) for nine years, successfully placing Harvard at the forefront of the green campus movement. In this presentation Leith will provide a detailed overview of the organizational requirements for achieving deep, lasting and ongoing transformation towards sustainability. Using the Harvard University case study, Leith will provide a variety of insights, strategies and examples touching upon aspects of leadership, finance and accounting, human resources, governance, communication and more. The purpose of the presentation is to assist the audience in leading their own organizations to take the greatest possible strides towards sustainability.

**3:00 PM Plenary**
**Building a Sustainability Strategy into a Consumer Products Business**: *Leon H. Bruner*[1]; Peter White[1]; [1]The Procter & Gamble Company

This paper presents a case study describing how a global consumer products company with 138,000 employees that markets over 300 brands in 180 countries is building sustainability into the rhythm of its business. This strategy extends beyond social investment programs by incorporating sustainability into the fabric of product design, manufacturing operations, employee engagement and stakeholder partnerships. During the implementation of this program it has been necessary to be explicit about sustainability and its importance to the business, to broadly define sustainability, to ensure that sustainability is not added work, to eliminate trade-offs between performance, value and sustainability and to implement programs to ensure that sustainability is incorporated into the DNA of the company. Our program continually emphasizes the importance of making no tradeoffs between consumer acceptance of sustainable products and sustainability investments. We also work diligently to overcome the challenges of engaging employees around the world to implement a single coherent sustainability strategy that delivers great results.

**3:25 PM Plenary**
**Practicing Informed Substitution of Restricted Materials in Electronic Products: Knowing Whether Replacements are Better for the Environment and Human Health**: *Helen Holder*[1]; [1]Hewlett-Packard Company

When a substance is targeted for restriction, often all unregulated substances are treated as equally suitable replacements. This practice has several disadvantages, most significantly that alternatives may be equally bad or worse than the substances they replace. A much better approach to material selection is to practice informed substitution when transitioning out of chemicals of concern such that the environmental and human health impacts of alternatives are considered in order to ensure that the new materials represent a move to safer chemicals or nonchemical alternatives. In order to practice informed substitution, a framework for assessing competing options is needed. A promising tool to support informed substitution is a hazard-based assessment method called the Green Screen, which was created by Clean Production Action, an environmental non-profit dedicated to advancing green chemistry, sustainable materials and environmentally preferable products. This talk will discuss the Green Screen as a tool for alternatives assessment as a part of informed substitution, and how this tool is being used to inform material selection decisions for replacing restricted substances in electronic products.

**3:50 PM Break**

**4:05 PM Plenary**
**Sustainability, a Strategic Opportunity for Umicore:** *Mark Caffarey*[1]; [1]Umicore, USA

How does one turn a historically burdened industrial company into a flagship of sustainability? How does one, literally and figuratively, come clean with a less than sustainable past? With a clear vision and keeping its sights on the future, Umicore has cleared the decks of all its past inheritance. New policies have been laid down, based on eco-efficiency and social responsibility. The outcome is a company that is re-energized believing in a future that is just as sustainable as profitable. Ten years ago, Umicore made fundamental choices by concentrating on two activities, i.e. the development of high quality materials with extensive technology content and the recycling of precious and rare metals, which form the basis of many of these high-tech materials. As the materials provider for rechargeable batteries, catalytic converters, solar cells and other high tech applications and as the recycler of end-of-life materials from these same technologies, Umicore has been able to illustrate and effectively close the material loop. Today, over half of Umicore turnover and profits is derived from this wide range of Clean Technologies. This illustrates the success sustainability as a strategy is having for Umicore.

**4:30 PM Plenary**
**Title Not Available: John Allison**[1]; [1]**Ford**
**Abstract not available.**

**4:55 PM Plenary**
**Energy (to be confirmed)**

**5:20 PM Concluding Comments by Markus Reuter**

## The Vasek Vitek Honorary Symposium on Crystal Defects, Computational Materials Science and Applications: Computational Materials Science II

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
*Program Organizers:* Mo Li, Georgia Institute of Tech; David Srolovitz, Institute for High Performance Computing, Agency for Science, Technology and Research, Singapore; Adrian Sutton, Imperial College London; Vaclav Paidar, Institute of Physics AS CR vvi; Jeff De Hosson, Univ of Groningen

Monday PM Room: 603
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* David Srolovitz, Institute for High Performance Computing, Agency for Science, Technology and Research, Singapore; David Pettifor, University of Oxford

**2:00 PM Invited**
**Atomic Level Stresses**: *Takeshi Egami*[1]; [1]University of Tennessee

When V. Vitek joined Penn in 1978 I was starting to think that the stress could be defined at the atomic level, and would be quite helpful in characterizing the local structure of metallic glasses. The trouble was I had no idea how to calculate it. But Vasek knew exactly how to do it, and we right away entered one of my most productive and enjoyable collaborations. David Srolovitz and Adrian Sutton just joining Penn as students also helped the development. The origin of the atomic level stresses is geometrical and topological fluctuation in the atomic environment. Since it has the dimension of energy (per volume), it can readily be related to the local energy landscape, and to various local properties. I will discuss how this concept helped advanced the understanding of glass and glass transition, and helped unlock one of the few remaining deep mysteries in solid state theories.

**2:25 PM Invited**
**Molybdenum at High Pressure and Temperature: Melting from Another Solid Phase**: *Shao-Ping Chen*[1]; Leonid Burakovsky[1]; A. B. Belonoshko[2]; [1]Los Alamos National Laboratory; [2]The Royal Institute of Technology

The Gibbs free energies of bcc and fcc Mo are calculated from first principles in the quasiharmonic approximation in the pressure range from 350 to 850 GPa at room temperatures up to 7500 K. It is found that Mo, stable in the bcc phase at low temperatures, has lower free energy in the fcc structure than in the bcc phase at elevated temperatures. Our density-functional-theory-based molecular dynamics simulations demonstrate that fcc melts at higher than bcc temperatures above 1.5 Mbar. Our calculated melting temperatures and bcc-fcc boundary are consistent with the Mo Hugoniot sound speed measurements. We find that melting occurs at temperatures significantly above the bcc-fcc boundary. This suggests an explanation of the recent diamond anvil cell experiments, which find a phase boundary in the vicinity of our extrapolated bcc-fcc boundary.

Other possible phases will also be discussed.Phys. Rev. Lett., 100, 135701 (2008); 101, 049602 (2008).

**2:50 PM Invited**

**Analytic Bond-Order Potentials Including Magnetism**: *Ralf Drautz*[1]; David Pettifor[2]; [1]Ruhr-Universität Bochum; [2]University of Oxford

Simulations of mechanical properties of materials require interatomic potentials that can predict interatomic interactions in stacking faults or dislocation cores, for example. In iron and ferritic steels the magnetic contribution to the binding energy depends strongly on the local atomic environment. We have developed an analytic interatomic bond-order potential (BOP) for non-magnetic transition metals that depends explicitly on the valence of the transition metal element and describes the structural trend from hcp to bcc to hcp to fcc across the transition metal series. This potential may be extended to include magnetic contributions within the 3d series. We show that the resulting magnetic interatomic potential displays the experimental trend from anti-ferromagnetic to ferromagnetic order across the 3d transition metal series. For iron, the potential correctly predicts a large magnetic energy for the alpha phase whereas the close-packed gamma and epsilon phases exhibit only a small magnetic contribution to the binding energy.

**3:15 PM Invited**

**Irradiated Point Defects in bcc Transition Metals and Alloys: A Multi-Scale Modeling Review**: *Duc Nguyen-Manh*[1]; [1]UKAEA

Body centered cubic (bcc) transition metals, especially tungsten alloys and ferritic-martensitic steels, are the leading candidate materials for fusion-power-plant applications. A quantitative understanding of point defects under neutron irradiations is essential for modeling the micro-structural evolution of these materials. Our recent systematic and multi-scale studies based on density functional theory (DFT) calculations of self-interstitial atom (SIA) defects, spanning the entire group of bcc metals, reveal that the SIAs adopt a linear <111> crowdion configuration in all non-magnetic bcc transition metals whereas in ferromagnetic bcc-Fe the most stable SIA configuration has the <110> orientation. The predicted SIA formation energy in bcc-W has been recently confirmed experimentally. Combining DFT calculations with the analytic expression for the Peierls potentials, a very small migration energy barrier has been obtained for crowdion in agreement with resistivity recovery measurements. The interactions between crowdion with impurities and other defects in bcc alloys are being investigated.

**3:40 PM Break**

**4:05 PM Invited**

**Deformations in Nanosized Metallic Glass Systems**: *Jeff De Hosson*[1]; C. Chen[1]; Y. Pei[1]; Vasek Ocelik[1]; Dave Matthews[1]; [1]University of Groningen

Size effect, or the lack thereof, during deformation of metallic glasses (MGs) has recently drawn great attention. After the computer modeling approach by Vasek Vitek and collaborators significant progress has been made in the understanding of deformation of amorphous metals. An intriguing question is why and how nucleation and propagation of shear bands are affected by the size of the system, and would it be possible to suppress brittleness and enhance ductility by changing the size of the samples? Our quantitative in-situ TEM deformations of metallic glass pillars with diameters ranging from 50 nm to 500 nm reveal that the deformation is controlled by nucleation of shear bands in larger pillars but becomes propagation controlled in smaller pillars. A micromechanical model based on quantitative description of shear banding events explains the size-dependent deformation behavior. Implications of our findings for applications in nanosized systems will be illustrated.

**4:30 PM Invited**

**Recent Advances and Ongoing Challenges in Accelerated Molecular Dynamics Methods**: *Arthur Voter*[1]; [1]Los Alamos National Laboratory

Many important materials processes take place on time scales that vastly exceed the nanoseconds accessible to molecular dynamics simulation. Typically, this long-time dynamical evolution is characterized by a succession of thermally activated infrequent events involving defects in the material. Over the last decade, we have been developing a new class of methods, accelerated molecular dynamics, in which the known characteristics of infrequent-event systems are exploited to make reactive events take place more frequently, in a dynamically correct way. For certain processes, this approach has been remarkably successful, offering a view of complex dynamical evolution on time scales of microseconds, milliseconds, and sometimes beyond. Examples include metallic surface diffusion and growth, radiation damage annealing in ceramics, and carbon nanotube dynamics. After an introduction to these methods, I will present some recent advances and results, and then describe the major ongoing challenges and our current thinking on how to overcome them.

**4:55 PM Invited**

**Bond-Order Potential for Iron**: *Matous Mrovec*[1]; Duc Nguyen-Manh[2]; Christian Elsaesser[1]; Peter Gumbsch[1]; David Pettifor[3]; [1]Fraunhofer Institute for Mechanics of Materials; [2]EURATOM/UKAEA Fusion Association; [3]Oxford University

Development of reliable interatomic potentials for atomistic simulations of lattice defects in iron and iron-based materials presents a significant challenge. Energetics and structural stability of magnetic materials is strongly influenced by magnetic effects and details of the Fermi surface. These materials therefore cannot be described adequately by common many-body central-force potentials, which are only density dependent. In this work we constructed a bond-order potential (BOP) for iron, which is based on a tight-binding bond representation. This model is not only able to capture the directional character of bonds present in transition metals but includes also a description of magnetic effects within the Stoner model of itinerant magnetism. A high reliability and predictive power of the constructed BOP will be demonstrated in studies of point defects, dislocations, and grain boundaries in iron.

**5:20 PM**

**Core Structure and Kinks of Screw Dislocations in Fe and W from First-Principles**: *Lisa Ventelon*[1]; François Willaime[1]; Emmanuel Clouet[1]; Mihai-Cosmin Marinica[1]; [1]CEA

Dislocation properties in bcc metals are closely related to the dislocation core structure, which itself is quite sensitive to details in the interatomic interactions. We have investigated the properties of [111] screw dislocations in iron and tungsten, using ab initio electronic structure techniques, primarily in the periodic dipole approach. In agreement with previous calculations, we find a compact structure of the core, but we also evidence a significant core dilatation effect, which can be successfully accounted for by an anisotropic elasticity model. The Peierls potential is single humped i.e. there is no metastable configuration at halfway position, at variance with predictions from EAM potentials. The methodology for constructing tri-periodic cells with one kink per dislocation line has been developed. Results obtained with empirical potentials and preliminary DFT calculations are presented for the Peierls stress and kinked dislocations.

**5:35 PM**

**Core Traction Contribution to the Elastic Energy of a Dislocation**: *Emmanuel Clouet*[1]; [1]CEA Saclay

The elastic energy of a straight dislocation can be decomposed into two contributions: one corresponding to an integration along the dislocation cut of the work necessary to create the dislocation; the other arising from the work done by the tractions exerted on the tube which isolates the dislocation core. This last contribution is often forgotten although it is necessary for the elastic energy being a state variable consistent with the work of the Peach–Koehler forces. We derive an expression of this contribution within linear anisotropic elasticity theory [1]. The obtained expression is then used to extract dislocation core energies from atomistic simulations. This is illustrated by calculating core energies of edge dislocation in bcc iron, where we show that dislocations gliding in {110} planes are more stable than those gliding in {112} planes.[1] E. Clouet, Philos. Mag. 89, p. 1565 (2009).

**5:50 PM**

**Ab Initio Study of Extreme Loading Conditions in Transition-Metal Disilicides with the C40 Structure**: *Martin Friak*[1]; Dominik Legut[2]; Mojmir Sob[3]; [1]Max Planck Institute for Iron Research; [2]Institute of Physics of Materials, Academy of Sciences of the Czech Republic; [3]Faculty of Science, Masaryk University

The ideal tensile test in transition metal disilicides NbSi2, TaSi2, CrSi2, and VSi2 with the hexagonal C40 structure is simulated by ab initio electronic structure calculations. The theoretical tensile strength for the [0001] loading direction is determined and compared with that of (i) MoSi2 and WSi2 disilicides crystallizing in the C11b tetragonal structure and (ii) NbSi2, TaSi2, CrSi2, and VSi2 with C11b structure. A full relaxation of both external and internal structural parameters is performed and their response to the tensile loading is

analyzed. The changes in the electronic structure induced by loading conditions far from equilibrium are demonstrated on the comparison of the densities of states in the ground state and at the limit of mechanical stability.

**6:05 PM**

**Modeling the Synergetic Interactions between Creep and the Growth of Thermal Scales**: *David Wilkinson*[1]; Somaradi Khiev[1]; Andi Limarga[2]; [1]McMaster University; [2]Harvard University

The growth rate of oxide and nitride scales can be considerably affected by the application of stress when the underlying substrate creeps. We have developed a model to calculate the residual stress induced in growing scales which demonstrates that the applied stress causes the metallic substrate and overlying scale to creep at different rates leading to the accumulation of a large stress in the thin scale layer. This influences the diffusional flux of anions towards the metal/nitride interface, controlling the scale growth kinetics. The model is applied to the growth of TiN scales on γ-TiAl in a pure N2 atmosphere as well as to oxidation in an Fe-based intermetallic. The scale growth rate is dependent on the sign and magnitude of the applied stress wherein a tensile stress accelerates the growth process and vice versa. The model predicts experimental results in TiN bars following flexural creep.

## Three-Dimensional Materials Science VI: Processing and Analysis of Large 3D Datasets

*Sponsored by:* The Minerals, Metals and Materials Society, ASM International, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, ASM-MSCTS: Texture and Anisotropy Committee, TMS/ASM: Phase Transformations Committee

*Program Organizers:* Alexis Lewis, Naval Research Laboratory; Anthony Rollett, Carnegie Mellon University; David Rowenhorst, Naval Research Lab; Jeff Simmons, AFRL; Stuart Wright, EDAX Inc-TSL

Monday PM Room: 401
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Jeff Simmons, U S Air Force Research Laboratory; Marc De Graef, Carnegie Mellon University

**2:00 PM Invited**

**Archival Storage of Three-Dimensional Data**: Mike Jackson[1]; Jeff Simmons[2]; *Marc De Graef*[3]; [1]Bluequartz Software; [2]Air Force Research Laboratory; [3]Carnegie Mellon University

The ever-expanding capability of 3-D characterization tools has lead to the development of high quality multimodal data sets. The vast majority of these are currently managed in an ad-hoc fashion on individual computers, a practice which runs a significant risk of data loss through random events; long-term storage is hence a non-trivial problem. We have developed a new file format suitable for the archival storage of 3D data. The Multidimensional eXtensible Archive (MXA) format is built on the robust open source Hierarchical Data Format (HDF-5), and provides a means of storing multidimensional data along with meta-data in a single file. The format is flexible and relies on Document Type Definition and eXtensible Markup Language files. A C++ library along with documentation is provided at http://mxa.web.cmu.edu. We will illustrate both the file format and its use by means of 3D microstructure examples.

**2:30 PM**

**Data Fusion by Means of Mutual Information and Image Entropy**: *Begum Gulsoy*[1]; Jeff Simmons[2]; Marc De Graef[1]; [1]Carnegie Mellon Unversity; [2]Air Force Research Laboratory

For years, the materials community has been developing experimental techniques that allow for the characterization of different aspects of materials, resulting in multi-modal data sets. When information from different detectors, such as scanning electron microscopy images and orientation imaging maps, needs to be fused together to obtain a more complete microstructural understanding, the data fusion process is not always straightforward. Mutual information, a concept borrowed from the field of information theory, is an entropy-based measure of how similar two pieces of information are. It is an easily implemented approach that can be used for general affine multi-modal image registration problems, and proves to be especially useful for cases where the traditional cross-correlation approach does not work. We will provide an introduction to this concept and show applications of image registration in nickel-based superalloys as well as two-phase titanium alloys.

**2:50 PM**

**Using Moment Invariants to Assess the Realism of Digitally Constructed Microstructures**: Patrick Callahan[1]; Mike Groeber[2]; *Marc De Graef*[1]; [1]Carnegie Mellon University; [2]AFRL/UTC

Digitally constructed microstructures can be generated from a combination of microstructural descriptors, such as grain size, aspect ratio, and number of neighbors, which are determined from experimental microstructures. Usually, a synthetic microstructure is compared qualitatively to the experimental microstructure. It is important to remove any user bias inherent in qualitative comparisons and obtain objective comparisons by using quantitative descriptors. Synthetic microstructures can be compared quantitatively to experimental microstructures using coordinate independent quantities such as moment invariants. We will define moment invariants and then use them in combination with other shape parameters, such as volume and surface area, to compare quantitatively experimental microstructures with synthetic microstructures. The synthetic reconstructions are generated starting with ellipsoids and superellipsoids as the initial grain shapes. Our work shows that moment invariants provide a tool for assessing the accuracy and realism of digitally constructed microstructures, and can validate or compare reconstructions produced using different models.

**3:10 PM Break**

**3:30 PM Invited**

**Acquisition and Analysis of Gigabyte-Scale Tomographic Spectral Images**: *Paul Kotula*[1]; Lysle Serna[1]; [1]Sandia National Laboratories

Spectral imaging where a complete spectrum is acquired from each of an array of points has been extended to 3D volume analysis via serial sectioning with FIB [1] and metallography. With the advent of automated data acquisition software for FIB in particular, the scale of data sets that can be acquired easily exceeds a gigabyte. This talk will describe both data acquisition strategies and robust multivariate statistical analysis methods of tomographic spectral images. The result is an unbiased and compact representation of the data in terms of correlated elemental contrast. Examples from corrosion, brazing, and thin-film analysis will be presented. [1] P.G. Kotula et al., Microsc. Microanal. 12, pp.36-48, 2006.Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

**4:00 PM**

**Application of Novel Techniques to the Three-Dimensional Characterization of Microstructural Features in Alpha+Beta Titanium Alloys**: *John Sosa*[1]; Santhosh Koduri[1]; Vikas Dixit[1]; Peter Collins[1]; Hamish Fraser[1]; [1]The Ohio State University

Advanced three-dimensional data collection techniques such as Robo.Met-3D™ has led to rapid acquisition of robust datasets on optical length scales. Implementation of such datasets may improve the accuracy of neural networks and phase-field models. However, the accurate statistical representation of three-dimensional microstructural features is challenging and thus requires further improvement to analytical methods. This work addresses the serial two-dimensional collection of datasets containing microstructural features such as prior-beta grain size, and equiaxed-alpha particle size/distribution in alpha+beta titanium alloys. Subsequent reconstruction and quantitative analysis has been facilitated by the development of a MATLAB-based toolkit. Furthermore, comparisons have been made between two-dimensional stereological measurements and their three-dimensional extensions. Additionally, crystallographic orientation data has been incorporated into Robo.Met-3D™ datasets via EBSD maps collected at regular intervals.

**4:20 PM**

**Prior Information for Segmentation of Large Serial Section Image Datasets**: *Jeff Simmons*[1]; Mary Comer[2]; Ilya Pollak[2]; Marc De Graef[3]; [1]AFRL; [2]Purdue University; [3]Carnegie Mellon University

Currently, segmented images are used to build inputs for property simulations. With image segmentation, pixels are classified according to two factors: (1) signal strength (the image) and (2) interpretation within a materials context.

Classical image processing extensively utilizes algorithms that process the observed signal, but are generally blind to knowledge of material behavior. Incorporation of "prior information," i.e. information that is known prior to the microscope observation can further aid in segmentation. While prior information has been used for segmentation in Materials Science [e.g. Krill and Chen, Acta Mater. 50, p. 3057, (2002)], the vast majority of segmentation only uses observation information. A wealth of prior information is available in Materials Science, examples being capillarity (boundary smoothing), diffusion (equalizing intensity), and particle size distributions. This presentation gives some examples of the use of prior information for developing segmentation algorithms that promise to reduce the necessary human interaction for this purpose.

**4:40 PM**

**Characterization of Complex Three-Dimensional Interconnected Microstructures via the Level-Set Method**: *Victor Chan*[1]; Katsuyo Thornton[1]; [1]University of Michigan, Ann Arbor

While the characterization of microstructures containing particles is mostly straightforward, characterizing complex, interconnected microstructures (those present in fuel cell and battery materials) poses a major challenge. Yet, the optimization of microstructures is an essential task toward improving the performance of these materials. We present a new method for characterizing complex three-dimensional microstructures, which utilizes the level-set method and topological measures such as the genus, the number of independent bodies, and the number of handles. The analysis provides the channel width distribution, similar to the particle size distribution for particulate systems. This type of characterization can lead to understanding of the microstructural effect on bulk transport in composite materials. The utility of the method will be illustrated by its application to complex, interconnected structures that form as a consequence of spinodal decomposition (Cahn-Hilliard dynamics) and interfacial motion by mean curvature (Allen-Cahn dynamics), for which composite transport properties have been determined.

**5:00 PM**

**Processing of 3D Data Sets from X-Ray Micro-Tomography of Impulse Atomized Powders**: Denise Thornton[1]; Jon Johansson[1]; *Arash Ilbagi*[1]; Hani Henein[1]; [1]University of Alberta

Avizo®, ImageJ and MATLAB® were used to create mask files for large data sets obtained from X-ray microtomography of impulse atomized powders. This is an essential step in analyzing the 3D microstructure of the samples since the mask file enables the user to distinguish between the interior and the exterior of the particles. Creating the mask file using Avizo® software required almost continuous user involvement and was time consuming. However, it was used as benchmark against other approaches. The mask file created using the ImageJ software was found to be less time consuming but did not preserve the outer surface details. It was found that MATLAB was the most efficient way to create the mask files. The feasibility to remove the x-ray artifacts in the large dataset will also be discussed.

## Ultrafine Grained Materials – Sixth International Symposium: Mechanical Response

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee
*Program Organizers:* Suveen Mathaudhu, U.S. Army Research Laboratory; Mathias Goeken, University Erlangen--Nürnberg; Terence Langdon, University of Southern California; Terry Lowe, Manhattan Scientifics, Inc.; S. Semiatin, Air Force Research Laboratory; Nobuhiro Tsuji, Kyoto University; Yonghao Zhao, University of California - Davis; Yuntian Zhu, North Carolina State University

Monday PM Room: 606
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* Josef Zrnik, Comtes FHT Ltd.; Jingtao Wang, Nanjing University of Science and Technology; Buyang Cao, Johns Hopkins University; Y.G. Ko, Yeungnam University

**2:00 PM**

**Deformation Behavior during Tensile Straining of Nano/Ultrafine-Grained Structures Formed by Reversion in Metastable Austenitic Steels**: Pavan Venkatasurya[1]; Venkata Ramuni[1]; *Sachin Mali*[1]; Jinesh Shah[1]; Sashank Nayak[1]; Devesh Misra[1]; Mahesh Somani[2]; Penti Karjalainen[2]; [1]University of Louisiana; [2]University of Oulu

The deformation behavior of nano/ultrafine-grained structures during tensile deformation has been examined by transmission electron microscopy in metastable austenitic steels. Special fine-grained structures were obtained by controlled reversion annealing of strain-induced martensite. Proper gradual strain hardening by the formation of ultra-fine martensite results in excellent tensile strength-ductility property combination.

**2:15 PM**

**Deformation Behavior of Nanocrystalline Pd-10 At.% Au Alloy Investigated in Compression Mode at Different Temperatures**: *Lilia Kurmanaeva*[1]; Yulia Ivanisenko[1]; Elena Tabachnikova[2]; Hans-Jörg Fecht[3]; [1]Institute für Nanotechnologie, Forschungszentrum Karlsruhe; [2]B. Verkin Institute for Low Temperatures Physics and Engineering, National Academy of Science of Ukraine; [3]Institute of Micro and Nanomaterials

Mechanical behaviour (strength, ductility, mechanims of deformation, strain hardening) of nanomaterials is one of the key topics of modern material science. Here, we present investigation of mechanical properties of nanocrystalline (nc) Pd-10 at.% Au prepared by inert gas condensation. This method allows to produce nc samples having uniform equiaxed microstructure with the mean grain size of 5-10 nm, and of very high purity. The specimens' microstructure was analysed by means of XRD analysis and TEM. Miniature nc samples with a gauge section of 1 mm was tested in compression mode in temperature range between 4.2 and 300 K. Conventional compression tests showed that samples demonstrate high strength (the yield strength was 1.1 and 2.1 GPa at room temperature and 4.2 K, respectively). Strain-rate jump compression tests revealed high strain rate sensitivity. The obtained results of microstructure and mechanical properties are discussed.

**2:30 PM Invited**

**Fatigue Behavior of Highly Nanotwinned Copper**: Carla Shute[1]; Benjamin Myers[1]; Sujing Xie[1]; Troy Barbee[2]; Andrea Hodge[3]; *Julia Weertman*[1]; [1]Northwestern University; [2]Lawrence Livermore National Laboratory; [3]University of Southern California

Nanotwinned metals combine the high strength of nanocrystalline materials with much improved elongation to fracture and good microstructural stability. While stress-strain curves have been reported little is known of the fatigue properties of such material. Tension-tension fatigue tests have been carried out on high-purity Cu specimens made by magnetron sputtering that are comprised of adjacent aligned nanotwinned columns. The average spacing between twin interfaces is around 35-40 nm but individual values vary over a wide range. Cycling in the LCF range leads to increased regions where de-nanotwinning occurs and some secondary twinning is seen. De-nanotwinned regions close to the surface are associated with extended surface depressions that turn into

fatigue cracks. Much of this work was performed in the EPIC facility of the NUANCE Center at Northwestern University. It was partially supported by DOE contract DE-AC52-07NA27344 at LLNL.

**2:50 PM**

**Influence of Stacking Fault Energy on Microstructures and Mechanical Properties of fcc Metals by Equal Channel Angular Pressing**: Yue Zhang[1]; *Jing Tao Wang*[1]; Jin Qiang Liu[1]; [1]Nanjing University of Science and Technology

In the present work 99.98% commercial pure copper, 99.6% commercial pure nickel and 99.6% commercial pure aluminum samples were processed to high strain levels of ~24, ~12 and ~44 by equal channel angular pressing (ECAP) via route Bc, respectively. Microstructures and mechanical properties are investigated by TEM observations, tensile tests and microhardness tests. It shows that grain sizes of pure copper, pure nickel and pure aluminum has been severed refined from several tens of microns into several hundreds of nanometers after ECAP processing, however, microstructure of copper are mainly consisted of equiaxed (sub) grains with illegible grains/ subgrains boundaries after a true strain level of ~8 by ECAP; while it is featured as lamellar boundaries in that of pure nickel and as elongated grains in that of pure aluminum after same ECAP processed to 8 passes. Different trends of mechanical properties upto ECAP strain levels is observed for different metals.

**3:05 PM**

**Microstructure and Tensile Properties of Ultrafine Grained, TRIP-Aided Low-Carbon Steel**: *Young Gun Ko*[1]; C.W. Lee[2]; S. Namgung[2]; D.H. Shin[2]; [1]Yeungnam University; [2]Hanyang University

Transformation-induced plasticity (TRIP)-aided carbon steels have been regarded as one of the promising candidates for automotive applications due to its good mechanical properties. Thus, the present study investigates microstructure and mechanical properties of the ultrafine grained, TRIP-aided low-carbon steel subjected to equal channel angular pressing and isothermal heat treatment. In tension tests at room temperature, the ultrafine grained, TRIP-aided steel shows much excellent combination of strength and ductility as compared to the coarse-grained counterpart due to its fine grain size of each constituent phase despite nearly same volume fraction. This result can be explained by concept of enhanced plasticity associated with strain-induced transformation during tension deformation.

**3:20 PM**

**Ultra-High Strength Aluminum Nanocomposites**: *Julie Schoenung*[1]; [1]University of California, Davis

Over the last several years, work has been ongoing to prepare and characterize bulk nanocrystalline metal matrix materials consisting of aluminum and boron carbide. These materials, fabricated by means of cryomilling to produce the nanostructured aluminum matrix and clean metal-ceramic interface, followed by powder metallurgy based consolidation methods (including isostatic pressing, extrusion and forging), have demonstrated compressive strengths of over 1000 MPa, which is extremely high for an aluminum alloy system. Furthermore, these materials exhibit high elastic strain when deformed at high strain rates. The microstructural features observed within these materials, which have been studied in great detail using a wide range of techniques, provide insight into the uniqueness of these materials and the properties they exhibit. Select modeling techniques have also been applied to further explain the observed behavior. This paper will present an overview of these research activities.

**3:40 PM Break**

**3:55 PM Invited**

**Grain Size Effects on Rate Sensitivity of FCC Metals**: *K. Ramesh*[1]; Emily Huskins[1]; Buyang Cao[1]; [1]Johns Hopkins University

Processes such as severe plastic deformation, E-beam evaporation, and cryomilling have become increasing popular techniques for producing ultra-fine grained (UFG) materials. These materials show a significant increase in strength as compared to their coarse grain counterparts; however, grain size effects extend beyond just strengthening. In UFG materials a new length scale, the grain size, is introduced which limits the available space within grains for dislocation motion. Therefore, both the deformation mechanisms and the strain rate sensitivity of the material experience grain size effects. This talk focuses specifically on UFG FCC metals including high purity aluminum, commercially pure aluminum, and an aluminum alloy. The dynamic response of the UFG materials is investigated in both compression ($10^3$ $s^{-1}$) and shear ($10^5$-$10^6$ $s^{-1}$) experiments.

**4:15 PM**

**Microstructures and Mechanical Properties of Ultrafine Grained Medium Carbon Steel Processed By HPT at Increased Temperature**: *Jozef Zrnik*[1]; Reinhard Pippan[2]; Stephan Scheriau[2]; Libor Kraus[1]; [1]Comtes FHT Ltd.; [2]Austrian Academy of Sciences

High pressure torsion method at increased temperature of 400°C was applied to refine microstructure in AISI 1045 steel. Deformation behavior of the steel was executed under different shear deformation and constant pressure of 7 GPa. The shear stress evolution and measurement of the torque were recorded. Transmission electron microscopy was used to characterize the microstructure development. Grain refinement was observed after the first turn at disc periphery. In disc centre, the structure had moderately deformed features. The effective strain increase, set an equilibrium between the fragmentation cementite phase and new grains restoration processes, led to saturation of the refinement process. Upon tensile testing, the yield strength and ultimate strength increased with increasing eeq. A small decrease in the hardness across the disc was measured after execution of 4 turns, which may be related to formation of fine grain structure in the disc and structure recovery in disc centre.

**4:30 PM**

**Mechanical Evaluation of Heavily Drawn Fe-Ni-Mn Martensitic Steel**: Hadi Ghasemi-Nanesa[1]; *Mahmoud Nili Ahmadabadi*[1]; Hassan Shirazi[1]; [1]University of Tehran

Fe-10wt%Ni-7wt%Mn martensitic steel categorized as a high strength steel which has excellent age hardenability.The steel shows a very good ductility in non-aged martensitic condition which means, it is a suitable material for severe plastic deformation (SPD) at room temperature. To study the effect of SPD on the mechanical properties of steel heavy cold rolling and wire drawing were used. Total strain in this process was about e ˜ 7. A sample with cross section of 22×8 mm was used which finally deformed to wires with 0.6 and 0.45 mm in diameters. After wire drawing process, x-ray diffraction pattern showed some peaks of austenite in the microstructure while before SPD process, the x-ray shows austenite free specimen. Microhardness results showed the hardness of thinner wire is lower than thicker one and formation of austenite after SPD was studied and mechanical properties of different wire were also measured.

**4:45 PM Invited**

**Mechanical Properties of Nanocrystalline Tantalum within a Wide Range of Strain Rates**: Zhiliang Pan[1]; Weihua Yin[1]; Xiaolei Wu[2]; Brian Schuster[3]; Laszlo Kecskes[3]; *Qiuming Wei*[1]; [1]University of North Carolina at Charlotte; [2]Institute of Mechanics, CAS; [3]US Army Research Lab

We have investigated the mechanical properties of nanocrystalline tantalum within a wide range of strain rates: from quasi-static rate (~$10^{-3}$ $s^{-1}$) to dynamic rate (~$10^3$ $s^{-1}$). Grain size below 40 nm has been achieved through high-pressure torsion, and is thus free of artifacts associated with bottom-up processing routes. The microstructure of the NC tantalum is characterized by equi-axed grains of high angle grain boundaries. Grain interiors are decorated with high density dislocations revealed by HRTEM. Quasi-static mechanical behaviors have been evaluated by means of nanoindentation at different loading rates, and by microcompression of pillars fabricated by focused ion beam technique. Quasi-static stress-strain curves show elastic-nearly perfect plastic behavior. Dynamic uniaxial compression via Kolsky bar technique is used to examine the high rate deformation and failure behavior of the NC tantalum. Experimental results are compared with those of other bcc metals such as W, V and Fe with various microstructures.

**5:05 PM**

**Deformation Twinning in High-Strain-Rate Sheared Nanocrystalline Aluminum**: *Buyang Cao*[1]; Bin Li[1]; Nitin Daphalapurkar[1]; En Ma[1]; K. Ramesh[1]; [1]The Johns Hopkins University

Nanocrystalline aluminum films with grain sizes of 50 nm to 100 nm were made through E-beam evaporation. The films were then subjected to high-rate shearing deformations with strain rates of $10^5$~$10^6$ $s^{-1}$. The experimental configuration is a compression-torsion Kolsky bar, where the specimen is a thin film on a silicon wafer ring. Strain rates during the shearing are determined from the measured shear waves in the bars. Deformed regions are observed on the sample surface by SEM. Site-specific TEM samples are prepared using Focused Ion Beam micromachining to investigate the regions of large plastic

deformation. Deformation twins and stacking faults are found to develop under the high-strain-rate shearing. The formation of twinning in pure aluminum with comparatively large grain sizes and the high-strain-rate promotion of twinning are discussed. The interactions of stacking faults/twins were also observed. Based on HREM observation, possible deformation mechanisms are proposed.

**5:20 PM**

**Methods for Improving Ductility in Nanostructured Titanium Prepared via Powder Metallurgical Routes**: *Osman Ertorer*[1]; Troy Topping[1]; Ying Li[1]; Yonghao Zhao[1]; Wes Moss[2]; Enrique Lavernia[1]; [1]University of California - Davis; [2]Toyota Racing Development

Bulk nanostructured metals are very well known for their high strength. Recent studies in the field aim to provide high strength without sacrificing ductility for advanced engineering applications. Current study focuses on the methods for improving ductility of cryomilled titanium. Several strategies presented are based on experimental results using a comparative approach. Accordingly, influence of cryomilling media, consolidation methods, addition of coarse grains and heat treatments were discussed.

**5:35 PM**

**Strain Rate Sensitivity of Ultrafine Grained Boron Carbide Reinforced Aluminum Metal Matrix Composites**: *Rustin Vogt*[1]; Zhihui Zhang[1]; Troy Topping[1]; Enrique Lavernia[1]; Julie Schoenung[1]; [1]University of California, Davis

Boron carbide particulate reinforced cryomilled aluminum alloy composites produced through various consolidation methods exhibit significant differences in microstructural and mechanical performance. The microstructural differences such as grain sizes and their distribution were characterized in detail, and the relationships between grain size and work hardening rate and strain rate sensitivity were studied in the characteristic length scale 150-2000 nm. The role of geometrically necessary dislocations (e.g., due to thermal expansion mismatch strain) on the mechanical behavior was investigated for select microstructures.

## Ultrafine Grained Materials – Sixth International Symposium: Processing Technologies

*Sponsored by:* The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee, TMS: Shaping and Forming Committee

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Monday PM Room: 607
February 15, 2010 Location: Washington State Convention Center

*Session Chairs:* M. Ravi Shankar, University of Pittsburgh; Sergey Dobatkin, A.A. Baikov Institute of Metallurgy and Materials Science of RAS; Gencaga Purcek, Karadeniz Technical University; Deliang Zhang, The University of Waikato

**2:00 PM Invited**

**A New Biaxial Extrusion Method to Create Sheets of Ultrafine Grain Size**: *Amit Ghosh*[1]; Rick Lee[1]; [1]University of Michigan

A new method of extruding a billet into sheet containing fine grain size is reported. This method permits extrusion of billet in 360 degree direction rather than extruding one direction at a time as used in some Severe Deformation Processes. This process helps achieve effective grain refinement without requiring a repetitive process. However, the extrusion die has flexibility to introduce multi-step extrusion with biaxial stretching within the same process and helps eliminate multiple steps. Grain sizes of 0.5 micron has been achieved with Ti alloy and Mg alloys. This is work-in-progress, reported under a new US Patent application in 2009, and in the talk we will report process details and mechanical properties. (work supported by Boeing Airplane Co.)

**2:20 PM**

**Ideal Engineering Materials by High Rate Severe Plastic Deformation**: *M. Ravi Shankar*[1]; Shashank Shekhar[1]; Jiazhao Cai[1]; [1]University of Pittsburgh

Traditionally, severe plastic deformation at small strain rates has been used to create nanostructured metals. These nanostructures are strengthened by incoherent high angle grain boundaries that are unfortunately typified by poor ductility and stability. Here, we demonstrate opportunities for engineered interface structures in fine grained metals by High-Rate Severe Plastic Deformation (HRSPD) to achieve a much more optimal balance of strength, ductility and stability. HRSPD under large strain but high strain-rate conditions was explored within the prototypical framework of large strain machining in an array of metal alloys including Cu, 70-30 brass and Inconel 718. These studies illustrate opportunities for creating, in a single deformation pass, fine-grained metals that are either multimodal nanostructure distributions or densely nanotwinned or composed of low-energy, low-mobility dislocation structures, each offering intriguing property combinations. The creation of these nanostructures is traced to the peculiar thermomechanical conditions inherent to HRSPD.

**2:35 PM**

**Mechanical and Dry Sliding Wear Behavior of Ultrafine-Grained AISI1024 Steel Processed Using Multi Axial Forging**: Aditya Padap[1]; *Gajanan Chaudhari*[1]; S.K. Nath[1]; [1]IIT Roorkee

AISI1024 steel was severely deformed by using warm (500°C) multiaxial forging (MAF) technique using up to 9 MAF passes. The initial coarser grains of average 17 μm size subdivided into submicron sized grains. Grain refinement is confirmed using TEM. After warm MAF, the strength related properties improved significantly, although total elongation values decreased with increasing strain steps. In present study, the tribological properties of ultrafine-grained low carbon steel produced by multiaxial forging have been investigated. Dry sliding was carried out against the counter face of a hardened and polished disk made of En-32 steel (HRC 62 to 65 hardness) in ambient environment and at varying load and constant sliding speed. The wear test results showed that the strengthening of AISI1024 steel by MAF processing does not lead to the improvement of wear resistance at least for the load and the sliding speed used in this study.

**2:50 PM Invited**

**Nanostructured Materials by Mechanical Alloying: New Results on Property Enhancement**: *Carl Koch*[1]; Ronald Scattergood[1]; Khaled Youssef[1]; Ethan Chan[1]; Yuntian Zhu[1]; [1]North Carolina State University

Mechanical alloying or milling – the ball milling of powders-is an effective and well studied method for the synthesis of nanostrucctured materials. Of the severe plastic deformation methods, it is the most consistent in preparing materials with the smallest nanocrystalline grain sizes. This talk will focus on recent results from our laboratory and the literature in the preparation of materials with property enhancement due to their nanocrystalline microstructures. The results of nanocrystalline Mg-based alloys with very high strength will be presented. A ternary nanocrystalline Cu-Al-Zn low stacking fault energy alloy with high strength and good ductility will also be discussed. Finally, attempts to enhance the thermoelectric behavior of p-type (Bi2Te2.7 Se0.3 ) and n-type (Bi0.4 Sb1.6 Te3 ) thermoelectric materials will be presented.

**3:10 PM**

**Microstructure and Mechanical Properties of IF-Steel Sheets after Equal-Channel Angular Sheet Extrusion (ECASE) and Subsequent Annealing**: *Gencaga Purcek*[1]; Onur Saray[1]; Ibrahim Karaman[2]; [1]Karadeniz Technical University; [2]Texas A&M University

The conventional ECAE has disadvantages for commercial applications, such as limited scalability and low uniformly deformed material yield. Also, long sheets are not available due to the discontinuity of the extrusion process. Therefore, several attempts have been made to overcome these problems and to transform ECAE into a continuous process. We have recently developed an ECAE tool called “equal-channel angular sheet extrusion (ECASE)” for processing of IF-steel sheets. IF-steel sheets were processed using ECASE system to various passes. After processing, the microstructure and mechanical properties were investigated. Moreover, the deformed sheets were annealed to enhance the formability of IF-steel sheets. Continuous shear deformation of the sheets successfully refined the grain size down to submicron upon multi-pass ECASE. As a result, abnormal improvement in yield strength exceeding 150%

was noted with remarkable increase in tensile strength. Annealing of the sheets after multi-pass ECASE led to improvement in formability of them.

**3:25 PM**

**Severe Plastic Deformation of a Pearlitic Steel by Wire Drawing**: *Reiner Kirchheim*[1]; Shoji Goto[1]; Christine Borchers[1]; [1]University of Göttingen

Cold drawing of pearlitic steel wires with total strains of up to 6 leads to a fiber like structure of ferrite and cementite lamella with thicknesses of a few tens of nanometers or a few nm, respectively. Maximum flow stresses of up to 6 GPa are obtained this way. Atom probe analysis reveals that the carbon concentration in cementite is reduced whereas the carbon concentration within the ferrite may reach a few at. %. In addition, the cementite becomes amorphous and, therefore, will contribute remarkably to the strength of the material. Further plastic deformation during ball milling leads to a total dissolution of cementite and yields nanocrystalline Fe-C alloys, where carbon is partitioned between saturated grain boundaries and ferrite grains. The formation of defects (vacancies, dislocations and grain boundaries) during severe plastic deformation and their interaction with carbon atoms is discussed based on the presently available experimental data.

**3:40 PM Break**

**3:55 PM Invited**

**Strength-Ductility Combination of Nanostructured 316L Stainless Steel Processed by Means of Dynamic Plastic Deformation (DPD)**: G.Z. Liu[1]; N.R. Tao[1]; *K. Lu*[1]; [1]SYNL, Institute of Metal Res. CAS

316L stainless steel was processed by means of dynamic plastic deformation (DPD, i.e., plastic deformation at high strain rates). Single-phased (austenite) bulk nanostructured stainless steel specimens were prepared, consisting of nano-sized grains embedded with nano-scale twin bundles. Tensile properties of the as-prepared DPD samples with different amounts of nano-twin bundles and grain sizes have been systematically investigated to reveal the effects of grain size and twin density on strength and ductility. Subsequent thermal annealing results in partial recrystallization of the nanostructured steel, forming a mixture structure of coarse-grains embedded with nano-twin bundles. Strength and ductility of the stainless steels with different structure characteristics are analyzed with emphasis of optimization of the strength-ductility combinations.

**4:15 PM**

**Structure, Texture, and Mechanical Properties of Copper and a Mg-Al-Zn-Mn Alloy after Constrained Groove Pressing**: *Sergey Dobatkin*[1]; Vladimir Serebryany[1]; Jozef Zrnik[2]; [1]A.A. Baikov Institute of Metallurgy and Materials Science of RAS; [2]COMTES FHT

The structure, texture, and mechanical properties of copper and a Mg-Al-Zn-Mn alloy after by constrained groove pressing (CGP) at room temperature to a true strain of ~7 (for copper) and at temperatures of 300-400C to a true strain of ~2.3 (for Mg alloy) have been studied. The CGP of copper leads to the formation of submicrocrystalline structure, increases the strength characteristics (YS up to 350 MPa). The final structure of the Mg-Al-Zn-Mn alloy is determined by the competition of the fragmentation processes and the dynamic processes of recovery and recrystallization. The maximum yield strength (YS = 216 MPa) was obtained after CGP at 350C. The Hall–Petch relationship in the Armstrong approximation is shown that, as the orientation factor N for the prismatic slip increases, the activity of prismatic slip and the plasticity decrease, and the strength increases. This effect was confirmed experimentally.

**4:30 PM**

**Improvement of Strength and Wear Resistance of Cp-Ti by ECAE and MAO Processes**: *Akgun Alsaran*[1]; Gencaga Purcek[1]; Yenal Vangolu[1]; Onur Saray[1]; [1]Ataturk University

Pure titanium are used in many applications ranging from biomedical to aerospace. However, the low strength and very poor wear resistance of CP-Ti limits its use in biomedical applications. In this study, CP-Ti (grade-2) was processed via multi-pass equal-channel angular extrusion (ECAE) using route-Bc in order to improve its strength and subsequently the processed samples were coated by micro-arc oxidation (MAO) for enhancing their wear resistance. The microstructural evolution, and tensile and wear properties of the processed and coated materials were investigated. It was found that ECAE-processed CP-Ti exhibited a significant increase in strength with a slight decrease in ductility. This process didnot lead to any improvement in its wear resistance. However, the MAO process after ECAE resulted in a considerable increase in wear resistance of CP-Ti without decreasing its bulk strength. Consequently, high strength CP-Ti with improved wear resistance was produced by combining the ECAE and MAO processes.

**4:45 PM Invited**

**Severe Plastic Deformation Processes for Thin Samples**: *Rimma Lapovok*[1]; Arnaud Pougis[2]; Dmitry Orlov[1]; Laszlo Toth[3]; Yuri Estrin[4]; [1]Monash University; [2]CSIRO; [3]Université Paul Verlaine-Metz; [4]Monash University / CSIRO

Among the known severe plastic deformation techniques, one group can be defined as SPD processing of thin samples. Their distinctive feature is that one of the sample dimensions, namely the thickness, is much smaller than the other two dimensions. The well-known process of High Pressure Torsion, as well as the less-known Cone-on-Cone Method and the High Pressure Tube Twisting process are all in this category of SPD techniques. For all of them, a severe shear strain is imposed within the thickness of the sample due to the difference in magnitude of the material flow velocities at two large surfaces, rather than by a change in the velocity direction. The microstructure, texture and mechanical properties of copper samples deformed by all three SPD processes mentioned will be reported and compared with those obtained by equal-channel angular pressing as a reference bulk forming SPD technique.

**5:05 PM**

**Tailoring Materials Properties of Aluminium Alloys by Sandwich-like Structures with Accumulative Roll Bonding**: *Tina Hausöl*[1]; Heinz Werner Höppel[1]; Mathias Göken[1]; [1]Friedrich-Alexander-University Erlangen-Nürnberg

Accumulative roll bonding (ARB) is used to produce ultrafine-grained materials with extraordinary mechanical properties. In this work ARB is used to tailor the material's properties by producing sandwich-like structures. The high strength aluminium alloy AA5754, after 4 ARB cycles (N4), is used as the core material. To achieve high corrosion resistance and good visual properties, it is cladded with commercial purity aluminium AA1050 (N4) at room temperature and alternatively with AA6014 (N4) at 230 °C. All materials show an ultrafine-grained microstructure and satisfactory bonding between the layers of the different aluminium alloys. Nanoindentation measurements reveal that there is a sharp transition in hardness at the interface. The yield and tensile strength of the core material are fully retained in the case of the AA6014/AA5754 sandwich material. The strength of the AA1050/AA5754 sandwich material is slightly reduced compared to the core material but still twice as high as the clad material.

**5:20 PM**

**Comparison of the Mechanical Properties for Equally Strained Ultrafine Grained Al 99.5 Produced by Accumulative Roll Bonding and Equal Channel Angular Pressing**: *Andreas Böhner*[1]; Verena Maier[2]; Heinz Höppel[1]; Mathias Göken[1]; [1]University of Erlangen-Nuernberg; [2]ZMP

Significantly increased strain rate sensitivity (SRS) in ultrafine-grained (UFG) metals it is commonly observed. It is also widely accepted that the enhanced SRS is the key issue for improved ductility of UFG metals.In order to evaluate the influence of the process - equal channel angular pressing (ECAP) or accumulative roll bonding (ARB) - on the mechanical properties, UFG aluminium of technical purity (Al 99.5) was produced using these methods. For this purpose quite similar total deformation strains of eARB=6.4 and eECAP=6.3 were selected. For both conditions the microstructure and the mechanical properties were investigated. For the same testing geometry significant differences in the ductility of the ARB and ECAP material are observed. The results will be discussed in terms of microstructural differences, texture effects and damaging mechanisms.

**5:35 PM**

**Synthesis of Bulk Nanostructured and Ultrafine Structured Metallic Materials by Thermomechanical Consolidation of Nanostructured Powders**: *Deliang Zhang*[1]; Aamir Mukhter[1]; Amro Gazawi[1]; Vijay Nadakuduru[1]; [1]The University of Waikato

Nanostructured metallic powders including nanostructured metal matrix composite powders can be effectively produced by high energy mechanical milling (HEMM). However, consolidation of such powders to produce high quality bulk nanostructured and ultrafine structured metallic materials which are highly desirable for numerous practical applications is high challenging. We take the challenge by utilising thermomechanical powder consolidation techniques such as powder compact forging and powder compact extrusion

which are also combined with rapid powder compact heating. A number of experiments on Cu, Al, and Ti based metallic materials have been done along this line, and some very interesting and informative results have been obtained. Theoretical considerations on the mechanisms and scientific principles underlying the thermomechanical consolidation of nanostructured metallic powders have also been developed to guide the experimental work. This talk is to present and discuss the major findings from the experimental study and theoretical thinking.

**5:50 PM**

**Preliminary Investigation of Novel Micro-Scale Current Activated Tip-Based Sintering (μ-CATS)**: *A. El-Desouky*[1]; S. Chang[1]; S. Kassegne[1]; K. Moon[1]; K. Morsi[1]; [1]San Diego State University

Spark Plasma Sintering (SPS) has emerged as a process with unique advantages such as lower sintering temperatures and shorter holding times than conventional sintering, in addition to the production of materials with unique microstructures and properties. However, the process has been largely limited to the production of bulk materials with simple geometries on the macro-scale. In this paper preliminary experimental and modeling results on novel current activated tip-based sintering (CATS) of ultrafine and nano-nickel powder are presented. CATS enables the selective sintering of micro-scale features using a moving or stationary (electrically conductive) tip configuration. A finite element model was also developed to investigate current and temperature distributions under typical CATS conditions.