2008 Nanomaterials: Fabrication, Properties, and Applications: Processing and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: Wonbong Choi, Florida International University; Seong Jin Koh, University of Texas at Arlington; Donna Senft, US Air Force; Ganapathiraman Ramanath, Rensselaer Polytechnic Institute; Seung Kang, Qualcomm Inc

Wednesday AM Room: 273
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Pawel Keblinski, Rensselaer Polytechnic Institute; Douglas Chrisey, Rensselaer Polytechnic Institute

8:30 AM Invited
Optical Assembly of Nanomaterials: *Paul Braun*[1]; [1]University of Illinois at Urbana-Champaign

The assembly of nanomaterials into higher order functional structures has remained a challenge, even as routes for the synthesis and chemical functionalization of nanomaterials has exploded. As a potential solution, we believe optically directed assembly may prove to be a powerful route for the assembly of nanomaterials into higher order complex and functional structures. Through optical holography, we have assembled semiconductor quantum dots and various ceramic nanoparticles into predefined 2 and 3 dimensional patterns within polymeric and multifunctional hosts. Also through optical holography, we have formed three-dimensionally patterned polymers and oxides. In both these systems, the resolution and 3-D control of structure obtained far surpassed that obtainable through conventional lithographic routes. In a related project, we have utilized laser tweezers to manipulate and assemble ZnS and Au nanoparticles inside of a self-assembled photonic crystal, giving us the ability to controllably place individual defect states within an ordered photonic lattice.

9:00 AM
Alkaline-Earth Metal Hexaboride One-Dimensional (1D) Nanostructures: Synthesis and Characterization: Syed Amin[1]; Shu-you Li[2]; John Roth[3]; *Terry Xu*[1]; [1]University of North Carolina, Charlotte; [2]Northwestern University; [3]University of Illinois, Chicago

In this presentation, we report our recent progress on synthesis and characterization of alkaline-earth metal hexaboride (MB_6, M=Sr, Ba) 1D nanostructures. The MB_6 1D nanostructures are appealing candidates for applications such as thermoelectric energy conversion, nanocomposites and nanoelectronics. Catalyst-assisted growth of MB_6 1D nanostructures was achieved by pyrolysis of diborane ($B_2 2H_6$) over alkaline-earth metal oxide (MO) or alkaline-earth metal carbonate (MCO_3) powders at elevated temperature (~890-960°C) and low pressure (~165 mTorr). Nickel, gold and palladium are effective catalytic materials. The as-synthesized MB_6 1D nanostructures were characterized by scanning electron microscopy, transmission electron microscopy, and Raman spectroscopy. Results show that the MB_6 nanostructures are single crystalline with preferred growth direction along [001]. The MB_6 nanostructures are several tens of nanometer in diameter and up to ten micrometer in length. The growth of these nanostructures involved the vapor-solid growth mechanism. Thermal stability of MB_6 1D nanostructures has also been studied.

9:15 AM
Synthesis of 1-D Nanostructures of Trititanate Thin Films on a Titanium Metal Flake: *Yupeng Guo*[1]; Nam-Hee Lee[1]; Hyo-Jin Oh[1]; Cho-Rong Yoon[1]; Kyeongsoon Park[1]; Sun-Jae Kim[1]; [1]Sejong University

One-dimentsional (1-D) layered trititanate nanotubes, nanoribbons films and self-assembled spherical aggregates were prepared by hydrothermal reacting Ti metal flake with concentrated NaOH solution. A combined TEM, SEM and XRD investigation of the reaction products as the functions of reaction temperature, duration, concentration of NaOH solution and post treatment conditions revealed that the 20-50 nm wide and ca. 1μm long nanoribbons with a 5 mol dm^{-3} NaOH aqueous solution at 140°C for 12 h. The 6-8 nm wide and several hundreds nanometer long nanotubes are prepared by using 10 mol dm^{-3} NaOH. Titanate sheets growth, split and roll-up model for the formation of 1-D titanate thin films was proposed.

9:30 AM
Wet Chemical Synthesis of Highly Aligned ZnO Nanowires with Optical Properties: *Jean-Claude Tedenac*[1]; Mezy Aude[1]; Ravot Didier[1]; Tichit Didier[1]; Gerardin Corinne[1]; [1]Institut Gerhardt Universite de Montpellier 2

ZnO nanowires have a great commercial stake, due to their physical and chemical properties. Many methods have been employed for the growth of ZnO nanomaterials (sputtering, chemical vapour deposition, molecular beam epitaxy, metal-organic chemical vapour deposition) which greatly improve the crystalline quality. However, low cost and simplicity of the synthesis processes are required for commercial applications. The wet chemical synthesis route seems to meet these requirements, enabling the preparation of high crystal quality and proper growth orientation of ZnO nanowires. Nethertheless, control of the ZnO size, morphology, dimensionality, and self-assembly remains a very important stake, due to their tight influence on ZnO properties. Efficient control of both nanostructure dimensionality and assembly is obtained by using appropriate synthesis parameters during the seeded growth process. The synthesis of well aligned nanowires with a perpendicularly growth to the substrate and a high crystal quality is useful for the study of their physical properties.

9:45 AM
Characterization of Electrodeposited Nanocrystalline Al-Mg Powders: *Fereshteh Ebrahimi*[1]; Mahesh Tanniru[1]; Sankara Sarma Tatiparti[1]; [1]University of Florida

Nanocrystalline aluminum-magnesium alloys were fabricated in the powder form using electrodeposition technique. The electrolyte was oganometallic based and the synthesis was conducted in low oxygen argon atmosphere. XRD profiles showed that these powders consist of supersaturated fcc-Al(+Mg) and hcp-Mg(+Al) phases. Compositional analysis using EPMA revealed that the fcc phase can accommodate up to 20at%Mg, however the hcp phase may dissolve as high as 40at% of Al. SEM observations indicated that the hcp-Mg phase develops over the supersaturated fcc-Al phase. TEM studies showed that both phases consist of nanocrystalline grains. These powders were developed for hydrogen storage applications. Selected powders were hydrogenated at various temperatures and high pressures. In this presentation the results of characterization before and after hydrogenation will be presented. The financial support by NSF through grant DMR-0605406 is gratefully appreciated.

10:00 AM
Influence of Processing Mode on the Nanostructure Development of Flux Melted Ag-40at%Cu Alloy: *D Aujla*[1]; C. Davy[1]; P. Kalu[1]; K. Han[2]; T. Shen[3]; D. Alexander[3]; R. Schwarz[3]; [1]Florida Agricultural and Mechanical University-Florida State University-College of Engineering; [2]National High Magnetic Field Laboratory; [3]Los Alamos National Laboratory

The need to generate higher magnetic field has led to renewed research into the development of high strength conductor materials. This paper presents a comparison of the microstructure, texture and strength in Ag-40at%Cu alloy fabricated by two methods: drawing and cold rolling. The AgCu alloy was initially produced by flux melting technique and cast into 25.4 mm rods, and then severely deformed by either method to a true strain of 4.6. Several techniques including scanning electron microscopy, X-ray diffractometry and Nanoindentation were utilized in analyzing the material. It was found that both processing method produced nano-composites with alternating Ag and Cu laminates on the order of 60 to 200nm in thickness. A correlation was made between the microstructure and strength of the material. The general effect of processing mode on the mechanical properties of the material is discussed.

10:15 AM
Sonochemical Synthesis of Nanostructured Anatase and Study of the Kinetics among Phase Transformation and Coarsening as a Function of Heat Treatment Conditions: *Leonardo Gonzalez-Reyes*[1]; Isaias Hernández-Pérez[2]; Francisco Robles Hernandez[3]; Hector Dorantes Rosales[1]; Elsa Arce Estrada[1]; [1]Instituto Politecnico Nacional; [2]Universidad Autónoma Metropolitana; [3]Transportation Technology Center, Inc.

The present paper shows that nanostructured anatase can be produced by sonochemical synthesis with an average particle size of 6.4nm and a specific surface area of 300m2g-1. The results of microstructure coarsening evolution

as well as phase transformation of nanometric anatase to rutile subjected to different heat treatment conditions are presented. The rates of transformation from anatase to rutile, critical particle size of transformation anatase to rutile and coarsening are directly related to heat treatment temperature, which in turns allows developing a mathematical model to predict particle size evolution. The developed model can be used to precisely predict particle size for anatase and/or rutile using exponential like equations similar to those described in the LSW theory. The exponent of the LSW-like equations went from ~1/8 to ~1/4 as heat treatment times increase from 8 and 72 h respectively. This research was supported by means of BET, TGA, XRD and TEM.

10:30 AM

Initial Grain Growth Behavior during Sintering of Nanosized Powders: *Hongtao Wang*[1]; Zhigang Fang[1]; [1]University of Utah

It is well known that rapid grain growth occurs in the final stage of the sintering of coarse powders. However, in the case of nanosized powder, notable grain growth has taken place during initial and intermediate stage of sintering, leading to loss of nano-structure. So it is critical for nanosintering to control initial grain growth in order to get full densification with final grain size on nanoscale. This paper is trying to understand enhanced grain growth behaviour during initial stage of sintering of nanosized powders from experiments observation to theoretical analysis by considering the unique issues of nanostructure such as high ratio of surface/interface area, non-equilibrium defects (vacancies, dislocations). The results show that the enhanced grain growth is related to recovery process during relaxation of excess energy in nanostructured materials.

10:45 AM Break

11:00 AM Invited

Magnetic Nanomaterials for Energy Efficient Systems: *Raju Ramanujan*[1]; [1]Nanyang Technological University

Nanostructured magnetic materials are being intensively studied for novel energy efficient systems. Giant energy product magnets, high temperature magnetocaloric nanomaterials and high permeability soft magnetic nanomaterials for innovative permanent magnet MRI systems, solid state cooling and low loss transformer systems, respectively, are focus areas of our work. Exchange coupling at the nanoscale can be used as a common guiding principle to improve the magnetic properties; efficient coupling requires optimum microstructural control. Hence, mechanisms of nanocrystallization were studied for melt spun ribbons and powders using DSC, XRD, CTEM, EDX, VSM and in situ TEM techniques. Synergistic compositional changes at the crystal:matrix interface were found to play a key role in determining the crystal morphology, crystal size and nucleation density. Microstructural control, and associated change of magnetic properties, of specific magnetocaloric materials, nanocomposites with high energy product and high permeability magnets based on FeCo-B, Fe-Ni-Mo-B and Fe-Si-B-Nb-Cu alloy systems will be described.

11:30 AM

Co-Rich Nanocrystalline Soft Magnetic Ribbons with Improved Mechanical Strength: *Maria Daniil*[1]; Todd Heil[1]; Matthew Willard[1]; [1]Naval Research Laboratory

Co-rich nanocrystalline have great technological interest due to their excellent response at high frequency magnetic fields and high temperature magnetic behavior. In this work we examine the crystallization behavior, crystal structure and mechanical properties of $(Co,Fe,Ni)_{88}Zr_7B_4Cu_1$ and $(Co_{0.95}Fe_{0.05})_{88}Zr_6M_1B_4Cu_1$ (M = Zr, Nb, Hf and Ta) nanocrystalline ribbons. According to the X-ray diffraction measurements, the amorphous as-spun ribbons crystallize partially into a very fine mixture of FCC and BCC phases (7-8 nm) at low temperature, whereas at higher temperature it transforms into coarser FCC (20-28 nm) and $Co_{23}Zr_6$ grains. The lattice size of the BCC phase is ~2.828 Å, which is larger than the bulk (Fe,Co)-BCC phase. Bend test experiments showed a significant improvement of the fracture strength in samples with the highest Co content (83.6 at.%), whereas M substitutions for Zr add an extra small improvement. These results will be discussed in correlation with the fracture surface microstructure.

11:45 AM

Magnetic Properties of FePt: A Comparison between Nanodots and Continuous Thin Film: *Gopinath Trichy*[1]; Jagdish Narayan[1]; [1]North Carolina State University

We report on the synthesis and magnetic properties of L10 ordered FePt nanodots using pulsed laser deposition (PLD). Size reduction to nanoregime in magnetic systems can lead to favorable changes in coercivity, remanence and domain reversal mechanisms. For an off-stoichiometric Fe41Pt59 system, we have shown that individual nanodots have higher coercivity (3200 Oe) when compared to a continuous thin film (2250 Oe). Improved coercivity for the nanodot system was attributed to the transition to single domain state. Another unique feature of this study is the integration of the FePt nanodots with Si (100). Remarkably, we achieved FePt growth along the magnetically hard c-axis using epitaxial TiN as a template buffer. Cross section and plan view TEM analysis showed that the FePt nanodots have an average size = 20 nm and thickness = 5nm. The magnetic storage density of such a system can potentially exceed 1 Tbit/inch2.

12:00 PM

Atomic-Scale Segregation and Fluctuations in FePt Thin Films: *Karen Torres*[1]; Sundar Naga[2]; Jennifer Thompson[2]; Richard Martens[1]; Gregory Thompson[1]; [1]University of Alabama; [2]West Virginia State University

A series of atom probe and transmission electron microscopy studies have been performed to investigate how compositional fluctuations in FePt contribute in the A1 to $L1_0$ phase transformation and grain coarsening. Fe_xPt_{1-x} and $(Fe_xCu_y)_zPt_{1-z}$ thin films have been sputter-deposited onto Si substrates. The addition of Cu lowers the ordering temperature but also accelerates the grain growth in films during the phase transformation. Atom probe specimens were analyzed in an Imago Local Electrode Atom Probe (LEAP). The atom probe reconstruction showed small levels of Pt segregation and clustering at grain boundaries in the as-deposited films. These results provide experimental verification of modeling predictions that Pt will segregate to free surfaces in FePt to lower the total free energy of the system. The effect of different times and temperatures on segregation, ordering and grain morphology will be addressed.

12:15 PM

Millisecond Regime Flash Annealing of FePt Thin Films: *Amanda Cole*[1]; Ron Ott[2]; Timothy Klemmer[3]; Chandan Srivastava[1]; James Harrell[1]; Gregory Thompson[1]; [1]University of Alabama; [2]Oak Ridge National Laboratory; [3]Seagate Technology

A pulsed-thermal-processing technique using a high density infrared plasm arc lamp with a radiant spectrum of 0.2 to 1.4 μm was used to facilitate the A1 to $L1_0$ phase transformation in FePt. The 20 nm and 100 nm films were annealed using single and multiple 100 or 250 ms pulse widths. XRD, TEM and AGM characterization confirmed that the time-temperature exposures studied were able to achieve $L1_0$ ordering. TEM micrographs confirmed that the mean grain size grew slightly with increasing time or temperature but was within the standard deviation of the as-deposited grain size. At lower annealing temperatures, a shoulder to the A1 {111} XRD peak was observed and indexed as $L1_0$, consistent with the nucleation of the $L1_0$ phase as a first-order phase transformation. At elevated temperatures, the $L1_0$ shoulder was not prevalent but the {111} peak qualitatively narrowed, consistent with the growth of the $L1_0$ phase.

12:30 PM

Hydrogen Uptake Properties of Nickel Doped Titanate Nanotubes: *Nam-Hee Lee*[1]; Hyo-Jin Oh[1]; Yeong-Ung Yun[1]; Yupeng Guo[1]; Kyung-Sub Lee[2]; Sang-Chul Jung[3]; Sun-Jae Kim[1]; [1]Sejong University; [2]Hanyang University; [3]Sunchon National University

Ni doped titanate nanotubes were hydrothermally synthesized using titania (TiO_2) nanoparticles with rutile phase as a starting material in 10M NaOH aqueous solution with $NiCl_2$. It was fully characterized by various techniques, including SEM, TEM, ICP, FT-IR, BET, and P-C-T test that conversions from nanoparticles to nanotubes were achieved at various temperatures and times for hydrothermal reaction. The Ni doped TNTs were found to be a multi-walled layered structure with an average outer diameter of ~ 10 nm, an inner diameter 2 ~ 4 nm and ~ 0.75 nm interlayer distance under optimized conditions. The hydrogen uptake amount of the Ni doped TNTs was shown more than 2.0 wt%

into many mesoporous adsorption sites of interlayers and interiors of nanotube at room temperature under 20 atm.

12:45 PM

Preparation of Hexagonal Barium Ferrite Nanoparticles by Carbon Combustion Synthesis: *Karen Martirosyan*[1]; Long Chang[2]; Dan Luss[1]; Dmitri Litvinov[3]; [1]University of Houston, Department of Chemical and Biomolecular Engineering; [2]University of Houston, Department of Electrical and Computer Engineering; [3]University of Houston, Department of Electrical and Computer Engineering, Department of Chemical and Biomolecular Engineering

We used high efficient carbon combustion synthesis method to produce well crystalline hexagonal barium ferrite nanoparticles (50-100nm). In this method exothermic oxidation of carbon nanoparticles ~5nm with surface area 80m2/g generates a thermal reaction wave with temperature of up to 1000C that propagates through the solid reactants (oxides and/or carbonates) mixture converting it to the desired complex oxide product. The carbon is not incorporated in the product and is emitted as a gas CO_2 from the sample. A stable self-propagating reaction front can be obtained only at carbon concentrations exceeding 6.5wt.%. A complete conversion to hexagonal structure was obtained only for carbon concentration exceeding 11wt.%. Solid-state interactions between the components and the nucleation of the nanoparticles started in the early period of combustion and continued in the post combustion zone. The magnetic properties Hc~2800Oe and Ms~50emu/g of the compact sintered ferrites compared well with those produced by other synthesis methods.

3-Dimensional Materials Science: Modeling and Characterization across Length Scales II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Michael Uchic, US Air Force; Eric Taleff, University of Texas; Alexis Lewis, Naval Research Laboratory; Jeff Simmons, US Air Force; Marc DeGraef, Carnegie Mellon University

Wednesday AM Room: 286
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Mike Groeber, Ohio State University; Martin Glicksman, University of Florida

8:30 AM

Finite Element Modeling of the Deformation of 3D Polycrystals Including the Effect of Grain Size Distribution: *Wei Li*[1]; Nicholas Zabaras[1]; [1]Cornell University

A finite element analysis of the large deformation of 3D polycrystals is presented using pixel-based elements as well as elements conforming with grain boundaries. The macroscopic response is obtained through volume-averaging laws using a recently developed homogenization model. A constitutive framework for thermo–elastic-viscoplastic response of single crystals is utilized along with a fully-implicit Lagrangian finite element algorithm for modeling microstructure evolution. The effect of grain size distribution is included by considering a physically motivated measure of lattice incompatibility which provides an updated shearing resistance within grains. A domain decomposition approach is adopted for parallel computation to allow efficient large scale simulations. The computed mechanical properties of polycrystals are shown to be consistent with experimental results for different grain size distributions.

8:50 AM

Measuring the Evolution of the Crystal Scale Stress State in Polycrystalline Copper during Cyclic Loading: *Jun-Sang Park*[1]; Matthew Miller[1]; Alexander Kazimirov[1]; [1]Cornell University

The micromechanical response of polycrystalline materials under cyclic loading is not well understood. Due to complicated microstructure and anisotropic single crystal mechanical properties the stress state of the crystals in the polycrystalline material can be significantly different from that imposed on the polycrystalline aggregate. In our study, cyclic loading was applied on recrystallized copper specimens. The loading was paused to perform high energy x-ray diffraction (XRD). Using XRD, the lattice strains and their evolution with respect to specimen life for many crystallographic planes of the crystals in the aggregate were measured. The lattice strain measurements were then used to compute the lattice strain distribution function (LSDF) which is the elastic strain tensor field over crystallographic orientation space. Using the LSDF and anisotropic elasticity, the crystal stress distribution over orientation space was computed and its evolution was monitored at different points in specimen life.

9:10 AM

Realistic Microstructure Based 3D Indentation Modeling of Heterogeneous Materials: *Arun Sreeranganathan*[1]; Arun Gokhale[1]; [1]Georgia Institute of Technology

Instrumented indentation, which provides a continuous record of the indentation load P as a function of the depth of penetration h of the indenter into the material, is a widely used technique to study the mechanical behavior of small microstructural volumes. Indentation modeling of heterogeneous materials such as particulate reinforced composites is of interest so as to study the effect of the second phase particles on the indentation response of the material. In this contribution, we incorporate realistic 3D microstructures of particulate reinforced metal matrix composites, reconstructed from serial sectioning data, in finite element based simulations of microindentation. Effect of microstructural parameters such as the reinforcement volume fraction, spatial distribution etc. on the indentation response will be reported.

9:30 AM

3D Evolution of Precipitates in Binary Aluminum-Lithium: *Martin Glicksman*[1]; Ben Pletcher[1]; James Lebeau[2]; [1]University of Florida; [2]University of California, at Santa Barbara

Phase coarsening in overaged Al-Li alloys is a diffusion-controlled process. Large particles grow by dissolution and mass transfer from smaller particles. Eight binary Al-Li alloys were aged for times between 3-240 h, to yield various distributions of δ' (Al3Li) precipitates. Transmission electron microscopy was used to image 10-100 nm diameter, spherical δ' precipitates via centered dark-field techniques. TEM images were then autonomously analyzed using a novel Matlab® function to process and provide good statistical 3D results. Computer analysis provides objective characterization and fast image processing, allowing practical access to larger sample sizes. Results, including the particle size distribution and maximum particle size, agree with predictions from diffusion screening theory.

9:50 AM Break

10:10 AM

3D Microstructural Theories and 3D Experimental Techniques: Martin Glicksman[1]; *Paulo Rios*[2]; [1]University of Florida; [2]Universidade Federal Fluminense

Traditionally, materials scientists developed microstructure theories that relied on experimental quantities measured from planar sections. With the advent of 3D simulation of microstructures, and some recent advances in 3D characterization, this situation changed dramatically. Specifically, it is now possible, and desirable, to test microstructure theories with 3D data. Two theories will be examined along with the measurements required to test them. First, a proposed extension is presented of Johnson-Mehl, Avrami, Kolmogorov theory to situations where phase nuclei are not randomly located. This theory requires measurement of the Gaussian curvature of the interfaces, which may be extracted from the 3D microstructure. Second, a recent treatment of grain growth kinetics is presented in terms of its metrical and topological properties. In this case, measured quantities include the distribution of grain volumes and/or the distribution of the number of faces per grain. Again, these require microstructural measurements are only available via 3D characterization.

10:30 AM

Flow in Mushy Zones Using 3D Measurements of Microstructure: *Dongchoul Kim*[1]; Roberto Mendoza[2]; Peter Voorhees[3]; Katsuyo Thornton[2]; [1]Sogang University; [2]University of Michigan; [3]Northwestern University

Dendritic microstructures that form during solidification modify fluid flow in a complex manner. While there have been investigations relating the solid volume fraction and other microstructural characteristics to permeability, a fundamental understanding of fluid flow in complex microstructures has

not been fully developed. Using experimentally obtained three-dimensional reconstructions of microstructures, we determine the steady-state flow field using the Navier-Stokes equation and the permeability using Darcy's law. The calculated values of permeability are compared to those predicted by previous investigations. This approach provides a method for assessing the empirically and theoretically obtained permeabilities and the limits of their applicability in realistic solidification microstructures.

10:50 AM
Porosity Distribution and Effective Elastic Modulus in ZrN as a Surrogate for PuN: Comparisons between 2-D and 3-D Measurements and Models: *Manuel Parra Garcia*[1]; Sung-Ho Park[2]; Kirk Wheeler[1]; Pedro Peralta[1]; Ken McClellan[3]; [1]Arizona State University; [2]Gyeongsang National University; [3]Los Alamos National Laboratory

A serial sectioning process was used to develop a three-dimensional (3D) representation of the microstructure of a ZrN dense pellet sintered in Argon at 1600°C. Twenty five two-dimensional (2D) images of the microstructure obtained by imaging with an optical microscope at 200X (slides spaced 2µm apart from each other) were used as a basis for reconstructing the 3D microstructure of the ceramic for modeling using Finite Elements (FE). This representation allows the quantification of the spatial distribution of porosity in the samples as well as the creation of a three-dimensional finite-element model (FEM) that accounts for the presence of pores. The porosity distribution, Young's modulus and stress-strain behavior of the reconstructed volume were predicted from the 3D model of the microstructure and correlations with existing two-dimensional (2D) models and experimental results obtained at Los Alamos National Laboratory (LANL) are discussed. Work supported under DOE/NE Agreement # DE-FC07-05ID14654.

11:10 AM
Self-Assembly of Three-Dimensional Composite Materials with Multifunctionality: *Hsun-Yi Chen*[1]; Yongwoo Kwon[2]; Peter Voorhees[2]; Katsuyo Thornton[1]; [1]University of Michigan; [2]Northwestern University

Multifunctional materials are of increasing interest because they facilitate the design of compact systems able to perform a variety of tasks. Composite materials are inherently suited for multifunctionality since desired properties can be achieved by hybridizing different materials. In recent research, microstructures having minimal-surface morphologies have been found to optimize simultaneous transport under certain symmetrical constraints. However, fabrication of microstructures with minimal-surface morphologies is challenging, often requiring special materials processing techniques. We find that bicontinuous structures resulting from coarsening after phase separation possess transport properties similar to those with minimal-surface morphologies. Our finding demonstrates that spinodal decomposition and other phase-separation processes may provide a plausible route to inexpensive self-assembly of three-dimensional two-phase composites with highly optimized multifunctionality.

Alumina and Bauxite: Additives

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Sringeri Chandrashekar, Rio Tinto Aluminium Limited; Peter McIntosh, Hatch Associates

Wednesday AM — Room: 296
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Songqing Gu, Chalco

8:30 AM Introductory Comments

8:35 AM
New Crystal Growth Modifiers for Bayer Process: *Dimitri Kouznetsov*[1]; Jianjun Liu[1]; Kevin O'Brien[1]; James Counter[2]; John Kildea[2]; [1]Nalco Company; [2]Nalco Australia

In Bayer process, Crystal Growth Modifiers (CGM) help to increase alumina trihydrate yield and improve quality. Recent work aimed at mechanistic issues has led to the development of a second generation of CGM. Traditional, first generation products were well known to impact three critical process areas, trihydrate particle sizing, oxalate stability, and oxalate morphology. While some plants utilize all three properties to enhance and control trihydrate precipitation, it has long been recognized that all three properties are not always desirable and that products with a more selective impact could provide even greater benefits. The development of the second-generation CGM product range has focused not only on developing products that are more effective, but also products that are more selective in their properties. Results on a range of new formulations are presented.

9:00 AM
The Effect of DSP Structure on Removal of Impurities from Refinery Liquor and Efficiency of Alkali Recovery Process: *Alyona Kuvyrkina*[1]; Natalia Kuznetsova[1]; Anatoliy Lapin[1]; Alexander Suss[1]; Andrey Panov[1]; [1]Russian National Aluminium-Magnesium Institute

The influence of temperature and sulpho-, carbo-, chlorid ions containing in alumina refinery liquors on formation of different modifications of sodium hydroaluminosilicate (desilication products - DSP) obtained as a result of bauxite digestion was investigated. To solve the problem of impurity anions removal from recycled liquors the relation between adsorptive properties of DSP and conditions of its formation was examined. The effect of DSP structure and composition was studied in terms of alkali recoverability in the course of red mud treatment with lime. The possibility was shown of recovery up to 96% of alkali.

9:25 AM
Performance Appraisal of Evaporation System with Scale Inhibitor Application in Alunorte Plant: *Ayana Oliveira*[1]; Juarez Moraes[1]; Jefferson Batista[1]; Jorge Lima[1]; Ricardo Diniz[2]; Emiliano Repetto[2]; [1]Alunorte; [2]Cytec Do Brasil Ltd.

Any alumina refinery plant has problems with sodalite scaling in heater exchange and pipes that promote a reducing in the life time of the evaporation train and other losses. Alunorte in conjunction with Cytec Industries Inc., has performed a plant trail with a bayer process scale inhibitor like a alternative action to reduce these losses in the process. This paper reports the plant trail results where the scale inhibitor achieved significant results like: increase the life time of the evaporation train, stability of the heater transfer coefficient, increase the availability of the evaporation and the condensate rate, improving on the operational maintenance safety. Additional benefits of this new polymer are currently under investigation.

9:50 AM
Cost Savings by Improved Filtration and Washing of Al-Hydrate Product: Reinhard Bott[1]; Thomas Langeloh[1]; *Juergen Hahn*[1]; [1]Bokela GmbH

A new generation of pan filters enables filtering and washing of Al-Hydrate product with improved capacity, reduced maintenance and operation cost. Modern pan filters have many innovative features like the Forced Feeding System, fast flow cells or the effective heel re-slurry system. The even filter cake and the excellent hydraulic improve cake washing and reduce wash water consumption by 25% which leads to significant evaporation cost saving. Steam can be used to improve cake washing and reduce cake moisture. The effective heel re-slurry system increases cycle times between filter cleaning and improves cloth lifetime. Thus, energy and operation cost reduce 10-15% compared to conventional pan filters. Another promising option enabled by the new filter technology is to replace the first washing step on pan filters by more compact and more efficient disc filters and re-slurrying the filter cake with wash filtrate from the pan filter.

10:15 AM Break

10:30 AM
Autoprecipitation Control with HXPAM Technology: *Qi Dai*[1]; Scott Moffatt[1]; Morris Lewellyn[1]; [1]Cytec Industries Inc

Autoprecipitation poses serious problems to alumina refineries, such as more raw material consumption, alumina loss, scaling of equipment, unstable process, lower liquor productivity, etc. Cytefc's SUPERFLOC HX® has been used as a main settler flocculant for over 1½ decades. It is known that the HX flocculant reduces autoprecipitation and settler scale in the presence of red mud solids. In fact, all hydroxamic acid-containing polymers of varying molecular weights

can reduce gibbsite autoprecipitation. One particular product, SUPERFLOC HX-800, shows enhanced performance in reducing auto-precipitation in both settlers and washers. This paper presents laboratory results of HXPAM as an autoprecipitation inhibitor.

10:55 AM

Application and Performance Characteristics of Superfloc® TF-8000 - A New HXPAM Based Hydrate Flocculant: Franklyn Ballentine[1]; *Scott Moffatt*[1]; Pat Clement[2]; [1]Cytec Industries Inc.; [2]Ormet Alumina Company

HXPAM's emulsions have been shown to be very effective in improving overflow clarity and underflow flowability in tertiary trays, however the need for specialized equipment to invert the emulsions coupled with the need for high dilution in most applications has previously made them less convenient to use. Superfloc® TF-8000 is a HX based polymer designed for application in tertiary trays. It can be applied neat or pre-diluted with spent liquor. Application of Superfloc® TF-8000 in secondary trays has also been very successful because of its ability to significantly improve the flowability of the hydrate. This has expanded the possibility for application in other hydrate handling areas where flowability is a concern. This paper provides laboratory data comparing the performance of Superfloc® TF-8000 to dextran as well as other HX polymers. The paper also summarizes the results obtained when applied to the secondary trays for several months at Ormet.

Aluminum Alloys: Fabrication, Characterization and Applications: Corrosion and Protection

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials

Wednesday AM Room: 293
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials; Yansheng Liu, SECAT Inc

8:30 AM

Naval Structural Aluminum Alloy Research: *Catherine Wong*[1]; [1]Naval Surface Warfare Center

Recent focus on light-weight, high-speed naval ships has caused the Navy to re-evaluate aluminum as a structural material. Aluminum alloys generally offer good strength-to-weight ratio and corrosion resistance, which are both important properties in Navy ships. Light weight ships offer both higher speed and increased payload capacity, expanding mission capabilities. Although aluminum has been widely used in Navy patrol craft, small ship hulls, deckhouses, hydrofoils, and many other applications it has not been without problems. These problems resulted in the initiation of several programs which focus on understanding and coping with the four key aluminum material issues: reduced as-welded strength, low corrosion-fatigue strength in unpainted structures, in-service sensitization, and strength reduction during fires. This paper will present an overview of these programs.

8:50 AM

Effect of Grain Size and Substructure on Sensitization in 5083 Al Alloys: *Kinga Unocic*[1]; Michael Mills[1]; Glenn Daehn[1]; Paul Kobe[2]; [1]Ohio State University; [2]Alcan Rolled Products

This study focuses on the effects of microstructure on the intergranular corrosion (IGC) resistance of modified 5083 aluminum alloys. Previous research has shown that small additions of Cu and Zn to AA5083 can improve the resistance to IGC. Our present research shows that the same Cu and Zn modified composition can become susceptible to IGC following sensitization if the microstructure is altered. To demonstrate this effect, the material was heavily rolled then subjected to various sensitization heat treatments. For example, following a sensitization treatment at 150°C for 20hrs the heavily worked microstructure was virtually resistant to IGC. When compared to a sample that was given a short heat treatment in order to alter the heavily deformed microstructure, followed by the same sensitization heat treatment, resulted in IGC. Mg-rich grain boundary phases were identified through TEM characterization methods and were linked to the decreasing resistance to IGC.

9:10 AM

The Effect of Grain Boundary Character on the Intergranular Corrosion Susceptibility of 2124 Aluminum Alloy: *Lisa Chan*[1]; Anthony Rollett[1]; Gregory Rohrer[1]; Hasso Weiland[2]; Soonwuk Cheong[2]; [1]Carnegie Mellon University; [2]Alcoa Inc.

The objective of the research is to find a correlation between grain boundary character and intergranular corrosion in a 2124 aluminum alloy. In the present study, the alloy was subjected to different solutionizing temperatures and corrosion tested with full immersion in a sodium chloride and hydrogen peroxide solution according to ASTM G110 standards. The samples that were heat treated at 40°C above the standard solutionizing temperature of 500°C have shown susceptibility to intergranular corrosion. The grain orientations of the corroded samples were then obtained from automated Electron Back-Scatter Diffraction (EBSD). Preliminary results on the relationship of grain boundary character and intergranular corrosion will be presented.

9:25 AM

The Corrosion Behaviour of Al–Zn Alloys in Abakaliki and Port Harcourt Rainwater: Ndubuisi Idenyi[1]; Israel Owate[2]; *Cajethan Okeke*[3]; [1]Ebonyi State University; [2]University of Port Harcourt; [3]University of Nigeria

A study has been carried out on the corrosion behaviour of Al–Zn alloys exposed to rainwater sourced from Abakaliki, Ebonyi State, a highly residential urban town and Port Harcourt, Rivers State, a highly industrialized urban town, both in Nigeria. Corrosion test coupons of four (4) differing alloy grades of varying Zn concentrations produced at First Aluminium Plc., Port Harcourt under carefully controlled atmosphere were immersed in beakers containing each of the rainwater separately. The setup was allowed to stand for 30 days with a set withdrawn 5-daily for corrosion rate evaluation using the normal procedures. Results showed that all the alloy grades followed the usual corrosion trends associated with passivating metals, but that samples exposed to Abakaliki rainwater were more severely attacked possibly due to the heavy presence of clouds of carbonate particles in the atmosphere generated from predominant quarrying works in the town which must have made the rainwater somewhat acidic as compared to the possible presence of flared glasses in Port Harcourt resulting from petrochemical refining operations that predominate the town.

9:45 AM

The Corrosion Susceptibility and Electrochemical Impedance Spectroscopy of Al-Cu-Mg-Zr New Aerospace Aluminum Alloy: Sheng Yang[1]; Sujuan Yao[1]; *Danqing Yi*[1]; Huiqun Liu[1]; [1]Central South University, School of Materials Science and Engineering

In order to evaluate the degree and severity of Al-Cu-Mg-Zr alloy in 3.5%NaCl corrosion solution, the pit corrosion susceptibility and electrochemical impedance spectroscopy(EIS)of Al-Cu-mg-Zr alloy with different corrosive grades was studied to analyze the dynamic process of corrosion behavior by TEM and electrochemical. Compared with 2524-T4 aluminum alloy, Al-Cu-Mg-Zr alloy is good in corrosion property, Cu-rich particles are the main influence factor of pitting corrosion behavior. The impedance of EIS is characterized by a high frequency capacitive arc which gives information on the performance of the unattacked surface. At lower frequencies, a diffusion control is apparent suggesting the specific contribution of the surface in which the localized attack takes place. the micro-process and the influent mechanics of interface reaction and the electrode surface condition are studied by EIS and equivalent circuit.

10:00 AM

Effect of Mn as Fe Corrector on Microstructure and Adhesive Wear of Eutectic Al-Si Piston Alloy: *Dheerendra Dwivedi*[1]; Mohit Dhiman[1]; R. Sehgal[1]; I. Bhat[1]; [1]Indian Institute of Technology, Roorkee

During the processing of cast piston alloys, iron frequently enters into the melt as an impurity and causes porosity and needle shaped Al-Fe-Si intermetallics. Literature however didn't reveal many reports on the effect of Fe and its intermetallics on wear behavior piston alloys. Therefore to increase the understanding in this area, an investigation has been undertaken to explore the effect of Fe (0.2 to 2.0) and consequently Fe-rich intermetallics and "Fe correctors" such as Mn in different Fe/Mn ratios. Sliding wear behavior was evaluated on pin-on-disk tribometer in the range of normal loads (1-4kg) at

2.0m/s speed. Metallurgical characterization of alloys were done by using image analyzer, scanning electron microscope (SEM), EDAX and EPMA. Sliding wear behaviour have been discussed in light of microstructure, particularly Fe-rich intermetallics and mechanical properties with and without the presence of "Fe corrector".

10:20 AM Break

10:30 AM

Comparative Analysis of the Corrosion Susceptibility of Al–Zn Alloys in De-Ionized Water, Rainwater and Seawater: Ndubuisi Idenyi[1]; C. Okeke[2]; Israel Owate[3]; *S. Neife*[4]; [1]Ebonyi State University; [2]University of Nigeria, Nsukka; [3]University of Port Harcourt, Choba; [4]Enugu State University of Science and Technology

A study of the corrosion behaviour of aluminium – zinc alloy systems exposed to de-ionized water, rainwater and seawater has been carried out. Test coupons of four (4) grades of Al alloys with differing weight percentages of Zn were prepared and immersed in baths containing each of the environments separately. They were allowed to stand for a 30-day period with a set withdrawn every 5 days for weight loss measurements and subsequent corrosion rate profile evaluation using standard equations in mm/yr. The results obtained showed that samples exposed to seawater were more susceptible to corrosion and that the degree of severity increased with increased zinc content. This is attributed to the presence of dissolved salts and other complexes in seawater and increased grain boundaries in the alloys as more zinc was added; both conditions that favour corrosion attacks.

10:50 AM

The Effect of Zinc Additions to the Corrosion Rates and Tensile Strengths of Aluminium System: *Israel Owate*[1]; Ephariam Chukwuocha[1]; [1]University of Port Harcourt

Zinc oxide concentrations of 0.2778, 0.3012, 0.3821, 0.4942 and 0.5915 wt % were added to a mixture of the composition of an aluminium system. The designed component systems were produced into rectangular flat sheets of aluminium products measuring about 5mm thickness. Standard corrosion tests by weight loss method was applied to the samples under acid and base-media environment for 740 hours. Thereafter, the rates of corrossion, damages caused and tensile strengths were determined. The results obtained indicated progressive increase of corrosion rates from deionized water, base to acid medium. Whereas the tensile strengths decreased in the reverse. In addition, the corrosion rates increased relative to zinc concentrations except sample C which deviated from the trend. Sample C showed high values of tensile strengths and lower corrosion rates when compared to other specimens. This initial result presupposes that controlled zinc additions could be applied to achieving lower corrosiveness.

11:10 AM

The Role of Segregation in the Eutectic Modification of Hypoeutectic Al-Si Alloys: *Kazuhiro Nogita*[1]; Masato Yoshiya[2]; Hideyuki Yasuda[2]; Arne Dahle[1]; [1]University of Queensland; [2]Osaka University

Modification of the eutectic silicon in hypoeutectic Al-Si alloys, which causes a structural transformation of the silicon phase from a needle-like to a fine fibrous morphology, is carried out extensively in industry to improve mechanical properties. It is known that the fibrous silicon phase in chemically modified alloys is heavily twinned. This impurity induced twinning has been explained by a twin plane re-entrant edge (TPRE) model. However, there are still some questions and contradictions that remain. In this paper, we discuss the mechanism of impurity induced twinning and modification in light of experimental data obtained by electron microscopy and synchrotron radiation, as well as predictions by first-principles calculations.

11:30 AM

The Effect of Chromium on Aluminum Base Alloy: *Hua Shen*[1]; Guangchun Yao[1]; Weidong Yang[2]; [1]Northeastern University; [2]Shengyang Institute of Chemical Technology

Aluminum was noticed for its good characteristics of low density, high stress, easy machining and rich resource etc. But its application was limited by poor corrosion-resisting, hardness and mechanical strength. In this work, Cr was added to Al-Mg-Mn-RE complex alloy. Then it was compared with aluminum and Al-Mg-Mn-RE alloy in hardness and mechanical strength, finally the better property was found. This was because Cr had particular character in aluminum base alloy by observing the microstructures of alloys. Hardness and mechanical tests were carried out at room temperature. The results showed that the property of aluminum base alloy was improved.

11:50 AM

Influence of Aging Time of Fracture Toughness Mechanisms of Al-Li Alloys: Yongxin Wang[1]; Zheng Chen[1]; Ri Ma[1]; *Yanli Lu*[1]; [1]Northwestern Polytechnical University, State Key Laboratory of Solidification

The fracture toughness mechanisms in aluminum-lithium alloys aged for different time was studied. Based on semi-quantity statistical and analytical results, we specially discuss the effects of aging time and the condition of stress on delamination. The results show that the percentage of delamination increase, the percentage of dimples decrease and little of quasi-cleavage can be found at the beginning zone with aging time prolonged. The maim fracture features is delamination at crack stable growth zone and is quasi-cleavage in near unstable crack growth zone. The percentage of delamination is increase at crack stable growth zone and decrease in near unstable crack growth zone with aging time prolonging. The aging time and the crack growth stage can take apparently effect on delamination percent, the longer aging time, the earlier delaminating, and those influence the effect of extrinsic toughness mechanism further.

12:10 PM

The Effect of Heat Treatment on the Microstructure and Hardness of A356 Aluminium Alloy: *M. Faraji*[1]; L. Katgerman[2]; [1]Netherlands Institute for Metals Research (NIMR); [2]Delft University of Technology (TUDelft)

The effect of heat treatment on the microstructure and macro-hardness of A356 aluminium alloy was investigated. Quantitative characterization of eutectic silicon showed a reduction of mean diameter and perimeter by ~3 and 5 times and an increase in aspect ratio, shape factor and elongation by a factor of ~5, 3 and 6 times, respectively; as a consequence of T6 heat treatment of the alloy. In this study the solution treatment and quenching condition remained constant and the artificial aging condition altered. The hardness results showed that among artificial aging temperatures of 155, 170, 200 and 240°C for a constant time of 2 hrs, aging at 170 and 200°C gives the highest hardness value (~1.5 times higher than the value for the as-cast sample). However, the hardness of the as cast alloy was doubled by solutionising, quenching treatment followed by aging for 1hr at 200°C. In comparison, aging at 170°C needs a time of 16 hrs to produce nearly the same hardness.

Aluminum Reduction Technology: Aluminum Industry in Mid-East: Joint Session with Electrode Technology Symposium

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee

Program Organizers: Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Aluminium Tech; Carlos Zangiacomi, Alcoa Aluminum Inc; John Johnson, RUSAL Engineering and Technological Center LLC

Wednesday AM
March 12, 2008

Room: 298/299
Location: Ernest Morial Convention Center

Session Chair: Abdulmunim Binbrek, DUBAL

8:30 AM Keynote

ALBA's Vision on Sustainable Development: *Mahmood Daylami*[1]; [1]Aluminium Bahrain

ALBA has taking a leading role in the aluminum production in the Middle East since its foundation in 1968. The company steadily grew its production capacity to more than 830,000 mt today. Along the increase in production capacity the workforce grew as well and training/education of people became an essential part of the success today. The fast growing aluminum industry in the Middle East with a number of new smelter projects in the pipeline will create competition and make the Gulf Region to become potentially one of the fastest growing areas in aluminum production but without being free from challenges regarding energy, employment and environment.

9:00 AM Invited
Aluminium Smelting Greenhouse Footprint and Sustainability: *Jeffrey Keniry*[1]; [1]Alumination Consulting Ltd

The continuing growth of smelting capacity in the Middle East and Russia, and the concurrent decline in Europe and the USA, demonstrate the vital importance of low-cost power in underpinning the industry's competitiveness. More recently, the greenhouse footprint of the smelter adds another dimension to competitive advantage, which will have increasing economic significance as concerns for global warming intensify. In this context, the power source itself may confer a competitive advantage well beyond the supply cost to the smelter. Direct greenhouse gas emissions from best-practice smelters are in the range 1.6-2.0 tonnes CO_2-e per tonne of aluminium, while the indirect emission attributable to power generation imposes an additional zero (hydro and nuclear sources) to as high as 16 tonnes CO_2-e for power sourced from low-grade coal. The implication of this indirect greenhouse footprint on the future competitive advantage of alternative smelting locations is discussed.

9:25 AM Invited
Development of Aluminum Production Capacity in the Middle East and Siberia: *Halvor Kvande*[1]; [1]Hydro Aluminium AS

Aluminum production is expected to grow outside the US and Europe. New capacity is being developed in areas with available and affordable energy and small domestic markets. The Middle East is an area of particular interest for both Brownfield and Greenfield smelters. Both Alba and Dubal have expanded their production in Bahrain and Dubai to above 800 000 tpy. Construction of Greenfield smelters has started in Qatar and Oman, and there are plans to build new smelters in Abu Dhabi and Saudi Arabia. The Qatalum project in Qatar will be one of the largest primary aluminum plants ever built, with 585 000 tpy in the first step. The site and infrastructure are planned for expansions up to 1.2 million tonnes. Qatalum will then be a mega plant, with a very competitive cost position. Another area with available energy is Siberia, and the world's largest aluminum producer, UC Rusal, has announced the construction of the Taishet aluminum smelter near Irkutsk, equipped with Rusal's new 400 kA cells. Also other Greenfield and Brownfield projects are under way in Siberia.

9:50 AM Invited
Rusals Expansion Based on Hydropower: *Horst Peters*[1]; [1]EN+ Group Ltd

Since March 2007 United Rusal is ranked by Primary Production the largestAluminum Producer in the world. The majority of this Primary Production is located in Sibiria with the smelters Bratsk and Krasnojarsk each 1 Mio.t/a production as well as Sayansk with 850.000 t/a and Irkutsk with 300.000 t/a production. Energy basis for all of these smelters is Hydropower from the huge Jennisei and Angara river system. The installed capacity is 25.000 MW.More can be developed.

10:15 AM Break

10:35 AM Invited
DX Technology: The Development and the Way Forward: Abdulla Kalban[1]; *Yousuf Alfarsi*[1]; Abdelhamid Meghlaoui[1]; [1]Dubai Aluminium Company Limited

Dubai Aluminium Company Limited (DUBAL) developed and implemented Reduction Cell technologies, D18 (185kA), CD20 (200kA), D20 (240kA) between 1990 and 2007 for six Brownfield expansions which increased production from 135,000 t/y (1979) to 940,000 t/y (2008). DUBAL continued in the path of high amperage Reduction Cell development, and introduced the DX technology as its latest in-house technological development. 5 DX prototype cells were commissioned from September to December 2005 at an operating amperage of 325 kA. In 2007, through a series of improvements to the cell design and components, anode configuration, shell, process control, busbars, superstructure and operational practices, and based on a learning curve associated with the 5 Prototype Cells, an improved version of DX cell targeting 340 – 350 kA was designed and a section of 40 cells is currently under construction to be commissioned during the first quarter of 2008. This paper summarizes the design features of the DUBAL DX Reduction cell technology, and operation performance of the five prototype cells for two years.

11:00 AM
Alba Line 5 Expansion - Fives Solios Solutions for a Better Environment: Maxime Depinoy[1]; *Thierry Malard*[1]; [1]Solios

In spring 2003, Aluminium Bahrain B.S.C.(c) (ALBA) undertook to expand the capacity of its Manama Plant (Line 5). Fives Solios has been awarded contracts for the supply of green anode plant, foundry, fume and gas treatment plants. Supported by its experience in the aluminium field and with the help of ALBA Engineer's professionalism, Solios Environnement has been in charge to supply economical solutions to minimize smelters emissions. This paper, focused on environmental challenges, recalls the project parameters including capacity and leading requirements, highlighting some specific solutions used for Gas Treatment Centre (pot gas). Pot room atmosphere study has been carried out in the frame of a cooperation agreement between ALBA and SOLIOS ENVIRONNEMENT. Benefits from the installed over suction system "YPRIOS" are described, and key factors to optimise such system efficiency are discussed.

11:25 AM Invited
Fuelling the Aluminium Industry in the Gulf: Abdulmunim Binbrek[1]; *Moosa Ismail*[1]; [1]Dubai Aluminium Company Limited (Dubal)

The presentation will analyse natural gas as the main fuel to be looked for by the aluminium industry in the Gulf. Gulf countries hold about 70% of the world's proven oil reserves and approximately 40% of the gas reserves. With growing challenges facing the export of natural gas from the Gulf, it is believed that much of the future gas development in the area would be "reoriented' more and more towards domestic and regional markets. That local reorientation of Gulf natural gas is predicted to add value to its exploitation by boosting the industrial development in the region. Aluminium industry experts forecast that by 2020 the global aluminium production will be in the region of 60 million tonnes per annum with 10 million tonnes being produced by Gulf countries.

11:50 AM
China Shakes the World - Chances and Risks of Anode Material Supply: *Paul Adkins*[1]; [1]Paul Adkins and Associates

China's demand for aluminum is set to continue growing at unprecedented rates, thanks to the continuing boom in the economy. The production needed to satisfy this demand is unlikely to come entirely from china's own industry, but it will have to come from somewhere. With the likelihood of 12% growth or more per year over the next five years, this is going to put a lot of stress on the supply of coke and anodes, cathodes, and aluminum fluoride. For Western smelters, the challenges are as great as the rewards awaiting them. The Chinese market is volatile, disjointed and unpredictable, despite the best efforts of we who try to do so. Only those smelters who are 100% committed, who understand the risks involved and who work with and in the market continuously are going to reap the benefits.

Biological Materials Science: Functional Biomaterials

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

Program Organizers: Ryan Roeder, University of Notre Dame; Robert Ritchie, University of California; Mehmet Sarikaya, University of Washington; Lim Chwee Teck, National University of Singapore; Eduard Arzt, Max Planck Institute; Marc Meyers, University of California, San Diego

Wednesday AM — Room: 390
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Ming-Yong Han, National University of Singapore, Institute of Materials Research and Engineering; Raju Ramanujan, Nanyang Technological University

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8:30 AM Keynote
Size/Composition-Tunable Quantum Dots and Their Bioapplications: *Ming-Yong Han*[1]; [1]National University of Singapore, Institute of Materials Research and Engineering

Colloidal semiconductor nanocrystals (quantum dots) have attracted great attention for their distinguished roles in fundamental studies and technical

applications such as biological labeling and optoelectronic devices. In the last two decades, the main efforts have been focused on the preparation of size-tunable binary or core-shell nanocrystals with different emission colors. In our research, we also focus on the development of highly luminescent composition-tunable quantum dots across the whole visible spectrum. The successful preparation of high-quality composition-tunable quantum dots makes the new class of nanomaterials very promising as multicolor biological nanoprobes for imaging, sensing, and drug delivery applications. There is an emphasis on the development of ultrasensitive imaging/spectroscopic detection for multiplexed analysis at cellular or tissue levels. Quantitative multi-parameter analysis of multianalytes is being carried out, which could provide a direct way to identify sets of genes and proteins correlating with certain diseases.

9:10 AM
Biomimetic Assembly of Functional Nanomaterials onto Polymers: *Song Jin*[1]; [1]University of Wisconsin-Madison

We employ a bottom-up, biomimetic strategy to assemble functional nanoscale inorganic materials into microarrays under low temperature, benign chemical conditions following principles derived from biomineralization. Our approach involves the selective functionalization of polymer surfaces to create regions of differing surface energy. By carefully controlling the surface functional groups heterogeneous nucleation can be promoted in designated regions but suppressed in others. We have demonstrated the arrays of CdS thin films and ZnO nanorods using a variety of engineering polymers. CdS materials assembled this way can be used as arrays of high performance photodetctors yet retains the low cost roll-to-roll solution processing on flexible polymer substrates. We will also report our success in using nanoscale self-assembled block copolymers and sequenced biopolymers to assemble nanoscale functional materials.

9:30 AM
Encapsulated Contrast Agent Marker (ECAM) for MRI Prostate Brachytherapy: *Karen Martirosyan*[1]; R. Jason Stafford[2]; James Bankson[2]; Steven Frank[3]; [1]University of Houston, Department of Chemical and Biomolecular Engineering; [2]University of Texas MD Anderson Cancer Center, Department of Imaging Physics; [3]University of Texas MD Anderson Cancer Center, Department of Radiation Oncology

We report a novel Co^{2+}-based contrast agent marker that provides high signal intensity in Magnetic Resonance Imaging (MRI) by shortening the spin lattice relaxation time (T1) of water protons. This contrast agent can be used to identify the radioactive titanium seeds used in prostate brachytherapy. Magnetic characterization of the contrast agent shows superparamagnetic properties with a large magnetic moment. Plastic (acrylic) and glass seeds with Co^{2+}-based aqueous solution (1-10%) with amount ~2 microliters were well visualized by MRI with a relative signal intensity ranging from 10751 to 32767 in a phantom under 1.5 T MRI (T1). Various combinations of plastic/(glass)-titanium-plastic/(glass) and titanium-plastic/(glass)-titanium row of seeds were identified in a dog prostate and the calculations verified the distance to the center of the titanium seeds. The developed marker is referred to as an Encapsulated Contrast Agent Marker (ECAM) and can provide a novel targeted magnetic resonance imaging for brachytherapy application.

9:50 AM
Targeted Dual Therapy for Disease Treatment by Multifunctional Coated Magnetic Nanoparticles: *Raju Ramanujan*[1]; Sibnath Kayal[1]; S. Purushotham[1]; [1]Nanyang Technological University

Coated magnetic nanoparticles have a wide variety of important, novel and unique bioengineering applications. Multifunctional materials for combined drug targeting, triggered drug release and MRI imaging in the context of human cancer treatment will be presented. Chemical synthesis was used to prepare core-shell structures of magnetic nanoparticles coated with a shell of thermosensitive polymers and anti-cancer drug doxorubicin. Heating these particles in an external AC magnetic field results in combined dual therapy of hyperthermia and triggered drug release. Iron oxide nanoparticles were coated with thermo-sensitive polymers based on PNIPA and doxorubicin. The temperature rise and drug release during AC field heating was examined for various magnetic particle sizes, concentration and external magnetic field strength. Our results show that temperature control is feasible and controlled drug release can be accomplished by this novel technique. MRI imaging, in vitro and in vivo studies in a buffalo rat model will be discussed.

10:10 AM Break

10:20 AM
Magnetic Nanocarrier for Tumor-Targeted Drug Delivery: *Qiang Yuan*[1]; Rajesh Venkatasubramanian[1]; Devesh Misra[1]; [1]University of Louisiana

The primary challenge in the delivery of a drug to a tumor site is to target the anticancer drug specifically in and around tumors and at concentrations that would decrease the growth rate and/or viability of the tumors. An excellent vehicle to achieve targeted delivery of anticancer drug in and around tumors is magnetic nanocarrier. We describe here that the magnetic nanocrystals hooked with anticancer agents and encapsulated with novel stimuli-responsive polymer (temperature and/or pH-responsive) are viable drug carriers. The specialized synthesis of the nanocarrier and in-vitro experiments are described for its unique drug delivery properties of effective and controlled release of drug at the targeted site.

10:40 AM
Antibactericidal Function of Composite Nanoparticles Consisting of Doped-Titania Photocatalytic Shell and Nickel Ferrite Magnetic Core: *Rajesh Venkatasubramanian*[1]; Devesh Misra[1]; [1]University of Louisiana

Titania is a potential photocatalytic material in applications involving killing of bacteria and cancer cells. While it can be directly used for bio-applications, it cannot be extracted because it is an electrical insulator. However, the removal of titania can be facilitated with the help of a small magnetic field, if we can synthesize composite particles consisting of magnetic core and photocatalytic shell. The paper describes enhanced antibactericidal function of doped-titania composite nanoparticles. The modification of the band gap of titiania by dopants minimizes the probability of electron-hole recombination process and enhances the effectiveness of destroying bacteria.

11:00 AM Invited
Inkjet Printing of Biopolymers and Cells: *Paul Calvert*[1]; [1]University of Massachusetts

We are using inkjet printing to deposit patterns of silk, collagen and ionic complexes of polypeptides. These guide the growth of cells deposited on the patterns. Yeast has been printed onto agar and the effect of overprinted biopolymer layers on the growth of yeast is being studied. Human fibroblasts and mesenchymal stem cells can also be printed and subsequently grown. This allows organized structures of differing cells and biopolymers to be printed in order to study cell-cell interactions.

11:30 AM
Synthesis, Patterning and Characterization of Biocompatible Parylene Coatings: *Varshni Singh*[1]; V. Gunda[1]; Jost Goettert[1]; [1]Louisiana State University

Microfabrication techniques and scale-up by replication have fueled advances in micro analysis chips and sensor systems for BioMEMS, medical, health, chemical and life science applications. The present and future technology in such areas is driven by the need for "miniaturization" without losing absolutely in terms of biocompatibility. In fabricating device(s) and their parts for these applications, it is extremely difficult to employ only such materials that are biocompatible and inert. Therefore, it is crucial to have a biocompatible coating (such as Parylene), conformably deposited on/in these devices, which is inert and has an excellent biocompatibility. Parylene coatings are known to possess the essential characteristics that includes excellent adhesion, conformal, pinhole free, smooth, lubricious or low friction coefficient, inert, autoclavable, thin, penetrate and coat complex surfaces and able to deposit at room temperature. Present study will discuss the synthesis, patterning and characterization of Parylene Coatings in perspective of BioMEMS devices.

11:50 AM
Antibacterial Effects of Copper Vermiculite against E. Coli, K. Pneumoniae and S. Aureus: Bowen Li[1]; *Jiann-Yang Hwang*[1]; Susan Bagley[1]; [1]Michigan Technological University

Copper vermiculite is a new material with excellent antimicrobial ability. In this study, the antibacterial effects of copper vermiculite against E. coli, K. pneumoniae and S. aureus were investigated by diffusion method, respectively.

The results show that copper vermiculite is efficient to inhibit the reproduction of these common bacteria indicators. The antibacterial mechanism of copper vermiculite is discussed.

Bulk Metallic Glasses V: Glass Forming Ability and Alloy Development

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Wenhui Jiang, University of Tennessee; Guojiang Fan, University of Tennessee; Hahn Choo, University of Tennessee; Yanfei Gao, University of Tennessee

Wednesday AM Room: 393
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Wenhui Jiang, University of Tennessee; Hahn Choo, University of Tennessee

8:30 AM Invited
Glass-Forming Ability as a Function of Various Factors: Intrinsic vs. Extrinsic: *Dmitri Louzguine*[1]; Akihisa Inoue[1]; [1]Institute for Materials Research

Several parameters and factors have been applied so far to predict the glass-forming ability of bulk metallic glassy alloys, and none of them can be treated yet as a comprehensive one to predict the glass-forming ability upon solidification of a liquid phase. In the present work various factors influencing the glass-forming ability are discussed and separated as intrinsic (belonging to a glass itself) and extrinsic (depending upon external conditions) factors. Actual glass-forming ability can be significantly limited by the extrinsic factors. These factors should be also taken into consideration to predict a real glass-forming ability of the alloy.

8:50 AM Invited
Improving Glass Forming Ability of Zr50.5Cu38.5Al11 Alloy by Suppressing the Precipitation of CuZr Phase: S.T. Deng[1]; *Z.Q Hu*[1]; H. F. Zhang[1]; [1]Chinese Academy of Sciences

Based on Zr50.5Cu38.5Al11 alloy, substituting Cu with Ni and Ag can improve glass forming ability greatly. The content of various elements in CuZr phase is analyzed with energy dispersive spectrometer of scanning electron microscope. The XRD patterns verify that adding Ag is very helpful to restrain CuZr phase from precipitating. Especially, with the increment of Ag content in Zr53.5Cu29.5-xNi5All2Agx alloy, the proceeding of suppressing CuZr phase precipitation is shown in the XRD patterns of arc melted ingots. Chemical interaction among various atoms plays an important role in affecting glass forming ability. From ternary alloy to quinary alloy, considering that Ni and Ag have similar chemical and physical properties with Cu, the additions of Ni and Ag cause competitions of Ni and Ag with Cu for Zr and this kind of competition weakens the interaction between Cu and Zr atoms.

9:10 AM
Enhanced Thermal Stability Amorphous Aluminum Alloys: *Timothy Wilson*[1]; Jianming Bai[1]; Hahn Choo[1]; [1]University of Tennessee

Amorphous aluminum alloy powders with compositions of Al_{85} Y_7Fe_8, $Al_{83}Y_7Fe_8Ti_2$, and $Al_{79}Y_7Fe_8Ni_3Ti_2Nd_1$ were developed with crystallization temperatures of 342°C, 400ºC, and 457ºC, respectively. In-situ high-temperature synchrotron diffraction results were correlated with differential scanning calorimetry studies to investigate the structural evolution of the alloy during the crystallization. The results show that, through microalloying with Ti and Nd, the onset-of-crystallization temperature was increased by 115ºC without increasing the melting temperature, and the resulting crystalline products changed from a mixture of fcc-Al and an intermetallic phase in the case of $Al_{85}Y_7Fe_8$ to only intermetallic phases. Pair distribution function studies have revealed insight into changes in the local atomic order.

9:25 AM
An Investigation of Viable Pathways to Achieve Highly Tunable Fe-Based BMG Composites: *Hesham Khalifa*[1]; Kenneth Vecchio[1]; [1]University of California-San Diego

Fe-based BMGs are an intriguing member of the metallic glass family on account of their impressive mechanical properties and reasonably low price. A new Fe-based glass forming system is introduced of the form Fe-Mo-Cr-W-C-B. Alloys of varying compositions within this system were subjected to several heating and cooling treatments resulting in a broad spectrum of microstructures. Comprehensive mechanical, thermal, and XRD analyses indicate that extremely fine control over phase nucleation and crystal size is achievable upon heat treatment from the amorphous state or alternate cooling pathways from the molten state. The presence of Molybdenum is unique to these Fe-based alloys and plays a critical role in the phase nucleation cascade of re-heated amorphous alloys. Furthermore, Molybdenum rich second phase particles that form upon cooling slowly from the molten state offer tremendous reinforcement to the surrounding matrix and exhibit Vickers hardness in excess of 2500.

9:40 AM Invited
Microstructure, Mechanical Properties of Mg-Ni-Zn-Y Bulk Metallic Glass Matrix Composites: *Keqiang Qiu*[1]; Qingfeng Li[1]; Wenhui Jiang[2]; Yinglei Ren[1]; Xiaoguang Yuan[1]; [1]Shenyang University of Technology; [2]University of Tennessee

Mg-Ni-Zn-Y bulk metallic glass (BMG) matrix composites were prepared by copper mold casting method. The microstructure, the mechanical properties were investigated by SEM, mechanical tester with different diameters. Compared with Mg-Cu-Zn-Y BMG matrix composites, Mg-Ni-Zn-Y BMG composites exhibit high fracture strength and lower mass density. The reinforcement was characterized as Mg solid solution flake of less than 5μm in thickness. The microstructure and mechanical properties vary according to diameters of the samples. The high fracture strength and plastic strain are discussed in term of amorphous phase effect and ductility of Mg flakes.

10:00 AM
Stabilization of Clusters in Metallic Glass Forming Melts: *Xiufang Bian*[1]; [1]Shandong University

We propose a new concept, the stabilization of cluster in metallic liquids described by the parameter B = dr/dT, for the prediction of the glass-forming ability (GFA) before their quenching, from the viewpoint of classical homogeneous nucleation theory. Metallic liquids with low B value possess high GFA, i.e., strong stabilization of clusters benefits to glass formation. When the value of B is lower than 1, the metallic liquid can be prepared into a metallic glass. The results of sixteen kinds of metallic liquids investigated by a high temperature X-ray diffractometer demonstrates that the stabilization ability of clusters determines the GFA.

10:15 AM
Nanocrystallization of a Bulk Metallic Glass in the Zr-Al-CuNiCo System – Structure and Mechanical Properties: Rohit Satapathy[1]; Rainer Wunderlich[1]; *Arnaud Caron*[1]; Indranil Manna[1]; Hans-Jörg Fecht[1]; [1]Ulm University

A controlled two-phase mixture in terms of volume fraction and morphology of nanocrystalline and amorphous components offers the possibility of tailoring the ductility and mechanical strength of BMG forming complex alloys. This has been investigated in the Zr-Al-CuNiCo alloy system as a function of the Al-concentration varied between 7 and 20 at%. At the composition limit of glass formation different amorphous to crystalline phase ratios and geometries have been obtained by casting. Minor compositional variations result in a BMG exhibiting excellent glass forming ability and two widely separated crystallization peaks which allows a wide variation of the nanocrystalline/glassy microstructure. Specimen were investigated by XRD, SEM,atomic resolution TEM, AFAM and nanoindentation. First results indicate that the microhardness and Young's modulus measured by nanoindentation considerably increase as a function of the nanocrystalline volume fraction while the Poisson ratio of the glass shows an unexpected maximum at some intermediate composition.

10:30 AM
Interfacial Reactions and Growth Kinetics for Intermetallic Compound Layer between Molten In-Sn Solder and CuZr-Based BMG: Guofeng Ma[1]; *Zhuang-qi Hu*[1]; [1]Chinese Academy of Sciences, Institute of Metal Research

The growth kinetics of intermetallic compound layers formed between In-Sn alloy and Cu40Zr44Al8Ag8 BMG substrates by solid state isothermal aging were examined at temperatures between 343 and 473 K for 1-10 hours. In the solder joints between the In-Sn solder and Cu40Zr44Al8Ag8 BMG, the intermetallic compound layer was composed of Zr, Cu, Sn As a whole, because the values of the time exponent (n) are approximately 0.5, the layer growth of the intermetallic compound was mainly controlled by a diffusion-controlled mechanism over the temperature range studied. The intermetallic compound layer thickness reached 4um after 10 hours of aging at 393 K. The apparent activation energy for growth of total intermetallic compound layers was 98.35 kJ/mol.

10:45 AM Break

10:50 AM Invited
Transition Metal Microadditions and Glass Formation in Rapidly-Quenched $Al_{88}Y_7Fe_5$ Alloys: *Kenneth Kelton*[1]; Lydia Longstreth-Spoor[1]; Nicholas Mauro[1]; Karyn Spence[1]; [1]Washington University

Studies of the influence of 3d transition metal microadditions on glass formation in $Al_{88}Y_7Fe_5$ are reviewed. Rapidly-quenched ribbons of $Al_{88}Y_7Fe_5$ show glass-like x-ray and transmission-electron-microscopy (TEM) diffraction patterns, but do not show the expected nucleation and growth peak in isothermal differential scaning calorimetry (DSC) measurements of primary crystallization to α-Al. The transformation kinetics, measured by DSC, TEM and electrical resistivity, are analyzed assuming both diffusion-limited nucleation and growth, and particle coarsening. Microalloying with any of the 3d transition metal elements improves glass formation and stability in this alloy, but to different degrees; the most effective element is Cr. High-q x-ray diffraction studies and EXAFS measurements made on $Al_{88}Y_7Fe_5$ and $Al_{87.5}Y_5Ti_{0.5}$ show that the microaddition induces structural changes in the liquid/glass structure, raising the nucleation barrier for α-Al. Schematic TTT diagrams that reflect DSC measurements of the change in devitrification pathway with microalloying are presented.

11:10 AM Invited
Synthesis and Properties of New Cu-Zr-Based Glassy Alloys with Unusual Glass-Forming Ability: *Wei Zhang*[1]; Akihisa Inoue[1]; [1]Tohoku University

Addition of Al to Cu-Zr-Ag alloys increased the stabilization of supercooled liquid and the reduced glass transition temperature, leading to remarkable enhancement in the glass-forming ability (GFA). The quaternary glassy alloy samples of over 15.0 mm in diameter are formed in the composition range of 35 to 45 at% Cu, 40 to 50 at% Zr, 5 to 10 at% Ag, and 5 to 8 at% Al. The best GFA is recognized for Cu36Zr48Ag8Al8 alloy which is close a eutectic composition in the quaternary alloy system. The glassy alloy samples were successfully fabricated in the diameter range up to 25 mm by an injection copper mold casting method. The addition of a small amount of Pd to the quaternary alloy is effective for the further increase in GFA. The glassy sample diameter of over 30 mm was fabricated. The bulk glassy alloys exhibited excellent mechanical properties and high corrosion resistances.

11:30 AM
Structural Changes in Bulk Metallic Glasses Induced by Compression: *Wojciech Dmowski*[1]; Takeshi Egami[1]; Yoshihiko Yokoyama[2]; Akihisa Inoue[2]; Bogdan Palosz[3]; Yang Ren[4]; [1]University Tennessee/MSE; [2]Tohoku University; [3]Polish Academy of Science; [4]Argonne National Laboratory

Mechanical properties of metallic glasses have been extensively studied, however much less is known about the underlaying changes in the atomic structure. There is a general consensus that deformation must be accompanied by a local rearrangement of atoms to accommodate shear strain. However, disordered nature of a glass or small deformation volumes makes it difficult to observe experimentally. We have investigated changes in the atomic structure in a bulk metallic glass of $Zr_{50}Cu_{40-x}Pd_xAl_{10}$ (x=0,6,10)that have been compressed in an isostatic and inhomogeneous way with pressures up to 8 GPa and at temperatures up to 800 K. We used high energy X-ray scattering and area detector to obtain atomic pair density function. The results indicate that inhomogeneous deformation in a glass involves rearrangement in a cluster of atoms (like bond exchange) that leads to structural anisotropy. However, isostatic compression results in isotropic changes in the local atomic structure.

11:45 AM
Microstructure and Mechanical Properties of Stainless-Steel-Continuous-Fiber-Reinforced Zr-Based Amorphous Alloy Composites Fabricated by Liquid Pressing Process: *Kyuhong Lee*[1]; Sang-bok Lee[2]; Sang-kwan Lee[2]; Sunghak Lee[1]; Nack J. Kim[1]; [1]Pohang University of Science and Technology; [2]Korea Institute of Machinery and Materials

Stainless-steel-continuous-fiber-reinforced Zr-based amorphous alloy composites were fabricated without pores and misinfiltration by liquid pressing process, and their microstructures and mechanical properties were investigated in this study. About 60 vol.% of continuous fibers were homogeneously distributed in the matrix, in which considerable amounts of polygonal crystalline particles and dendritic crystalline phases were formed by the diffusion of metallic elements from fibers to the matrix. In compressive test, strength of 700~830 MPa was maintained until reaching strain of 40% since fibers interrupted the propagation of shear bands initiated at the matrix and took over a considerable amount of loads. Under tensile loading, deformation and fracture occurred by consequent processes of crack formation at matrices, necking of fibers, fiber/matrix interfacial separation, and cup-and-cone-type fracture of fibers, thereby resulting in high tensile elongation over 6%. These findings suggested that new possibilities might be open to be applied to structural materials requiring excellent properties.

12:00 PM
Effect of Mo Addition to $Fe_{65}Cr_{15}C_{14}B_6$ Alloy during Mechanical: *Uma Maheswara Seelam*[1]; C. Suryanarayana[1]; [1]University of Central Florida

In this investigation, the effect of addition of Molybdenum to $Fe_{65-x}Cr_{15}Mo_XC_{14}B_6$ (X = Mo = 0 to 20 at.%) was studied on the ease of formation of an amorphous phase by MA. The milled powders were characterized using XRD, DSC and TEM methods. The alloy with 15 at.% Mo produced an amorphous phase after 30 h of milling. High temperature heat treatment (600-900°C) led to crystallization of the amorphous alloy. The type of crystallization products depended up on Mo content in the alloys. A carbide phase formed if the Mo content was less than 15 at.%. On the other hand, Fe solid solution and Fe/Mo borides formed if the Mo content was above 15 at.%. Thus, the type of phases present seems to be important in deciding whether amorphous alloys can be synthesized by MA.

12:15 PM
Thermodynamic Evaluations of Intermetallic Metallic Glass Systems Utilizing Interatomic Bond Energies: *James Dahlman*[1]; [1]Air Force Research Laboratory/MLLMD‘

Experimental evidence has demonstrated the tendency for metallic glass systems to form structures with a high degree of short range order. Ordering amongst nearest neighbors indicates the presence of underlying thermodynamic criteria as interatomic bond strength largely governs localized interactions. Previous work has corroborated the assumption thermodynamic parameters influence glass stability by relating critical thickness to glass transition temperature, heat of formation and critical cooling rates. Additionally, it is understood that strong A-B bond energies in binary systems result in a high degree of short range order. However, previous work has failed to show a correlation between nearest neighbor bond energies and glass forming ability. This work seeks to establish whether a connection between nearest neighbor bond energy and structural stability is possible through evaluation of bond energies and the heat of formation for multiple intermetallic glass forming systems.

12:30 PM Invited
Rare-Earth Based Bulk Metallic Glasses with High Thermal Stability and Excellent Magnetocaloric Properties: *Xudong Hui*[1]; Liang Liang[1]; G. L. Chen[1]; [1]University of Science and Technology Beijing

Several heavy rare-earth Gd-, Dy- and Er-based bulk metallic glasses (BMG) with high thermal stability and excellent magnetocaloric properties has been prepared by a copper mold casting. Compared with the other known rare-earth based BMGs, this BMG alloy possess higher glass transition temperature and crystallization temperature, larger effective activation energy for glass transition and crystallization. Under a modest magnetic field, this BMG alloy exhibits a comparable or even larger magnetocaloric effect than that the previously

reported Re-based BMGs and crystalline compounds. The high thermal stability and the excellent magnetocaloric effect together with other merits of the BMGs make this BMG alloy suitable candidate for use as magnetic refrigerant in a temperature range below 100 K.

Carbon Dioxide Reduction Metallurgy: Electrolytic Methods

Sponsored by: National Materials Advisory Board, Metallurgical Society of the Canadian Institute of Mining Metallurgy and Petroleum, The Minerals, Metals and Materials Society, American Iron and Steel Institute, TMS Light Metals Division, TMS Extraction and Processing Division, TMS: Reactive Metals Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Neale Neelameggham, US Magnesium LLC; Masao Suzuki, Al Tech Associates; Ramana Reddy, University of Alabama

Wednesday AM Room: 294
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Donald Sadoway, Massachusetts Institute of Technology; John Hryn, Praxair Inc

8:30 AM
Carbon-Free Metals Extraction by Molten Oxide Electrolysis: *Donald Sadoway*[1]; [1]Massachusetts Institute of Technology

Molten oxide electrolysis (MOE), which is the electrolytic decomposition of molten metal-oxide into liquid metal and oxygen gas, represents an improvement over today's carbon-intensive thermochemical reduction processes for metal production. For MOE the feedstock can be concentrate derived from ore or from hazardous waste such as chromate sludge. The process avoids the use of consumable carbon anodes and thus eliminates greenhouse-gas emissions as the by-product of the metal-recovery step. The concept applies to a variety of chemistries including titanium, iron, and ferroalloys. The electrochemistry of multicomponent oxide melts from the family FeO - SiO_2 - TiO_2 - Al_2O_3 - MgO - CaO has been studied by voltammetry at temperatures up to 1750°C. Galvanostatic electrolysis in laboratory-scale cells operating with a variety of feedstocks has demonstrated the extraction of liquid iron, ferrosilicon, and titanium with the simultaneous co-generation of oxygen.

8:50 AM
Electrochemical Reduction of Carbon Dioxide: *Melvin Miles*[1]; [1]University of La Verne

Considerable research has been conducted on the anodic oxidation of methanol in fuel cells. The electrochemical reduction of carbon dioxide in aqueous solutions to form methanol is the same reaction in reverse, i.e. CO_2 + 6 H+ + 6 e— CH_3OH + H_2O. The same catalysts and conditions may operate for both reaction directions. This carbon dioxide reduction reaction, however, must be able to compete with the electrochemical reduction of water to form hydrogen gas. Thermodynamically, the reduction of CO_2 to form CH_3OH is slightly more favorable than the reduction of water. Kinetically, carbon dioxide reduction can be favored by electrodes that are poor catalysts for water reduction such as Mo, Cu, In, Sn, and Sb. The use of nearly neutral electrolytes rather than acidic electrolytes makes the reduction of CO_2 kinetically more favorable. The thermodynamics of various reaction steps will be presented.

9:10 AM
Soda Fuel Cycle Metallurgy – Choices for CO_2 Reduction: *Neale Neelameggham*[1]; [1]US Magnesium LLC

Ferrous and non-ferrous metal industries are used to handling several billion tonnes a year of ore, fuel and fluxing agents by mineral processing techniques and varieties of extractive metallurgy "Reduction" processes. Metal industry know-how can be applied to meet the challenge of Reducing CO_2 to below 550 ppm in the atmosphere by the year 2050. Processes for generating alloys of carbon and hydrogen by Reducing the CO_2 and water mixture Soda, as in Carbonated water, to easily stored fuels are normally endothermic and are similar to generation of difficult to store hydrogen. This recycling of CO_2 and water are possible by using other forms of energy by innovative carbon capture methods. Thermodynamic diagrams for metal-soda [double oxide] reactions provide hints for using low cost unused stored energy in un-recycled metallic waste for thermal conversion. Choices for electrodes for electrometallurgical approaches [using solar - wind - hydro power] are also discussed.

9:30 AM
Electrocatalytic Reduction of CO_2 to C_2H_4 at a Gas/Solution/Metal Interface: *Kotaro Ogura*[1]; [1]Yamaguchi University

The problem of greenhouse effect should be largely mitigated if CO_2 can be recycled for use as fuels or chemicals. Although various processes for converting CO_2 to valuable compounds have been proposed, a matter of infinite importance is that the conversion process is feasible under mild conditions. This is because the secondary generation of CO_2 is feared if high energy is required and supplied with a fossil fuel. We have developed the electrochemical reduction process of CO_2 to C_2H_4 which occurs at a three-phase(gas/solution/metal)interface in concentrated potassium halide solutions. The input energy required for this process was supplied by a solar cell or a commercial power source. In the closed circulation system, the conversion efficiency of CO_2 increased with an increase of input electric charge to reach 100%, and the maximum selectivity for the formation of C_2H_4 was more than 80%.

9:50 AM Break

10:05 AM
CO_2 Free Electrochemical Process for Production of Light Metals Using Ionic Liquids as Electrolytes: *Mingming Zhang*[1]; Ramana Reddy[2]; [1]University of Alabama; [2]University of Alabama

Carbon dioxide (CO_2) is one of the most common by-products produced during light metals production. It is considered to be the cause of greenhouse effects. Efficient reduction of CO_2 can help to minimize the rate of global warming and improving energy sufficiency. In this paper, a novel light metals production process was proposed using ionic liquid electrolytes. This process has significant advantages compared with traditional high temperature processes such as zero CO_2 and CF_4 emission, low energy consumption due to the low temperature operation and non-consumable electrodes. Two types of anode materials (graphite, extruded carbon) were assessed in electrolysis experiment. The results showed that both anode materials were stable (negligible weight loss). However, the extrude carbon was found to be superior to graphite in terms of current density and current efficiency. The calculated energy consumption for aluminum production was less than 5kWh/kg at current densities up to 350 A/m^2.

10:25 AM
Carbon Dioxide Recycling by High Temperature Co-Electrolysis and Hydrocarbon Synthesis: *Joseph Hartvigsen*[1]; S. Elangovan[1]; Lyman Frost[1]; Anthony Nickens[1]; Carl Stoots[2]; James O'Brien[2]; J. Herring[2]; [1]Ceramatec, Inc.; [2]Idaho National Laboratory

Carbon dioxide recovered from concentrated sources, such as metallurgical reduction furnaces, cement kilns and fossil power plants can provide a valuable feedstock for a synthetic fuels industry based on energy from nuclear, solar, wind and hydropower sources. High temperature co-electrolysis of CO_2 and steam to produce synthesis gas has been demonstrated using reversible solid oxide fuel cell technology. Where a source of high temperature process heat is available, such as from an advanced high temperature nuclear reactor or solar dish concentrator, the endothermic electrolysis reactions can utilize both thermal and electrical inputs in such a way that the conversion efficiency within the cell is 100%. Synthesis gas produced in a co-electrolysis cell has been further reacted over a catalyst to produce conventional hydrocarbon fuels. Widespread implementation of synfuel production from CO_2 will enable a much larger reliance on intermittent renewable energy than can be accommodated by conventional electric demand profiles.

10:45 AM
CO_2 Reduction by Nanoscale Galvanic Couples: *Kanchan Mondal*[1]; [1]Southern Illinois University

Methanol, lower hydrocarbons, CO, and HCOOH formation have been by the reduction of CO_2 with H_2. Uncatalyzed electroreduction requires a significant overvoltage. An alternate route has been conceptualized. Production of hydrogen from water can be achieved by chemical oxidation of a metal in water followed

by oxidation of the hydronium ion by the released electrons to form hydrogen radical. It is hypothesized that circumventing the problem of the formation of hydrogen molecule is essential while having the hydrogen radical to react with CO_2 forming methanol, etc. This may be possible by a bimetallic catalyst acting as a galvanic couple, wherein one metal acts as the electron donor for the production of hydrogen radical while the other acts as a catalyst for the reduction of CO_2. This may enhance the reaction rates. The current concept circumvents the need for an external electric field unlike the electrochemical process.

11:05 AM Panel Discussion

11:20 AM Concluding Comments

Cast Shop Technology: Foundry Ingots and Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Hussain AlAli, GM Casthouse and Engineering Services, Aluminium Bahrain Company (ALBA); David DeYoung, Alcoa Inc

Wednesday AM Room: 295
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Graham Banister, Bahrain Alloys Manufacturing Company

8:30 AM Keynote
Chain Conveyor Remelt Ingot Casting Improvement: *John Grandfield*[1]; Vu Nguyen[1]; Pat Rohan[1]; [1]CSIRO

Operators of open mould chain conveyor remelt ingot casting machines are interested in reducing the oxide levels in the ingots and reducing production costs by improving the productivity of the machines. CAST has developed two patented technologies for ingot casting; a low dross filling system (CASTfill) and a mould design (CASTmould) with reduced ingot solidification time to achieve 20% higher production rates. This paper describes the development including application of Smooth Particle Hydrodynamic (SPH) modeling, protype design testing on a full scale pilot system, fully coupled heat and stress modelling and a purpose built rig for measuring mould temperatures, air gaps and displacement. New mould designs were proposed and tested on the rig. Following successful plant testing both CASTfill and CAST mould have been adopted at a number of smelters.

9:00 AM
Quality and Productivity Improvements in the Manufacture of Foundry (A356.2) Alloy Ingots at Aluminium Bahrain: Michael Jacobs[1]; Anand Sandhanam[1]; *Ebrahim Fathi*[1]; Aref Noor[1]; [1]Aluminium Bahrain

Aluminium Bahrain (Alba) produces 80,000 MT/annum of high quality re-melt ingots for the aluminium foundry industry. This paper discusses the various technologies and practices implemented at Alba's new production facility to improve the productivity and quality of the alloys produced in order to meet the increasing quality demands of the customers. These improvements are related to pot room metal treatment, closed door furnace operation, an electromagnetic stirring system, in line degassing, filtration and rod feeder systems, ingot chain casting wheel and commissioning of a new Properzi bar casting machine in order to achieve consistently high quality product at the maximum productivity are discussed.

9:25 AM
The Achievement of a Robust Al-Si Alloy Casting Process: Paul Cooper[1]; *Arne Dahle*[2]; [1]London and Scandinavian Metallurgical Company Ltd.; [2]University of Queensland

The Al-Si alloy casting process has suffered a long-standing reputation for lack of robustness when compared to wrought alloy casting. Both industries are able to benefit from the addition of effective master alloys for the purposes of grain refinement and structural modification. However the Al-Si alloy casting process has inherent disadvantages in terms of the achievement of a sound low porosity, high mechanical property casting. Recent advances in the technology of master alloy additives for Al-Si alloys are allowing this disadvantage to be effectively addressed. These advances are described and the potential for improved Al-Si alloy castings are explored.

9:50 AM Break

10:00 AM
Microstructural Improvement of Semi Solid Hypoeutectic Al-Si Foundry Alloys through Boron Addition: *Shahrooz Nafisi*[1]; H. Vali[2]; Jerzy Szpunar[3]; Reza Ghomashchi[4]; [1]ISPCO Inc.; [2]McGill University, Facility for Electron Microscopy Research; [3]McGill University, Department of Metals and Materials Engineering; [4]Advanced Materials and Processing Research Institute (AMPRI)

Comprehensive study was performed on the impact of grain refiners addition to the microstructural evolution of semi-solid hypoeutectic Al-Si slurries. The most widely used industrial refiners are AlTiB master alloys in spite of their well proven drawbacks including fading and settling of the so-called nucleant particles. Al-B master alloy is re-introduced as a potent substitute of traditional AlTiB master alloys. In semi solid casting of Al7Si0.35Mg alloy, it is shown that by boron addition, the number of primary Al-globules is increased. Improved globularity coupled with smaller primary particle size leads to superior rheological characteristics of the SSM billets.

10:25 AM
Characterization of Zn-Al Alloys with Different Structures Using Solidification and Electrochemical Parameters: Alicia Ares[1]; Liliana Gassa[2]; *Carlos Schvezov*[1]; [1]CONICET/University De Misiones; [2]CONICET/INIFTA

The objective of the present research consist on studying the type of structure (columnar, equiaxial or with the columnar to equiaxed transition, CET) using solidification and electrochemical parameters in Zn-Al alloys with different concentrations (Zn-2%Al, Zn-4wt%Al, Zn-16wt%Al, Zn-27wt%Al and Zn-50%Al, weight percent). In order to obtain columnar, equiaxial and the CET structures, the alloys were directionally solidified upwards in an experimental set up with a set of thermocouples in the samples which permit to determine the time dependent profiles during growth. From these profiles and the location of thermocouples it was possible to calculate the cooling rates, growth velocities and the temperature gradients along the samples. The electrochemical studies in the samples were realized by electrochemical impedance spectroscopy and polarization curves.

10:50 AM
Study of AlTiBC Master Alloy: *Xiong Guangcheng*[1]; [1]Aida Aluminium Alloys Company, Ltd

The AlTiBC grain refining master alloy has been discussed and researched for years by some researchers, but now it's still in argument. So, by cooperating with Shandong University, Aida make a study of this master alloy. Experimental results showed that the AlTi5C0.4 master alloy which was after melting again exhibited better grain refining performance for commercially pure Al than the ones that had not been remelted. As a result, it was discussed a new preparation craft of AlTiBC master alloy in this paper in detail. And the grain refining potentiality of the AlTiBC master alloy was investigated in this article by optical microscopy and electron probe micro-analyzer (EPMA).

11:15 AM Panel Discussion

Characterization of Minerals, Metals, and Materials: Characterization of Microstructure and Properties of Materials III

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Jian Li, Natural Resources Canada; Toru Okabe, University of Tokyo; Ann Hagni, Intellection Corporation

Wednesday AM Room: 284
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Arun Gokhale, Georgia Institute of Technology; Mingdong Cai, University of Houston

8:30 AM
Cathodic Polarization Behavior of Ionic Liquid (TMHA-Tf_2N) Containing Titanium Ions: *Kazuaki Tsuchimoto*[1]; Tetsuya Uda[1]; Kuniaki Murase[1]; Yoshitaro Nose[1]; Yasuhiro Awakura[1]; [1]Kyoto University, Department of Materials Science and Engineering

To develop novel technology for the electroplating of titanium layers from ionic liquid (room temperature molten salt) baths at low temperatures, cathodic polarization behavior of trimethyl-*n*-hexylammonium bis[(trifluoromethyl)sulfonyl]amide (TMHA-Tf_2N) ionic liquid containing titanium ions was investigated at 50°C. Titanium electrode was dissolved anodically to feed titanium ions into the ionic liquid, and cyclic voltammograms were taken for the resulting baths. When a single-compartment electrolytic cell was used, two reduction waves were found at -1.0 V and -2.5 V. In contract, an additional reduction wave appeared at 0 V with a two-compartment cell where anolyte and catholyte were separated by a sintered glass. It is considered that, in the single-compartment bath, Ti^{4+} ions generated through the anodic dissolution can migrate to cathode and reduced to a stable Ti^{3+} state, hence we can conclude here that the reduction at 0 V should be attributed to reduction of Ti^{4+} to Ti^{3+}.

8:50 AM
Corrosion of Nickel-Based Superalloy in Molten Fluoride: Bowen Li[1]; Xiaodi Huang[1]; *Jiann-Yang Hwang*[1]; [1]Michigan Technological University

Nickel-based alloys have been used in a wide variety of severe operating conditions, including corrosive environment, high temperature, high stress, and their combinations. To determine the feasibility of using nickel-based superalloy as an electrode material in primary aluminum production, the corrosion rate of a nickel-based superalloy from INCO was investigated in an industrial fluoride melt using the metallographic method. The results show that fluoride melt has high wettability to the alloy. When corrosion time was less than 0.5 hours at 1000°C, the microstructure of the specimen surface had no obvious change. The maximum corrosion rate of the fluoride solution to the superalloy is approximately 27 microns per hour.

9:10 AM
Comparison between Aluminium 6063 Hardened Thermochemical and Mecanochemically: *Isaías Hilerio*[1]; Miguel Barrón[1]; [1]Universidad Autonoma Metropolitana Azcapotzalco

The objective of this work is to compare the properties of a material treated thermochemically in relation to mecano-chemical treatment. The advantage of this last treatment is not to add a material to the substratum, because the hardened material with the mecanochemical treatment has not the adherence problem by hardened layer using thermochemical treatment. The process was carried out in 5 samples with the following dimensions: 35 x 100 x 8 mm. It has been utilized a vibrator machine with a different abrasive charge in each stage of the process. To study the kinetics of the process was withdrawn a sample for each stage of the process. It has been done the micro hardness study, MEB and Ray X diffraction. It has been founded higher hardness values in the material treated thermochemically, but the difference is in the cases which it is not possible to use that method.

9:30 AM
Thermally Activated Sintering of Kaolinitic Clay: *Sergio Monteiro*[1]; Carlos Maurício Vieira[1]; [1]State University of the Northern Rio de Janeiro - UENF

The sintering of kaolinitic clay ceramics can be achieved by solid state diffusion at temperatures below 900°C. Owing to the plate-shaped morphology of the kaolinitic clay particles, surface diffusion can consolidate the porosity by thermally activated mechanisms at temperatures as low as 500°C. In the present work, the thermally activated parameters for the sintering of a kaolinitic clay were characterized in the interval from 500 to 900°C. The mechanical strength obtained in cylindrical specimens, that were fired in this interval of temperatures for different times and then tested by diametral compression, serve to evaluate the thermally activated parameters. The results showed a relatively smaller value for the activation energy implying a more efficient mechanism of porosity consolidation, which justifies, in practice, the low temperature sintering of kaolinitic clay ceramic.

9:50 AM Break

10:10 AM
Investigations of Retained Deformation Energy after Hot Forming and Its Influence on the Abnormal Grain Growth for the Nickel Based Alloy 80a: *Mirza Candic*[1]; Stefan Mitsche[2]; Baohui Tian[3]; Christof Sommitsch[1]; [1]University of Leoben; [2]University of Graz; [3]Bohler Edelstahl GmbH

For a hot forming process, residual (i.e. retained) strains exist due to different degrees of recovery and recrystallization, which can be related to inhomogeneous deformation, strain rate and temperature gradients with deformation. During the subsequent annealing the retained strain may lead to non-uniformed grain structures, or even worse, coarse grains due to secondary recrystallization and abnormal grain growth, respectively. In the present work, for the nickel based alloy 80a, single and multi hit hot deformation tests followed by annealing were performed for variable deformation and annealing parameters. In order to evaluate the retained strain evolution, the strain distribution of related grain structures was investigated by using the finite element method as well as corresponding microhardness values and EBSD data. For the quantitative analysis of the local grain misorientation some new evaluation methods were developed.

10:30 AM
Fundamental Study on Titanium Production Process by Disproportionation Reactions of $TiCl_2$ in $MgCl_2$ Molten Salt: *Taiji Oi*[1]; Toru Okabe[1]; [1]University of Tokyo

In order to establish a novel titanium production process, the synthesis and disproportionation reactions of $TiCl_2$ in molten salt were investigated. This process comprised (1) the synthesis of $TiCl_2$ by reacting $TiCl_4$ with titanium metal in $MgCl_2$ molten salt at 1073-1273 K and (2) the formation of titanium by the disproportionation reactions of $TiCl_2$ in molten $MgCl_2$ at 1300-1373 K. The results revealed that $TiCl_2$ was successfully obtained in the synthesis step and the efficiency of $TiCl_2$ formation was drastically improved by using molten $MgCl_2$ as the reaction medium. Some preliminary experiments demonstrated that titanium powder of approximately 99% purity was produced by the disproportionation reactions of $TiCl_2$ in $MgCl_2$ molten salt. The titanium production methods investigated in this study can be applied to a new titanium production process and/or titanium plating technology.

10:50 AM
Influence of the Precursors Setup on the Yield and Properties of HPHT Synthesized Diamonds: *Ana Lucia Skury*[1]; [1]Universidade Estadual do Norte Fluminense

The production yield of diamonds synthesized at HPHT conditions from graphite and catalyst metallic alloy precursors depends on parametric variables, which include the initial setup of the precursor materials. This setup is associated with the arrangement of both, the graphite and the catalyst alloy, in either homogeneous mixture or alternated layers. The present work studied the influence of the type of setup on the yield and properties of HPHT synthesized diamond crystals. Precursor reactive system composed of graphite and Ni-Mn alloy, in homogeneous mixture or layer, was processed at 4.7GPa and 1300°C for 10 minutes. It was found that the yield is sensibly affected by the type of setup. A 10% decrease in productivity occurs for the alternated layers setup, while the brittleness of the diamond, measured by friar tests, increases for the homogeneous mixture setup. Moreover, significant differences were observed in the shape and size distribution of the crystal.

Complex Oxide Materials - Synthesis, Properties and Applications: Scaling, Dynamics, and Switching

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division
Program Organizers: Ho Nyung Lee, Oak Ridge National Laboratory; Zhiming Wang, University of Arkansas

Wednesday AM Room: 277
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Gyula Eres, Oak Ridge National Laboratory; Guus Rijnders, University of Twente

8:30 AM Invited
Atomic-Scale Characterization of Interfaces in Oxide Heterostructures: *Matthew Chisholm*[1]; Ho Nyung Lee[1]; Maria Varela del Arco[1]; Stephen Pennycook[1]; [1]Oak Ridge National Laboratory

Recent advances in materials synthesis, theoretical simulation and atomic scale imaging/spectroscopy is being used to design, create and characterize three-dimensional materials with functional physical properties resulting from electronic effects of two-dimensional interfaces. It is expected that correlating macroscopic properties of these new "interfacial solids" with atomic structures will lay the foundation for the ultimate miniaturization of devices. We will discuss some of our results on interface-induced, two-dimensional electrical conduction in thin film oxide superlattices. Macroscopic physical properties of these artificial solids will be correlated with results from ORNL's aberration-corrected scanning transmission electron microscopes, which provide the smallest electron probes. These instruments allow simultaneous sub-Å resolution imaging and electron energy-loss spectroscopy, which is currently the only means for obtaining a highly local measure of the chemical, crystallographic and electronic properties of materials. Research sponsored by the Office of Basic Energy Sciences, Division of Materials Sciences and Engineering.

9:00 AM Invited
Influence of Defects on the Dielectric and Transport Properties of $SrTiO_3$ Thin Films: *Susanne Stemmer*[1]; Junwoo Son[1]; Nicholas Finstrom[1]; [1]University of California Santa Barbara

In this presentation we report on the key properties of complex oxide thin films relevant for their application in metal/insulator/metal structures such as voltage tunable capacitors, and the relation of these properties to defects in the films. We study the dielectric and electrical properties of $Pt/SrTiO_3/Pt$ thin film structures with controlled microstructures at frequencies up to 1 GHz. We discuss the origins of dielectric losses in these films and the role of point defects, such as oxygen vacancies. We report on dielectric deadlayers, which cause a significant reduction in the dielectric tunabilities. We discuss the influence of dielectric relaxation on the thermal leakage characteristics of $Pt/SrTiO_3/Pt$ thin film structures. We show the influence of temperature and time-dependent leakage currents on phenomena such as anomalous positive temperature coefficient of resistivity and resistive switching.

9:30 AM Invited
Towards Defect Engineering in Resistive Switching Perovskite Thin Films: *Regina Dittmann*[1]; Kristov Szot[1]; Ruth Münstermann[1]; Paul Meuffels[1]; Shaobo Mi[1]; Chunlin Jia[1]; Rainer Waser[1]; [1]Forschungszentrum Juelich

A variety of perovskite materials exhibit electrically induced resistive switching effects and has been proposed for future non-volatile memories. For $SrTiO_3$ single crystals it was shown that switching occurs along crystalline defects which form a network of filamentary conduction paths (Szot et al., Nature Mater. 5, 312 (2006)). Since the distribution of defects in $SrTiO_3$ is generally very irregular, the application of this material class in future resistive RAM crucially depends on the success in developing thin film fabrication routes which guarantee a more registered arrangement of these filaments. We will present the impact of growth mode conditions and the corresponding defect distribution as well as the influence of doping atoms on the nanoscale resistive switching properties of titanate thin films analyzed by conductive-tip AFM (LC-AFM. For Nb-doped $SrTiO_3$ thin films epitaxially grown under appropriate deposition conditions, we succeeded to obtain well ordered arrays of nanoscale resitive switching blocks.

10:00 AM Break

10:20 AM Invited
Opportunities in Ferroelectrics and Multiferroics at Short Times and High Fields: *Paul Evans*[1]; [1]University of Wisconsin, Madison

Thin film structures with high-bandwidth electrical contacts make it possible to understand complex oxide materials at short timescales – in the range of hundreds of picoseconds at present and perhaps a few to tens of picoseconds in the near future. The magnitudes of electric fields applied under these conditions can reach several MV/cm, far higher than the fields associated with dielectric breakdown at low frequencies. Our time-resolved synchrotron microdiffraction structural studies of ferroelectric $Pb(Zr,Ti)O_3$ thin provide insight into polarization domain dynamics under electric fields close to the coercive field and into piezoelectricity at higher fields. Thin films of multiferroic $BiFeO_3$ has a more complicated symmetry than the tetragonal $Pb(Zr,Ti)O_3$ and structurally non-uniform at several length scales. We will describe structural studies of $BiFeO_3$ under high fields and switching between different polarization domain configurations.

10:50 AM Invited
The Mechanism of Pulsed Laser Deposition of Epitaxial Oxide Films Studied by Time-Resolved Surface X-Ray Diffraction: *Gyula Eres*[1]; [1]Oak Ridge National Laboratory

The laser plume ejected from a solid target consists of a complex mixture of neutral and ionized atoms, molecular fragments, and even small clusters with kinetic energies up to a few hundred eV. The extra kinetic energy provides a transient enhancement of surface mobility that affects the nucleation and growth of thin films. We use time-resolved surface x-ray diffraction to study the formation and evolution of surface structure during PLD of $SrTiO_3$. The use of x-ray diffraction greatly simplifies growth kinetics studies because in the kinematic limit the x-ray intensity changes correspond directly to coverage evolution. We analyze the intensity transients using a model independent approach that allows direct determination of the time-dependent surface coverages from the transient intensities. This analysis reveals that energy-enhanced interlayer transport occurs on a time scale of microseconds or less and it dominates crystallization and growth with the thermal component playing a negligible role.

11:20 AM Invited
Dynamics of Strained Film Growth on Vicinal Substrates: *Mina Yoon*[1]; [1]University of Tennessee

We theoretically study the morphological evolution in heteroepitaxial growth. We establish the existence of a deposition flux window within which stable and persistent step-flow growth can be achieved. We express the phase boundaries in terms of intrinsic physical parameters and experimentally controllable growth conditions.[1] Also, investigating the growth dynamics in the step-bunching regime, we found that there is a critical film thickness above which step-bunching occurs. The critical thickness shows a scaling behavior depending on the terrace width and the deposition flux.[2] We compare above predictions with experimental results from PLD growth studies of metal oxide thin films.[1,2] Our results may open a way to grow films in a desired way. We will also discuss the role of step meandering in the above findings. [1]W. Hong et al., Phys. Rev. Lett. 95, 095501 (2005). [2]M. Yoon et al., Phys. Rev. Lett. (2007).

11:50 AM
Domains Evolution under a Spatially Non-Uniform Applied Electric Potential: Phase-Field Simulation and Experiment: *Samrat Choudhury*[1]; Jingxian Zhang[1]; Sergei Kalinin[2]; Long Chen[1]; [1]Pennsylvania State University; [2]Oak Ridge National Laboratory

A three-dimensional (3-D) phase-field model is developed for predicting the evolution of domain structures in ferroelectric thin films under a non-uniform externally applied electric potential. In this work, we considered lead zirconate titanate (PbZr(1-x)TixO3) ferroelectric thin films as model systems. The temporal evolution of domain structures during switching will be presented. It was observed that the electric potential required to nucleate new domains during polarization switching in ferroelectric thin films varies spatially within a domain structure. The lowest electric field for nucleation is observed near the

twin domain boundaries. Comparison of the spatial distribution of nucleation voltage obtained from phase-field approach shows an excellent agreement with our experimental measurements using Piezo-force microscopy technique. Preliminary simulation and experimental results on the effect of grain boundary on switching behavior of ferroelectric thin films under a non-uniform applied electric potential will be discussed.

12:10 PM
Phase-Field Prediction of Enhanced Piezoelectric Response in Ferroelectric Islands: *Jingxian Zhang*[1]; Ryan Wu[1]; Samrat Choudhury[1]; Shenyang Hu[2]; Yulan Li[1]; Long-Qing Chen[1]; [1]Pennsylvania State University; [2]Los Alamos National Laboratory

In continuous ferroelectric thin films, the extrinsic contribution to piezoelectric response from the non-180° polarization switching is highly limited by the substrate constraint. Therefore, enhanced piezoelectric responses are expected by cutting the film into discrete islands to relieve the substrate clamp. In this work, a phase-field model has been developed for studying the domain structures and their evolutions in epitaxial ferroelectric islands. The piezoelectric properties of PbZr0.2Ti0.8O3 ferroelectric islands are investigated by using the phase-field model. Much larger piezoelectric coefficients are obtained for the islands due to the 90° polarization switching, and the magnitude of the piezoelectric coefficients depends on the substrate strains and the lateral sizes of the islands. These results clearly demonstrate that the relaxation of substrate constraint plays a critical role on the piezoelectric response in the ferroelectric islands.

Computational Thermodynamics and Kinetics: Phase Transformations

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Yunzhi Wang, Ohio State University; Long-Qing Chen, Pennsylvania State University; Jeffrey Hoyt, McMaster University; Yu Wang, Virginia Tech

Wednesday AM Room: 288
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Chen Shen, GE Global Research; Hamish Fraser, Ohio State University

8:30 AM Invited
On the Nucleation of Alpha-Ti in Alpha/Beta-Ti and Beta-Ti Alloys: Soumya Nag[1]; Arda Genc[1]; Rajarshi Banerjee[2]; Gopal Viswanathan[1]; *Hamish Fraser*[1]; [1]Ohio State University; [2]University of North Texas

This paper presents experimental observations that lead to mechanistic details of the nucleation processes in alpha/beta and beta Ti alloys. For alpha/beta Ti-alloys, the formation of Widmansaetten plates of the alpha phase from prior beta grain boundaries producing colony microstructures and the nucleation of the alpha plates that make up basketweave microstructures have been considered. Here, 3D characterization of the effects of grain boundary misorientation and grain boundary plane orientation on sideplate growth in has been studied, and observations have been made of the very early stages of nucleation in both types of microstructures. The nucleation of secondary alpha in beta ribs has been the subject of study, and the possible role of martensitic transformations in nucleation considered. Finally, the refined precipitation of alpha-Ti in beta stabilized alloys has been studied and the effects of beta phase separation and the omega phase on nucleation have been identified.

8:55 AM Invited
Physical Models for Heterogeneous Nucleation in Aluminium-Based Solid Solutions: *Barry Muddle*[1]; Yury Kryvasheyeu[2]; [1]Monash University, Centre for Design in Light Metals; [2]Monash University, School of Physics

Effective computational analysis of phase transformations requires that not only are the algorithms employed sound in thermodynamic and kinetic principles, but that the physical models of the processes involved be reliably based. In the case of precipitation from supersaturated solid solution, this includes both the mechanisms of nucleation and growth, and the nature of any interactions that influence, in particular, nucleation. In the specific case of precipitation-strengthened aluminium alloys, nucleation of key strengthening phases is invariably heterogeneous and preferred locations for nucleation may range from pre-existing static defects to sites defined dynamically during early stage decomposition. In most cases, the mechanisms by which such sites reduce the barrier to nucleation remain ill-defined and an inadequate physical basis limits the effectiveness of process models. The limitations of available physical models will be examined for a selection of the more common modes of heterogeneous nucleation in aluminium-based solid solutions as model systems.

9:20 AM
Phase Field Simulation of Autocatalytic Precipitation in the Al-Cu System: *Yury Kryvasheyeu*[1]; Barry Muddle[1]; Chen Shen[2]; Yunzhi Wang[2]; [1]Monash University; [2]Ohio State University

Phase transformations in coherent systems are strongly influenced by the matrix-product coherency strains, in some cases responsible for the formation of self-organized structures. In the case of θ' precipitation in Al-Cu, patterns of precipitates, formed in inclined parallel stacks or dual-variant cross-like arrays, are presumed to be the product of autocatalytic nucleation. It is suggested that elastic interaction promotes nucleation in specific locations within the strain field of a precursor crystal, leading to strong spatial correlation in the precipitate distribution. The model proposed is based on phase field evolution kinetics coupled with an explicit nucleation algorithm. Microstructural patterns formed during autocatalysis are compared for two forms of transformation strain – a pure uniaxial distortion and an invariant plane strain. The latter implies a diffusional-displacive mechanism of transformation and, aside from simulation of observed precipitate arrays, the model has the potential for verifying indirectly the nature of transformation strain during nucleation.

9:35 AM
A Comparison of Simulated Crystal Nucleation Times with Nucleation Theory for the Lennard-Jones Liquid: *Lujian Peng*[1]; Rachel Aga[2]; James Morris[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

In previous work, we showed that when simulation size effects and transient nucleation theory were considered, crystal nucleation times from simulations could be predicted using classical theory. We have now extended this to the Lennard-Jones system. Homogeneous crystal nucleation is simulated by molecular dynamics. We study nucleation time as a function of system size and temperature. We present quantitative comparisons between our results and classical nucleation theory, including transient effects. This is done without parameter fitting, using interfacial free energies and other properties calculated from separate equilibrium simulations. The role of temperature-dependent interfacial free energies is explored. We compare with previous results that showed good agreement with the classical theory. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under contract with DE-AC05-00OR-22725 with UT-Battelle.

9:50 AM Invited
Phase Field Modelling of Rafting in Ni Base Superalloys: Alphonse Finel[1]; *Yann Le Bouar*[2]; Anaïs Gaubert[1]; [1]ONERA; [2]CNRS

Mechanical behaviour of Ni base superalloys depends largely on the microstructure at meso scale. Initially, the alloy consists in cuboidal precipitates of the γ' phase separated by channels of γ matrix. When such a material is submitted to a creep loading at high temperature, we observe a directional coalescence of the γ' precipitates. This morphological evolution, usually called rafting, is a result of the elastic stresses generated by the misfit between the precipitate and matrix phases. In addition, during rafting, plastic strain is generated in the matrix and enables a partial relaxation of the misfit stresses. The aim of this study is to propose a phase field model to represent in a quantitative way the microstructural changes in superalloys during a creep loading. In the model, the plastic activity in the γ phase is introduced by the means of a viscoplastic law coupled with the phase field model.

10:15 AM
The Pre-Wetting Transition at Anti-Phase Boundaries: An Atomistic Modeling Study of Ni_3Al: *Chandler Becker*[1]; Yuri Mishin[2]; William Boettinger[1]; [1]National Institute of Standards and Technology; [2]George Mason University

Using an embedded-atom potential for Ni-Al alloys, we have examined interfacial properties of antiphase boundaries (APBs) in the Ni_3Al-based γ' phase. These interfaces between different ordering domains of the γ' phase can undergo a pre-wetting transition, in which the APB region becomes disordered and then transforms into a thin layer of metastable γ phase. To understand this transition, we have performed detailed thermodynamic, compositional, and structural analyses of the APBs using semi-grand canonical Monte-Carlo simulations, with particular interest in composition profiles of Ni segregation and local atomic disorder. We discuss our findings in the context of previous studies of these interfaces and the possible impact of the APB pre-wetting on properties of γ-γ' alloys.

10:30 AM Break

10:50 AM Invited
Determinisitic Sidebranching Mechanism in 3-D Dendritic Growth: *Martin Glicksman*[1]; John Lowengrub[2]; Shuwang Li[2]; [1]University of Florida; [2]University of California, at Irvine

We demonstrate the existence of a new mechanism for the generation of 3D side-branches in dendritic solidification. The proposed deterministic branching mechanism is induced directly via the interfacial boundary conditions, without benefit of noise or other stochastic phenomena. To support the new finding we developed accurate boundary integrals for simulating 3D solidification. Numerical results reveal that both anisotropic capillarity and kinetics induce non-monotone temperature distributions along the dendritic interface. The consequent dynamical process operates as a "limit cycle" generating a sequence of time-periodic protuberances near the dendrite tip. These protuberances grow and develop into secondary side-branches, setting the all-important scale of chemical microsegregation. Unlike conventional explanations of side branching, based on extrinsic influences, such as selective noise-amplification and loss of stability, we demonstrate that the generation of side-branches during dendritic growth is intrinsic to the solidification process, occurring via deterministic interactions at the solid-liquid interface.

11:15 AM
Orientation Selection in Dendritic Solidification of Materials with Hexagonal Symmetry: *Tomorr Haxhimali*[1]; Alain Karma[1]; Mark Asta[2]; Jeffrey Hoyt[3]; [1]Northeastern University; [2]University of California, Davis; [3]McMaster University

The prediction of dendrite growth rate and orientation during dendritic solidification is crucial to control the morphology of solidification microstructures and thus the associated mechanical properties of cast and welded materials. In this talk we present recent results from a theoretical and phase-field study of dendrite growth rate and orientation selected during the dendritic solidification of materials with hexagonal symmetry with magnesium as a special case. The phase-field and theoretical study were performed by varying parameters that characterize the anisotropy of the crystal-melt interfacial free-energy. For anisotropy parameters that were estimated from recent atomistic simulation for pure magnesium, we show that dendrite grow along the basal plane as expected. Moreover we predict the existence of anomalous directions off basal plane by varying the anisotropic parameter, which is equivalent to changes in composition. These directions have been often observed by experimental studies in different magnesium and hcp alloys.

11:30 AM
Microstructure Formation in Ni-Base Superalloys during Solidification and Its Relation to the Eutectic-Peritectic Nature of the Gamma/Gamma-Prime Reaction: *Nils Warnken*[1]; Roger Reed[1]; [1]University of Birmingham

The microstructure formation in directionally solidified Ni-base superalloys is simulated using the phase-field method (MICRESS) with a special emphasize on the last stages of solidification and the interdendritic gamma-prime formation. The latter is commonly referred to as eutectic islands, although recent research indicates that it should be renamed to peritectic islands. Thermodynamic databases model it either way. The calculated microstructures yielded from both types of databases are compared in order to identify whether the type of the reaction has a significant influence on the as-cast microstructure formed during directional solidification.

11:45 AM
Stress-Induced Instability and Break up in Multilayers: B. Chirranjeevi[1]; *Mogadalai Gururajan*[2]; Thennathur Abinandanan[3]; [1]Indian Institute of Technology-Madras; [2]Northwest University; [3]Indian Institute of Science

Evolution of multilayers of elastically stiff and soft phases has been studied using a phase field model. When the interfaces are coherent and the phases have a lattice parameter mismatch, the hard phase is known to be unstable. Our phase field simulations capture — quantitatively — the key features of this instability for a single, isolated stiff film sandwiched between two semi-infinite soft matrix substrates. In a multi-layer setting (i.e., with an increasing volume fraction of the stiff phase), several new features emerge: (a) the onset of instability changes from a predominantly anti-symmetric mode to a symmetric one; (b) the dominant wavelength increases, thereby delaying the eventual break-up; (c) inter-layer correlations develop right from early stages of evolution, leading to a highly correlated microstructure after the complete break-up. These features will be presented and rationalized.

12:00 PM
A New Approach for Modeling Strongly Anistropic Crystal Systems: *Solmaz Torabi*[1]; John Lowengrub[1]; Steven Wise[2]; [1]University of California-Irvine; [2]University of Tennessee

We present a new approach for modeling strongly anisotropic crystal and epitaxial growth using regularized, anisotropic Cahn-Hilliard-type equations. Such problems arise during the growth and coarsening of thin films. When the surface anisotropy is sufficiently strong, sharp corners form and unregularized anisotropic Cahn-Hilliard equations become ill-posed. Our models contain a high order Willmore regularization to remove the ill-posedness. A key feature of our approach is the development of a new formulation in which the interface thickness is independent of crystallographic orientation. We also have added strain energy to this model to study the effect of elasticity. We present 3D numerical results using an adaptive, nonlinear multigrid finite-difference method. In particular, we find excellent agreement between the computed equilibrium shapes using the Cahn-Hilliard approach, with a finite but small Willmore regularization, and an analytical sharp-interface theory recently developed by B. J. Spencer [2004].

12:15 PM
Monte Carlo Simulation of Binary bcc Structured Layers Growth: Ruiqiang Zhang[1]; *Xiaohong Xu*[1]; Shunyan Zhang[1]; Gillian Gehring[2]; [1]Shanxi Normal University; [2]University of Sheffield

A very promising method for thin (of nanoscale thickness) layer fabrication is the pulsed laser deposition (PLD) method. In this paper, the growth of perovskite structured film grow on a perovskite substrate is simulated using the Monte Carlo procedure, and $LaMnO_3$ is taken as an example. In the model, we consider the La and Mn atoms and assume the oxygen atoms follow the metal atoms. The quality of the film is controlled by three parameters, namely, the temperature of the substrate, the kinetic energy of the atoms and the average coverage of each pulse. The simulated results show that the quality of the films is strongly dependent on the three parameters. We analyzed the mismatch of the films and find an interesting phenomenon; the displacement parameter presents odd-even staggering (OES) when the deposition condition near the best situation.

Emerging Interconnect and Packaging Technologies: Pb-Free Solders: Reliability and Microstructure Development

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Electronic Packaging and Interconnection Materials Committee
Program Organizers: Carol Handwerker, Purdue University; Srinivas Chada, Medtronic; Fay Hua, Intel Corporation; Kejun Zeng, Texas Instruments, Inc.

Wednesday AM Room: 275
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Laura Turbini, Research In Motion; Eric Cotts, Binghamton University

8:30 AM Invited
Microstructure, Defects, and Reliability of Mixed Pb Free/SnPb Assemblies: *Polina Snugovsky*[1]; H. McCormick[1]; S. Bagheri[1]; Z. Bagheri[1]; C. Hamilton[1]; M. Romansky[1]; [1]Selestica

The paper describes the results of an intensive study on Pb free BGA / SnPb solder assemblies as well as some lessons learnt dealing with mixed assembly production at Celestica. In the reliability study, four types of Pb free ball grid array components were assembled on test vehicles using the SnPb eutectic solder and typical SnPb reflow profiles with 205-220°C peak temperatures. Accelerated thermal cycling was conducted at 0 to 100°C. The microstructure after reflow and ATC was analyzed. The influence of the microstructure on Weibull plot parameters and failure mode will be shown. Interconnect defects such as non-uniform phase distribution, low melting structure accumulation, and void formation will be discussed. Recommendations on mixed assembly and rework parameters will be given.

9:00 AM Invited
Thermomechanical Behavior and Reliability of Pb-Free Solders: *Nikhilesh Chawla*[1]; [1]Arizona State University

The thermal fatigue resistance of Sn-rich solder joints is dependent on the initial microstructure and the damage evolution during thermal cycling. In this talk we have report on the thermal fatigue behavior of single lap shear Sn-rich solder/Cu joints under cyclic thermal loading. Microstructure characterization of the initial and damaged solder joint microstructures was carried out by scanning electron microscopy (SEM). Persistent slip bands, was followed by crack nucleation and propagation at the tip of the lap shear geometry. The influence of Sn grain orientation on thermal fatigue damage was modeled using the finite element method (FEM). The grain orientation was determined by orientation image mapping (OIM) and a Schmid factor analysis was performed to determine the active slip systems in the Sn grains. It was found that the geometry of the lap shear joint, rather than Sn grain orientation, was more significant in determining the location of crack initiation.

9:30 AM Invited
Numerical and Experimental Analysis of Ball Grid Array Packages during Random Vibration: Fengjiang Wang[1]; *Matthew J. O'Keefe*[1]; Brandon Brinkmeyer[1]; [1]University of Missouri-Rolla

With the transition from Sn-Pb to Pb-free solder, board level vibration testing for high reliability applications, i.e. the aerospace industry, is needed. In particular, ball grid array (BGA) packages are susceptible to mechanical failure due to the stress/strain in the solder balls from the relative motion between the BGA and substrate. The location of components on the printed circuit board (PCB) impacts reliability as a function of component type and solder alloy composition. Currently, there is limited information on the dynamic response of PCBs and Pb-free solder joints that relates stress/strain to solder joint reliability under random vibration. In this work, finite element modeling was used to determine how size and location impacts Pb-free and Sn-Pb BGA packages on a PCB. Random vibration in the frequency range of 20-2000 Hz was simulated using different root mean square accelerations. Correlation of the modeling to experimental results will also be discussed.

10:00 AM Break

10:15 AM
Bending Reliability of Combination Solder Joint in Mounted on Several Types of Pad Finish of Halogen Free PCB: *Si-Suk Kim*[1]; Hun Han[1]; Hyun-Jeong Ham[1]; Yong-Hyun Kim[1]; Dong-Chun Lee[1]; Si-Don Choi[1]; [1]Samsung Electronics

According to the transition of pb-free and demand for halogen free based on environmentally friendly product, we are considering applying low temperature soldering which is a combination solder joint between Sn3.0Ag0.5Cu solder ball and Sn57Bi.1.0Ag solder paste into halogen free pcb. It has lower soldering temperature compared to Sn3.0Ag0.5Cu solder system. So we have studied on bending reliability about this system. We used several types of halogen free pcb finish and pad designs in 512MB memory module. We carried out 3 point bending test. After bending test, we analyzed fracture mode using EDX, SEM and D&P(Dye & Pry). Consequently, we figured out good reliability in order ENIG+NSMD>OSP+SMD=Immersion Ag+SMD>OSP+NSMD=Immersion Ag+NSMD > ENIG+SMD. Solder joint strength for OSP or Immersion-Ag pad finish is stronger than that of ENIG pad. In addition, Cu adhesion strength of halogen free pcb is lower than normal FR4 pcb, which leaded to low reliability life.

10:30 AM Invited
Effect of Crystal Orientations of Copper and Tin on Recrystallization during Thermomechanical Cycling of Pb-Free Solder Joints: Amir Zamiri[1]; *Thomas Bieler*[1]; Adwait Telang[1]; Farhang Pourboghrat[1]; [1]Michigan State University

It is known that many, and possibly most lead free solder joints consist of only a few crystal orientations. In many cases, sections of joints show only one orientation, suggesting that one crystal orientation may have a highly favorable orientation that minimizes elastic and/or plastic strain energy during thermal cycling. If so, then it is important to identify what orientations are energetically favored by the alternating stress states in a joint, and to determine how these orientations may grow to consume other existing orientations. Experimental measurements of a particular example of this phenomenon are presented along with computational modeling using elastic and plastic FEM analysis of particular orientations to quantify energy changes. Minimum elastic energy orientations are identified, and the texture of the copper appears to be influential, as copper has a strong elastic anisotropy.

11:00 AM
Interfacial Reaction between the Electroless Nickel Immersion Gold Substrate and Sn-3.0Ag-0.5Cu Solder: *Ruihong Zhang*[1]; Zhidong Xia[1]; Fu Guo[1]; [1]Beijing University of Technology

Electroless nickel immersion gold (ENIG) is one of the most widely used surface finishes on the Cu substrate in electronic packages. The formation and growth of intermetallics compounds (IMCs) at the solder/substrate interface are key factors affecting the solderability and reliability of electronic solder joints. This study will be focused on the morphology and growth of the interfacial IMCs at the interface of Sn-3.0Ag-0.5Cu solder and ENIG substrate. Three kinds of ENIG finishes with different Au thickness will be chosen to study the effects of Ni and Au layer on the IMCs and reliability of solder joints through isothermal aging studies, shear testing, and thermomechanical fatigue testing. The effects of Au layer on the morphology of the IMCs both at the solder/substrate interface and in the bulk solder will be looked into in details. The relationship between interfacial IMCs and reliability in various solder/ENIG/ Cu substrate systems will be established.

11:15 AM
The Effect of Microstructure on the Fatigue Life of Lead Free Solders: *Babak Arfaei*[1]; Peter Borgesen[2]; Pushkraj Tumne[1]; James Woods[1]; Jeremy Wolcott[1]; Eric Cotts[1]; [1]State University of New York; [2]Unovis-Solutions

To understand the mechanical behavior of Sn-Ag-Cu solder joints, the evolution of Sn grains morphologies, Sn grain orientations, and precipitate (Ag3Sn and Cu6Sn5) microstructures must be characterized. This study examined the mechanical response of Sn-Ag-Cu solder (SnAg2Cu0.5 (weight percent) on Cu pads on a high-Tg FR-4 substrate) in a load controlled shear fatigue test on individual solder balls. After failure, balls were individually mounted, cross sectioned, polished and examined by means of optical microscopy (with cross polarizers and in bright field conditions), and by electron backscattered

diffraction in an scanning electron microscope. Differences in the plastic deformation fields in different Sn grains in selected solder joints were examined. Correlations of variations in fatigue life of solder balls with the Sn grain size, number and orientation, and with precipitate size and number are reported.

Emerging Methods to Understand Mechanical Behavior: Diffraction Methods: Electron and Neutron

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Brad Boyce, Sandia National Laboratories; Mark Bourke, Los Alamos National Laboratory; Xiaodong Li, University of South Carolina; Erica Lilleodden, Forschungszentrum

Wednesday AM Room: 285
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Apurva Mehta, Stanford Synchrotron Radiation Laboratory; Jun Lou, Rice University

8:30 AM Invited
New Insides in Deformed Microstructures by 3D EBSD-Based Orientation Microscopy: *Stefan Zaefferer*[1]; [1]Max Planck Institute for Iron Research

We have developed a system for fully automated 3D orientation microscopy. It is based on precise serial sectioning by a focused ion beam (FIB) and EBSD-based orientation microscopy for characterisation of the sections. The technique delivers 3D crystal orientation maps with a resolution of 50 x 50 x 50 nm³. We have applied this new technique to the characterisation of various deformed microstructures: in order to get a better inside into the deformation process occurring during nano-indentation we observed the rotation fields and dislocation density distribution created by nano-indents into a copper single crystal. It was found that the deformation field expands in a self-similar manner with increasing indentation depth. Second, we applied the technique to study the microstructure of a heavily cold rolled FeNi alloy. Aim was to investigate the internal structure and local surrounding of cube-oriented areas. These areas are known to recrystallise first during an annealing treatment.

9:00 AM
The Importance of Grain Boundary Network Structures in Intergranular Failure: *Bryan Reed*[1]; Robert Rudd[1]; Eira Seppala[2]; Roger Minich[1]; Mukul Kumar[1]; [1]Lawrence Livermore National Laboratory; [2]Noki Research Center

With the rising availability of electron backscatter diffraction (EBSD), it is now possible to generate reliable statistical information about the networks of grain boundaries in polycrystalline materials. This analysis reveals strongly correlated clusters containing tens (sometimes hundreds) of grains with unusually high concentrations of coincident site lattice (CSL) grain boundaries. These clusters are particularly well developed in grain boundary engineered materials. When engineered and normal versions of nickel alloy are subjected to intergranular stress-corrosion cracking (in which the engineered material failed up to five times more slowly), we find that the fracture surfaces look completely different on the length scale governed by these clusters. We show how the combination of EBSD, mathematical models, and fracture analysis yields insights into the workings of intergranular fracture in general and grain boundary engineered materials in particular.

9:20 AM
Combining Experimental and Simulation Methods for Investigation of Microstructural Cyclic Plasticity: *Luke Brewer*[1]; Corbett Battaile[1]; Brad Boyce[1]; [1]Sandia National Laboratory

To develop truly predictive models that connect specific microstructures with changes in cyclical mechanical behavior, it is vital to combine experiment and simulation at the same, microstructural length scale. This talk will discuss the use of microstructurally-sensitive experimental techniques, such as electron back-scattered diffraction (EBSD), to provide starting microstructural information to simulation methods and to validate the predictions from these simulations. This study involves high-purity, polycrystalline nickel microstructures of varying grain size. Individual microstructures were tracked throughout their deformation using both ex situ and in situ mechanical testing followed by EBSD after a certain number of cycles. This data is compared to crystal plasticity-based finite element method simulations (both local and non-local models) of the same, starting microstructure. We will discuss the technical aspects of comparing experimental and simulation data at these length scales: including choice of boundary conditions, merging of coordinate systems, and algorithms for quantification of the comparison.

9:40 AM
Effect of the Environment on Crack Initiation and Propagation within 7XXX Series Aluminum Alloys: *Vipul Gupta*[1]; Sean Agnew[1]; Richard Gangloff[1]; [1]University of Virginia

Electron backscattered diffraction (EBSD) and SEM-based stereology have been applied along with focused ion beam surface preparation to investigate the crack path crystallography within 2-3 μm of the fatigue crack initiation site of precipitation hardened aluminum alloys, 7075 and 7050. Fatigue cracking within humid air and vacuum never led to fracture surface facets parallel to {111} planes, calling into question the slip band initiation mechanism. Backscattered electron imaging and energy dispersive spectroscopy of fracture surface indicate that crack initiation always occurs at constituent clusters in alloy 7075, which are also the most common initiation site within comparatively purer alloy 7050. EBSD is used to map the grain orientations and local plastic damage near the crack initiation sites. Combining EBSD results with crack growth rates measured on the fracture surface within the microstructurally small crack (MSC) regime using a marker banding technique allows validation of microstructure-based models to explain MSC behavior.

10:00 AM
Nanoindentation-Correlated EBSD Study of Delamination Fracture in Al-Li Alloys: *Wesley Tayon*[1]; Roy Crooks[2]; Marcia Domack[3]; John Wagner[3]; Abdelmageed Elmustafa[1]; [1]Old Dominion University and the Applied Research Center; [2]National Institute of Aerospace; [3]NASA Langley Research Center

Al-Li alloys offer attractive combinations of high strength and low density. However, a tendency for delamination fracture has limited their use. A better understanding of the delamination mechanisms may suggest a solution to this problem through processing modifications. We have brought a combination of new techniques to bear on this issue. Both high quality electron backscattered diffraction (EBSD) information and valid nanoindentation measurements were obtained from the same sampled regions. Samples were taken from as-received material and from fractured test specimens. Correlations were drawn between delamination, nanoscale property variations and local texture; and compared to associated processing parameters.

10:20 AM
Grain Boundary Engineering Alloy 800HT: *Daniel Drabble*[1]; Milo Kral[1]; [1]University of Canterbury

Grain boundary engineering has recently shown promise in its ability to improve mechanical properties of metals. The aim of the present work was to investigate the feasibility of using grain boundary engineering to improve the high-temperature properties of a common superalloy, alloy 800H/HT. A variety of sequential thermo-mechanically processing conditions were first applied to the alloy. Samples were then analysed using Electron Backscatter Diffraction (EBSD) to characterize the grain boundary character and Σ3 grain boundary connectivity. The effects of the sequential thermo-mechanically processing on steady-state creep rate were then obtained in uniaxial tension creep tests with real-time strain measurement under common service conditions, in contrast to accelerated creep rupture tests for example.

10:40 AM Break

10:55 AM
Analysis of Orientation Gradients and Discontinuous Microstructure in Phase-Transformed Tantalum Thin Films: *Ray Fertig*[1]; Robert Knepper[1]; Max Aubain[1]; Shefford Baker[1]; [1]Cornell University

The equilibrium (alpha) phase of bulk Ta at room temperature and pressure is bcc, but it is possible to deposit thin films of Ta in the metastable tetragonal (beta) phase under certain conditions. We have recently discovered a unique

microstructure in thin alpha-Ta films created by phase transformation from the beta phase. This microstructure is characterized by continuous changes in orientation with position and a discontinuous boundary structure. In the present work, we use electron backscattered diffraction (EBSD) to study the orientation gradients in this microstructure. A detailed analysis of the crystal rotations was used to determine the minimum density of geometrically-necessary dislocations needed to generate the observed microstructure. Variations in dislocation density with film thickness and oxygen content suggest that the unusual microstructure arises from kinetic limitations during the phase transformation.

11:15 AM

Using Orientation Imaging Microscopy Data for Direct Input and Validation of Subgrain Texture and Microstructure Evolution of Polycrystalline Copper: *Ricardo Lebensohn*[1]; Renald Brenner[2]; Olivier Castelnau[2]; [1]Los Alamos National Laboratory; [2]Université Paris XIII

We present results of a numerical formulation based on Fast Fourier Transforms (FFT) to obtain the micromechanical fields in plastically deformed 3-D polycrystals using direct input from a digital image of their microstructures. The FFT-based formulation provides an exact solution of the governing equations in a periodic unit cell, has better performance than a Finite Element calculation for the same purpose and resolution, and can use voxelized microstructure data as input and for validation. To illustrate the capabilities of this model, we first discuss the construction of a 3-D unit cell using 2-D orientation maps obtained by means of Orientation Imaging Microscopy (OIM), and we then show FFT-based predictions, together with the corresponding OIM measurements, on subgrain texture evolution in a recrystallized Cu polycrystal deformed in unixial tension. The measured rotations of the average orientations and the orientation-dependent magnitudes of the average misorientations are well reproduced by the FFT-based model.

11:35 AM Invited

In-Situ Neutron Scattering Studies of Magnetic Shape Alloys under Stress, Temperature and Magnetic Fields: *Donald Brown*[1]; Yandong Wang[2]; Saurabh Kabra[1]; Thomas Sisneros[1]; [1]Los Alamos National Laboratory; [2]Northeastern University

We have utilized the SMARTS engineering diffractometer to study the crystallographic orientation and phase transformations in the ferromagnetic shape memory alloy NiMnGa under conditions of temperature and stress. Neutrons are uniquely suited to probe the crystallographic response of materials to external stimuli because of their high penetration, which allows them to sample the bulk of the material as well as pass through environmental chambers. We observed that the addition of relatively small stresses greatly effects the selection of variants during transformation from the austenitic to martensitic phase. Since the initial experiments, the capability to apply a magnetic field of 2T transverse to the loading direction has been integrated to SMARTS. The details of this new and unique capability to control all three stimuli simultaneously on a diffractometer will be presented in this talk. If all goes well in the upcoming run-cycle, preliminary results from initial experiments will also be presented.

12:05 PM

Detwinning of Pure Zirconium: In-Situ Neutron Diffration Experiments: *Gwenaelle Proust*[1]; George Kaschner[1]; Donald Brown[1]; Carlos Tome[1]; Irene Beyerlein[1]; [1]Los Alamos National Laboratory

Twinning is an important deformation mode in hexagonal polycrystalline metals to accommodate deformation along the c-axis. Twinning differs from slip in many ways, but we focus here on the behavior of twins during reversal loading. Previous in-situ neutron diffraction experiments performed on magnesium at the Lujan Neutron Scattering Center (LANSCE) show that during load reversal, the material detwins once the loading direction is changed. We have performed similar experiments on pure zirconium to see if it behaves as magnesium and to determine what role temperature plays in the detwinning mechanism. The experiments were motivated by previous studies of clock-rolled zirconium, deformed in the quasi-static regime for temperatures ranging from 76K to 450K, where it was observed that two tensile and one compressive twinning modes can be activated. We limit our study here to the behavior of the {10-12}(10-1-1) tensile twins during reversal loading at room temperature and 76K.

12:25 PM

Dynamical Diffraction Studies of Deformation Mechanisms in Ductile Intermetallic YCu: *Scott Williams*[1]; Donald Brown[2]; Bjorn Clausen[2]; Alan Russell[3]; Karl Gschneidner[3]; [1]Iowa State University; [2]Los Alamos National Laboratory; [3]Ames Laboratory

A recent discovery has shown a family of rare earth intermetallic compounds having the B2 crystal structure which show intrinsic ductility. In-situ neutron diffraction tests while loading, conducted on YCu using SMARTS at LANSCE, have revealed the sample to be operating within the dynamical diffraction regime. The observed extinction effect is relatively uncommon within diffraction of engineering polycrystals, either because of the over-shadowing effect of true absorption (as with XRD) or because samples lack the necessary crystalline perfection. By analyzing diffraction data for changes in dynamical behavior, a new method arises for studying deformation and dislocation structure development. Examining these changes in YCu data indicate the development of large regions of perfect crystallinity as a result of annealing. With deformation, dislocation structures disrupt the perfect lattice, destroying the dynamical diffraction condition. These changes in the dislocation structure may be related to the deformation mechanisms within ductile rare earth intermetallic YCu.

Energy Conservation in Metals Extraction and Materials Processing: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Aqueous Processing Committee, TMS: Pyrometallurgy Committee, TMS: Recycling and Environmental Technologies Committee

Program Organizers: Edgar Vidal, Brush Wellman, Inc.; Cynthia Belt, Aleris International Inc

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March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Christopher Keller, Aleris International Inc; Cynthia Belt, Aleris International Inc

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8:30 AM

Enhanced Energy Efficiency and Emission Reduction through Oxy-Fuel Technology: Dietrich Gross[1]; Mark Schoenfield[1]; Thomas Weber[1]; Brian Patrick[1]; *Norman Bell*[1]; [1]Jupiter Aluminum Inc.

This paper describes an advanced oxy-fuel combustion technology for re-melting aluminum as well as alumina calcinations processing that significantly reduces energy consumption and emissions. This oxy-fuel combustion technology developed by Jupiter Oxygen is a patented process for combustion of fossil fuels with pure oxygen while excluding air, thereby creating an undiluted high flame temperature and increased efficiency for process heating in industrial furnaces. This unique process has led to improved heat transfer and longer residence time while maintaining the same process temperatures as with air combustion. This energy efficient combustion process allows aluminum companies to operate at a substantially lower cost due to dramatic fuel savings of approximately 70% for natural gas and waste oil. The results from more than 10 years of day-to-day experience with oxy-fuel combustion in the aluminum re-melting business are discussed.

8:50 AM

Computational Modeling of Gas Fired Immersion Heaters for Recycled Aluminum Melting: *Mohammed Hassan Ali*[1]; Marwan Khraisheh[1]; Shridas Ningileri[2]; Subodh Das[1]; [1]University of Kentucky; [2]Secat Inc

Recent results have shown that the use of immersion heaters in aluminum melting is more efficient and clean compared to melting using the regular reverberatory furnaces. Immersion heater parameters including size, number, distribution, material, and heat flux characteristics affect the efficiency of the melting process. A detailed parametric study is needed to optimize the design of immersion heaters. In the present study a 3-D computational model is developed to simulate the melting process of pure aluminum using different immersion heater parameters and configurations. The results clearly show the

effect of immersion heaters on the temperature distribution, molten metal natural circulation and solid fraction during melting.

9:10 AM
Improving Combustion with a Low Velocity Vortex Generator Premix Industrial Gas Burner: *Tibbs Golladay*[1]; [1]Burner Dynamics, Inc

Until recently, the resilience of our environment and unlimited availability of natural resources have been taken for granted and have not been seriously considered in the design of the machines that drive our industrialized world. The fact that the basic design of an industrial gas burner has not changed in over 100 years clearly demonstrates this. Unfortunately, many methods used to reduce emissions generated by the combustion of natural gas come with a trade off in efficiency and operating costs. This paper will present the advantages of utilizing a low velocity vortex generator as a method of thoroughly premixing the fuel and air immediately prior to combustion which results in closer to stoichiometric combustion than the typical design of industrial gas burners. The obvious benefits of stoichiometric combustion are more complete combustion, less undesirable by-products of combustion and more efficient heat generation.

9:30 AM
Oxyfuel - Solutions for Energy and Environmental Conservation: *Thomas Niehoff*[1]; [1]Linde Gas

Many industrial processes require energy. The energy is often used for melting or heating purposes. The heat can be generated by electrical heating or combustion/oxidation of hydrocarbons. Oxygen supports the combustion process. Oxygen is naturally contained in air with about 20.9 vol.-%. The use of technical oxygen will allow an increase to the natural oxygen concentration of combustion air. Altering the combustion process by increasing the oxygen content has various effects. These effects are described and analyzed with regard to heat transfer, emissions and energy consumption. Industrial processes requiring oxyfuel are described and discussed regarding energy aspects.

9:50 AM
Global Warming Expands the Scope of Heat Pipe Applications: *Dumitru Fetcu*[1]; [1]Econotherm (UK)

The paper examines several heat pipe heat exchanger approaches to heat recovery. In each approach waste energy is recovered and used to preheat air or water, to provide electricity, to provide free heat or simply to improve the efficiency of a system. The presented heat pipe concepts provide the waste heat recovery system designer with viable alternatives to classical heat transfer techniques and heat exchangers.

10:10 AM Break

10:30 AM
Waste Heat Recovery: Opportunities and Challenges: *Ilona Johnson*[1]; William Choate[1]; Sara Dillich[2]; [1]BCS, Inc.; [2]US Department of Engergy

United States metals and non-metallic minerals manufacturing consume about 5,000 trillion BTU annually. Various studies estimate that as much as 20 to 50 percent of this energy is lost as excess or waste heat. Recovery of waste heat can conserve energy, reduce operating costs, and lower environmental emissions. It would be valuable to quantify recoverable waste heat sources and identify the barriers that hinder its recovery. This paper provides an overview of waste heat losses specific to high temperature processes in primary metals and minerals manufacturing. It summarizes findings from a broader study conducted for the Department of Energy Industrial Technologies Program; the study evaluates energy losses, recovery practices and opportunities, and Research and Development needs for waste heat technology development.

10:50 AM
Analysis of Energy Efficiency for Industrial Processes: *Cynthia Belt*[1]; Ray Peterson[1]; [1]Aleris International Inc

Improved energy efficiency has grown in importance within industry as energy costs have escalated. The analysis of trends and changes to a process is critical in making the best business decision in energy management. Frequently, energy use per day (Kilowatt-hour or British Thermal Units) is used to track energy efficiency. Sometimes, the somewhat more complex method of energy per processed weight (Kilowatt-hour per Metric Ton or BTU per Pound) is used to make this evaluation. Yet both methods ignore the effect due to throughput and weather. In this paper, different thermal processes and systems will be evaluated using statistical methods to highlight alternate methods to evaluate energy efficiency. Examples and potential problems will be discussed for both a single process and a collection of processes within a plant or corporation.

11:10 AM
An Investigation of the Flame-Burden Interaction during Remelting in an Experimental Aluminum Reverbatory Furnace: *Ashwini Kumar*[1]; Raj Venuturumilli[1]; Paul King[2]; [1]ANSYS, Inc.; [2]National Energy Technology Laboratory

Flame impingement on the burden is routinely encountered during the initial phase of melting in aluminum reverberatory furnaces. This impingement causes an obstructed pathway for the hot gases, and hence the circulation and residence time of the hot gases are greatly impacted. Moreover, as the load melts, the flames gradually achieve an unobstructed path leading to reduced gas residence times. This flame impingement and constantly changing combustion space volume will lead to vastly different fuel and oxidizer mixing patterns and thus affect the overall furnace performance. Fine tuning the burner operating conditions such as flow rates and injection angles with the changing combustion space could result in significant improvements to the furnace efficiency. However, one has to gain a better understanding of the furnace dynamics to know the suitable parameters to adjust. Physical modeling can be elaborate and expensive to conduct on a regular basis while Computational Fluid Dynamics (CFD) can cost-effectively address this challenge. In this study, the furnace model is created with a particular loading pattern to understand the flame dynamics in the presence of a piled load. This configuration can be thought of as a computational model of the furnace with the burden at a particular stage of the melting process. Thermal efficiency and behavior of the furnace are quantified and the predicted values are compared with the operational data from an experimental reverberatory furnace.

11:30 AM
Low Temperature Conversion of Organic Waste into Energy and Energetic Gases: *Edgar Vidal*[1]; Scott Shuey[2]; Patrick Taylor[1]; Paul Kruesi[3]; [1]Colorado School of Mines; [2]Phelps Dodge Corporation; [3]Cato Research Corporation

Experiments have bee performed to transform polymeric type materials into energy, energetic gases and manageable secondary products. Typically a mixture of methane, hydrogen, carbon monoxide/dioxide have been obtained in controlled pyrolisis of polymeric materials. The supression of dioxin/furane formation was a must, and kept under control. The by-product obtained was easily handled and contained carbon black and water-soluble salts. Interpretation of results obatined are presented along with conclusions.

General Abstracts: Electronic, Magnetic, and Photonic Materials Division: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS: Electronic Materials Committee, TMS: Electronic Packaging and Interconnection Materials Committee, TMS: Nanomaterials Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Sung Kang, IBM Corporation

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Session Chairs: Long Qing Chen, Pennsylvania State University; Sung Kang, IBM Corporation

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8:30 AM
Studies on ZnO Based Diluted Magnetic Semiconductors: *Sasanka Deka*[1]; [1]National Nanotechnology Laboratory of INFM

The carrier induced ferromagnetism in the transition metal (*TM*) doped wide band gap II-VI and III-V nonmagnetic semiconductors opened up a new area of research and potential applications in spintronics devices. Such systems, called diluted magnetic semiconductors (DMS), exhibit inherent properties from the 'charge' and 'spin' of an electron in a single material. DMSs combine their

transport and optical properties with magnetism. The widely discussed DMS system, (Ga,Mn)As in the III-V series, shows the highest Curie temperature as 110 K. In the II-VI series, the widely discussed DMS system is $Zn_{1-x}TM_xO$ (TM = Mn, Co, Ni, Fe, Cu, Cr, etc). However, the origin of ferromagnetism in this oxide based DMS is still under controversy. In most of the reports, the Curie temperature and the order of magnetism is not identical even for an identical system. As most of the studies on DMSs were on thin films, we have carried out synthesis and studies on magnetic and optical properties on TM doped ZnO nano crystalline powder. We observed the evidence of the origin of ferromagnetism in the Co and Ni doped ZnO system as the metal nano-clusters, though optical and electronic structure studies indicate the incorporation of divalent metal ions inside the ZnO crystal lattice. [1]Pearton S. J., Abernathy C. R., Overberg M. E., Thaler G. T., Norton D. P., Theodoropoulou N., Hebard A. F., Park Y. D., Ren F., Kim J. and Boatner L. A. *J. Appl. Phys.* (2003) **93**, 1. [2]Ohno H. Science (1998) **281**, 951. [3]Deka S. and Joy P. A. *Chem. Mater.* (2005) **17**, 6507. [4]Deka S. and Joy P. A. *Phys. Rev. B* (2006) **74**, 033201. [5]Deka S. and Joy P. A. *Appl. Phys. Lett.* (2006) **89**, 032508.

8:50 AM

Characterization of 3-Phase (Ternary-Like) N-Type and P-Type Thermoelectric Materials Fabricated by Explosive (Shock) Consolidation: *V. Muñoz*[1]; Lawrence Murr[2]; D. Nemir[3]; R. Lovrenich[3]; E. Martinez[2]; S. Gaytan[2]; M. Lopez[2]; [1]OSRAM Sylvania, Inc.; [2]University of Texas at El Paso; [3]TXL Group

We prepared 3-phase powder mixtures of Bi-Te-Se and Bi-Te-Sb by cylindrical explosive consolidation to emulate traditional, melt-grown n-type and p-type doped Bi_2Te_3 thermoelectric materials. The powder diameters ranged from 40 µm to 3 µm, with starting Bi and Te particle Vickers microindentation hardnesses of 0.18 and 0.56 GPa, respectively in contrast to average shocked n-type and p-type monolith microindentation hardnesses of 1.15 and 1.31 GPa, respectively; compared to 0.72 and 0.68 GPa for the melt-grown n-type and p-type materials respectively. Shock consolidated monolith microstuctures were characterized by optical metallography, SEM and TEM along with EDS analysis and XRD of all materials, including the melt-grown materials which had measured thermoelectric parameters considerably in excess of the shock (explosively) consolidated monoliths. Notable shock microstructures were characterized as deformation twins, while the melt-grown materials contained considerable, fine eutectic microstructures.

9:10 AM

Electrical and Thermal Stress Effect on Switching Process of VDMOSFETs: *Chafic Salame*[1]; Pierre Mialhe[2]; [1]Lebanese University; [2]Perpignan University

Evolutions of the switching parameters of commercial VDMOSFET after a hot carrier injection are inspected. Experiment was done by hot carrier injection, where a large drain-source voltage is applied to reverse bias the Body drain junction then inducing a 30 mA reverse current. Switching time parameters were measured at different temperature and up to 300°C. The experimental results show that the device rise time decreases significantly for the first period of stress at room temperature, making the device faster during his turn-on switch. This event was associated to the high electric field in the junction that pulls electrons from the oxide gate into the channel, thus leaving trapped holes in the oxide bulk due to their low mobility. This study has an important value in terms of engineering application where speed of electronic devices is one of the most valuable parameters in communication and information technology fields.

9:30 AM Break

10:00 AM

Electrical Characterisation of Tantalum Anodes for Use in Semiconductors: *Rizwan Rahman Rashid*[1]; Siva Jyoth[1]; Rajkiran[1]; Prasad Goud[1]; [1]Mahatma Gandhi of Technology

This paper reveals the studies on the processing and electrical properties of tantalum anodes using tantalum powder. Processing methods of tantalum anodes using high purity sodium reduced tantalum powder, compaction, sintering and dielectric layer formation were studied. Different loads were applied while compacting the tantalum powder for making green anodes prior to high vacuum sintering to study its effect on the electrical properties of the Ta pellet. Different formation voltages were applied during the dielectric layer formation and then the capacitance measurements were carried out. This paper discusses these studies carried out as a project work in C-MET (Centre for Materials for Electronics Technology).

10:20 AM

Experimental Study of Co-Deposition of SiO_2 and GeO_2 in a Modified Chemical Vapor Deposition (MCVD) Reactor: Mainul Hasan[1]; *Shiliang Zhan*[1]; [1]McGill University

MCVD reactors are used to commercially manufacture high quality optical fibers. A MCVD reactor consists of a slowly rotating, fire polished silica tube wherein a mixture of gaseous silicon tetrachloride ($SiCl_4$), germanium tetrachloride ($GeCl_4$) and carrier oxygen gas is introduced at one or both ends of the tube. The tube is heated by slowly traversing an oxy-hydrogen torch to allow for the high temperature chemical reactions to produce fine particles of SiO_2 and GeO_2. These particles move with the un-reacted gas mixture and deposit onto the inner surface of the tube. Co-deposition experiments were carried out on a laboratory scale MCVD reactor. The thickness of the deposited layer and the concentrations of germanium, silicon and oxygen were measured by EDS. Experimental results were obtained by varying the torch heat fluxes (temperatures), torch moving speed, tube rotational speed, carry-on gas amount and in-take ratio of $GeCl_4/SiCl_4$.

10:40 AM

Extraction of MOSFET Devices Electrical Parameters Evolution at High Temperatures: *Chafic Salame*[1]; Roland Habchi[1]; [1]Lebanese University

Junction parameters of a MOSFET (Metal Oxide Semiconductor Field Effect Transistor) device are calculated based on a numerical double exponential model. The temperature dependence of these parameters is investigated; their evolution allows the evaluation of device's operation reliability in high temperature environments. The values of the shunt and series resistance, the ideality factor, the diffusion and recombination currents are determined. The accuracy of the model is investigated at high temperature.

General Abstracts: Materials Processing and Manufacturing Division: Forging, Forming, and Powder Processing

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: Ralph Napolitano, Iowa State University; Neville Moody, Sandia National Laboratories

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Session Chairs: John Carsley, General Motors Corporation; Amine Benzerga, Texas A&M University

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8:30 AM

Further Development on a Flashless Precision Forged Two-Cylinder-Crankshaft: *Sven Mueller*[1]; [1]IPH - Institut für Integrierte Produktion Hannover GbmH

Precision forging is defined as a flashless near-net-shape forging. A quality in the tolerance class from IT 8 to IT 10 can be achieved. The flashless precision forging offers an integrated heat treatment with the forging heat, because a clipping process is not necessary. After the forging and heat treatment often only a final fine machining of the functional surface with small chip volume is required. In the collaborative research project SFB 489 a two-cylinder-crankshaft is selected as an example for complex flat long pieces with a characteristic mass distribution along the longitudinal axis. The continuative development considers the thermal and mechanical tool stresses, which are also as complex as the crankshaft itself. Further on a parameter study on a bi-directional forging tool is made. It is planned to test the forging sequence, tools, cooling systems and measuring technology of the developed process in the end of 2007.

8:50 AM

The Effect of the Residual Stress on the Inner Surface Cracking during Small-Radius Bending of AA6111 Sheets: *Zhuoqun Li*[1]; Xin Wu[1]; [1]Wayne State University

Small-radius bending is one of the widely used methods in sheet metal hemming for manufacturing autobody closure panels, and by bending over a straight-edge the bendability of sheet metals can be assessed. During unloading from bending residual stress develops that not only causes geometrical change due to springback, but also leads to a residual tensile stress on the inner surface which, depending on the bending geometry and material's fracture behavior, can be sufficiently high to cause material fracture. To date, majority of the bending studies have emphasized mainly on outer surface fracture and springback, but limited study exists on inner surface fracture. In this paper, fracture of inner surface in bending AA6111 sheets was observed and characterized by SEM and OM, and the stress development during small-radius bending and springback were analyzed that can describe the inner surface cracking process.

9:10 AM

Controlled Sheet Metal Straightening: *Richard Krimm*[1]; Bernd-Arno Behrens[1]; [1]Institute for Metal Forming and Metal-Forming Machines

Straightening the sheet-metal in advance of forming operations needs to be improved. Today the straightening rolls are positioned manually or by means of an adjustment unit by the machines operator using his expert knowledge. A controlled adaptation of the rollers positions to the depending on the coil-radius variable curvature and properties of the sheet-metal is not realised yet. For this reason the flatness of the straightened sheet metal is limited and modifies while unreeling the coil. The paper shows a way to control the roller positions depending on the residual bending of the straightened sheet metal. The controlling system of a testing straightener was amended to vary the roller positions in dependence of the sheet metals residual bending. Using adequate controller settings it was possible to stabilise the straightening process to a certain extend and to obtain good straightening results from coiled sheet metal.

9:30 AM

Metal Hardening Models at Large Strains - FEM Validation with Digital Image Correlation: Wayne Cai[1]; *John Carsley*[1]; Dan Hayden[1]; Louis Hector[1]; Tom Stoughton[1]; [1]General Motors Corporation

Simulation accuracy of large strain deformation of sheet metals, as occurs during hemming and in vehicle crash situations, is limited because existing hardening laws (true stress - strain relationships) are calibrated from data only in the uniform deformation regime and require extrapolation in the diffuse and localized necking stages. A reverse engineering method was developed to predict hardening laws for large strains beyond uniform elongation. The method was implemented as a code requiring uniaxial tensile test data and finite element analysis. The true stress - true strain data pairs were determined when the load - displacement history of a tensile test matched the FEA results. Test cases showed that true stress - true strain relationships were generated for very large strains (75% for AA6111, and 85% for DP600). The method was validated using digital image correlation techniques for strain measurement on the surface of a tensile sample.

9:50 AM

An Improved Plasticity Model of Anisotropic Ductile Porous Solids: *Shyam Keralavarma*[1]; Ahmed Benzerga[1]; [1]Texas A&M University

Forming limits and fracture modes in structural materials are significantly influenced by microstructural details such as material texture, porosity, shape and distribution of microvoids. Accurate prediction of plastic flow localization and fracture during processing and product manufacturing requires the development of constitutive models that incorporate the effects of evolving microstructural parameters. In this work, we present a contribution toward this goal by developing, on rigorous micromechanistic bases, a constitutive model of porous solids incorporating the effects of plastic anisotropy and cavitation-induced damage. In particular, the model accounts for void shape effects and void closure. Plastic anisotropy data for a range of practically important materials is tabulated and the model predictions for the yield loci and microstructure evolution are compared with numerical limit analysis results and finite element simulations of porous unit cells for representative materials. Greater accuracy is obtained vis-a-vis previous models in predicting the plastic response of anisotropic solids.

10:10 AM

Drilling and Milling of Intermetallic Gamma-TiAl: *Stefan Bergmann*[1]; Dirk Biermann[1]; [1]Department of Machining Technology

Gamma-Titaniumaluminide is an intermetallic compound. It offers a very good hot strength, a high Youngs Modulus and tensile strength as well as a low density (3.9 g/cm3). The fields of application are e. g. conrods, turbochargers for combustion engines and turbine blades. The advantage of deploying Gamma-TiAl results from a reduction of moving masses together with a minimization of engine vibrations. Contrasting with the outstanding lightweight material properties, problems occur when machining operations are carried out. These problems result from the very high hardness and brittleness, which leads to high cutting forces and rapid tool wear. The presentation features investigations on the machining of a Gamma-TiAl-alloy. For this reason, studies on drilling and milling were conducted, focusing on appropriate conditions for a high-quality machining process. The presentation shows results from selected machining tests, e. g. tool wear, cutting forces, surface integrity as well as tool and process optimizations.

10:30 AM Break

10:40 AM

Hot Isostatic Pressing of Titanium Powder Compacts Using a Ceramic Secondary Pressing Medium (HIP-SPM): *Fatos Derguti*[1]; Richard Dashwood[1]; [1]Imperial College London

We present a new low cost Near Net Shape (NNS) technique for titanium powder. Commercially pure Hydride DeHydride (HDH) Ti powder was compacted to rod samples of 70% of theoretical density using "wet-bag" Cold Isostatic Pressing (CIP) followed by HIPing using ceramic beads as a secondary pressing medium (SPM). The effect of SPM media and powder size of the mechanical properties will be presented, and compared to an FE model for HIPing. Some observations are also presented on porosity, the reaction layers that form and oxygen uptake in the samples.

11:00 AM

The Adiabatic High Velocity Compaction of Titanium Powder: *Gordon Goranson*[1]; [1]LMC Inc.

LMC compacts titanium powders to net shape components with reduced sintering time and temperature, or no sintering. One Tool – One Impact – One Part. The patented impact unit achieves ram velocities up to 100 meters per second creating adiabatic phenomena in powder, dislocating molecules and rejoining them in a solid state. Material yield is maximized. Surface finish is determined by tooling. High cycle rates are achieved. No lubricants. Part densities have been achieved to over 99%. The LMC process will produce: pellets that can be controlled for processing by smelters for extrusions, rolled or machined; near net shape billets to be finished machined; net shape parts. Presses capable of components to 20 kilos. The High Velocity Adiabatic Impact Process is fully commercialized for cutting, blanking, and forming. Environmentally friendly. No recyclables, minimum material waste and up to an 85% reduction in energy consumption compared to conventional presses.

11:20 AM

World Record Thermal Conductivity of Nano-Tungsten-Copper Powder: Seong Lee[1]; Joon-Woong Noh[1]; Young-Sam Kwon[2]; John Johnson[3]; *Seong Jin Park*[4]; Randall German[4]; [1]ADD; [2]CetaTech; [3]ATI Alldyne; [4]Mississippi State University

Tungsten-copper is a leading candidate material for thermal management. However one of the huddles for this material is the difficulty to improve interfacial properties between tungsten and copper. In this study, nano tungsten coated copper powder has been developed with a range of compositions from 90W-10Cu to 10W-90Cu in weight percentage through a mechano-reduction process starting from oxides. Powder injection molding and die compaction were used to make samples to evaluate microstructure, thermal conductivity (TC) and coefficient of thermal expansion (CTE) with density and heat capacity for several compositions of tungsten coated copper powder. The measured TC lies among theoretical values predicted by the model with consideration of microstructure. Furthermore, the presentation will present mechanical properties such as hardness and compressive strength with shear rate dependency and electrical property of conductivity (or resistivity).

11:40 AM

Preparation of High-Purity Strontium Carbonate from Celestite Concentrate by Double Decomposition-Roasting Process: *Mudan Liu*[1]; Tao Jiang[1]; Guanghui Li[1]; [1]Central South University

The technology of using celestite concentrate to prepare high-purity strontium carbonate by double decomposition-roasting process was studied. The results showed that a maximal $SrSO_4$ conversion of 97.77% was obtained when Na_2CO_3 was used under the conditions of liquid/solid ratio of 5/1, Na_2CO_3 concentration of 1.1mol/L, Na_2CO_3/ $SrSO_4$ molar ratio of 1.2, temperature of 75°C, and time of 75min. The maximal conversion was only 78.66% when $(NH_4)_2CO_3$ was used under the conditions of liquid/solid ratio of 5/1, $(NH_4)_2CO_3$ concentration of 1.7mol/L,$(NH_4)_2CO_3$/ $SrSO_4$ molar ratio of 1.2, temperature of 60°C and time of 150min. The crude strontium carbonate were roasted at 1200°C for 45min with anthracite, and the ratio of strontium carbonate and anthracite was 10/1, then the conversion was 72.30%. The high-purity strontium carbonate were obtained after the roasted products were leached, got rid of impurity and carbonated, in which the content of $SrCO_3$ exceeded 98%, and $CaCO_3$ was less than 0.5%.

General Abstracts: Structural Materials Division: Microstructure/Property Relations in Steel I

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: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizer: Ellen Cerreta, Los Alamos National Laboratory

Wednesday AM Room: 387
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Michele Manuel, University of Florida

8:30 AM

Austenite Grain Stability of Low-Manganese, High-Chromium Carburizing Steel: *Takeshi Fujimatsu*[1]; Kazuya Hashimoto[1]; Shinji Fukumoto[2]; Atsushi Yamamoto[2]; [1]Sanyo Special Steel Company Ltd.; [2]University of Hyogo

Since automotive drive train parts require cost reduction as well as reduction in size and weight for improving fuel consumption, carburizing steel for those parts is expected to have lower cost, better cold formability and machinability, and higher strength. We found that the steel with reduced manganese and increased chromium contents meets these needs. It was also confirmed that this developed steel showed higher austenite grain coarsening temperature during carburizing than conventional steel. The microstructures of the low-manganese, high-chromium carburizing steel were investigated at various stages from hot forging through carburizing processes, and the improvement mechanisms of austenite grain coarsening temperature were discussed in this study.

8:50 AM

Deterioration of Fracture Toughness of Stainless Steel Due to the Phenomenon of Sensitization: *Swati Ghosh*[1]; Kalyan Ray[1]; Vivekananda Kain[2]; [1]Indian Institute of Technology; [2]Bhaba Atomic Research Center

This investigation aims to illustrate how degree of sensitization of a stainless steel affects its fracture toughness, estimated by a simplified technique. Solutionized nuclear grade 304LN stainless steel were subjected to sensitization treatment at 1023 K for different time periods in the range 1-100 hr. Corrosion resistance of the differently heat-treated samples were assessed by oxalic acid-etch and electrochemical potentiokinetic reactivation tests. The fracture toughness of the heat treated specimens was determined by a recently developed procedure based on Ball Indentation principle and validated by standard J-integral tests. The results reveal that the fracture toughness of stainless steels gets significantly deteriorated by sensitization. The supplementary examination of microstructure, grain size and hardness and tensile tests explains the cause of such a degradation in toughness as to be due to martensite formation and stress build up due to the grain boundary carbide precipitation.

9:10 AM

Development of High Strength Non-Oriented 3% Silicon Steel: *Hee Yong Park*[1]; Sam Kyu Chang[1]; Bruno De Cooman[1]; [1]GIFT

Current electrical steels usually are not fatigue resistant and so fatigue failure occurs during the operation, in motors for example, due to the overload from high frequencies. To use the electrical steels in large motors used in power plants, ship, aircraft and etc, we need to overcome this problem by increasing the yield strength and toughness of the electrical steel without losing its magnetic properties. Ability to withstand high frequencies and temperature is also required. To obtain the increase in mechanical properties without losing its magnetic properties, new elements such as B, Sb, Mo, Ni and Nb are added and higher amount of Al and Mn are added to the current electrical steel.

9:30 AM

Magnetic Properties and Textural Development of High Strength 3%Si Electrical Steels: *Sam Chang*[1]; [1]Pohang University of Science and Technology

For the development of high strength electrical steels having yield strength of 450 N/mm2 to meet the recent requirement for high fatigue resistance for large motors, it is also of important to investigate the magnetic properties and structural characteristics in such a high mechanical property electrical steel because in a high strength steel contains several alloying elements which are known detrimental to magnetic properties. Current electrical steels usually are not fatigue resistant and so fatigue failure occurs during the operation, in motors for example, due to the overload from high frequencies. To use the electrical steels in large motors used in power plants, ship, aircraft and etc, we need to overcome this problem by increasing the yield strength and toughness of the electrical steel without losing its magnetic properties. Ability to withstand high frequencies and temperature is also required. To obtain the increase in mechanical properties without losing its magnetic properties, new elements such as Sn, Sb, Ni and Nb are added and higher amount of Al and Mn are added to the current electrical steel.

9:50 AM

Characteristics of Hybrid Solid Solution Layer Formed on the Surface of 316 L Austenitic Stainless Steels: *Ozgur Celik*[1]; Eyup Sabri Kayali[1]; Huseyin Cimenoglu[1]; [1]Istanbul Technical University

Among metallic materials, AISI 316L quality austenitic stainless steel is extensively used in manufacturing of orthopedic implants. Although it exhibits very good corrosion resistance in many acidic environments, synergetic attack of corrosion and wear in the body causes severe surface damage and loosening of the implant. It has been reported that surface hardening by low temperature plasma nitriding provides a considerable improvement in wear resistance as well as the corrosion properties of austenitic stainless steels. In the present study, low temperature nitriding was applied to an AISI 316L quality austenitic stainless steel in a fluidized bed to form a hybrid solid solution layer composed of nitrogen and carbon rich expanded austenite zones. The surface characteristics of nitrided alloy was examined through structural examinations, mechanical tests as well as biocompatibility tests and compared with those of Rex 734, which was recently produced austenitic stainless steel for manufacturing of implants.

10:10 AM

Deformation-Induced Martensitic Transformation in High-Nitrogen Metastable Austenitic Stainless Steels: *Chang-Seok Oh*[1]; Tae-Ho Lee[1]; Sung-Joon Kim[1]; [1]Korea Institute of Materials Science

Deformation-induced martensitic transformation(DIMT) in high-nitrogen metastable austenitic stainless steels was investigated in terms of the effects of nitrogen content and the microstructural change. During tensile deformation, the stress-strain response was changed at the transient point(at around 0.1~0.3) depending on nitrogen contents, induced by DIMT. The critical strain for DIMT was shifted to higher strain with increasing nitrogen contents, and DIMT did not occur in the nitrogen contents above 0.5wt.%. At the beginning of deformation, the austenite to epsilon martensitic transformation occurred, whereas the formation of alpha prime martensite prevailed after the transient point, which caused the shape change of stress-strain curve. Based on analyses of SAD patterns, the orientation relationships between austenite and deformation-induced martensites were determined. The changes of martensite volume fraction

and crystallographic features(lattice parameters and orientation change) during deformation were also analyzed using EBSD and neutron diffraction.

10:30 AM Break

10:50 AM

Effect of Cooling Rate on the Hot Ductility of Boron Bearing Steel: *Kyung Chul Cho*[1]; Yang Mo Koo[1]; Joong kil Park[2]; [1]Pohang University of Science and Technology; [2]Pohang Iron and Steel Company

In boron bearing low carbon steels, corner cracking can occurs if the hot ductility of these steels at the unbending stage is poor during continuous casting. 25ppm boron bearing steel was examined in order to study the effect of cooling rate on the hot ductility. Since the rapid cooling in the corner of this slab during the continuous casting lead to as corner cracking, hot tensile test applied to the different cooling rate was taken into account. The results revealed that increasing cooling rate deteriorate hot ductility of B bearing steel. Low hot ductility of B bearing steel is associated with intergranular fracture along the austenite grain boundaries. Rod-like BN precipitates co-precipitated with MnS were formed along austenite grain boundaries when the cooling rate was increased. Intergranular fracture of B bearing steel is caused by the formation and growth of grain boundary cavities which nucleate at grain boundary precipitates of BN+MnS.

11:10 AM

Fracture Toughness of Cast Stainless Steels for Shield Module Applications: *Mikhail Sokolov*[1]; Randy Nanstad[1]; Jeremy Busby[1]; [1]Oak Ridge National Laboratory

Cast austenitic stainless steels are under consideration for structural components of the International Tokomak Experimental Reactor (ITER). An improved cast stainless steel has been developed as a substitute for wrought stainless steel for shield module applications. The improved castings were developed based on the commercially available CF3M specification. The modifications utilize combinations of Mn and N, which result in significant increases in strength. Additionally, two other alloys have been developed with enhanced solid solution strengthening by virtue of Cu and W additions to increase strength. The developmental process leading to these improved cast stainless steel alloys will be briefly reviewed, but the major focus of this paper will be the fracture toughness and other mechanical properties of at least two of the developmental alloys. Comparisons of these properties will be made with standard cast stainless steel. Metallography and scanning electron fractography of the tested steels will also be discussed.

11:30 AM

Impact of Sensitization on the Fracture Toughness of Austenitic Stainless Steel: *Swati Ghosh*[1]; Kalyan Ray[1]; Vivekananda Kain[2]; [1]Indian Institute of Technology; [2]Bhaba Atomic Research Center

This investigation aims to illustrate how degree of sensitization of a stainless steel affects its fracture toughness, estimated by a simplified technique. Solutionized nuclear grade 304LN stainless steel were subjected to sensitization treatment at 1023 K for different time periods in the range 1-100 hr. Corrosion resistance of the differently heat-treated samples were assessed by oxalic acid-etch and electrochemical potentiokinetic reactivation tests. The fracture toughness of the heat treated specimens was determined by a recently developed procedure based on Ball Indentation principle and validated by standard J-integral tests. The results reveal that the fracture toughness of stainless steels gets significantly deteriorated by sensitization. The supplementary examination of microstructure, grain size and hardness and tensile tests explains the cause of such a degradation in toughness as to be due to martensite formation and stress build up due to the grain boundary carbide precipitation.

Hael Mughrabi Honorary Symposium: Plasticity, Failure and Fatigue in Structural Materials - from Macro to Nano: Mechanical Properties of Ultrafine-Grained (UFG) Metals I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: K. Jimmy Hsia, University of Illinois; Mathias Göken, Universitaet Erlangen-Nuernberg; Tresa Pollock, University of Michigan - Ann Arbor; Pedro Dolabella Portella, Federal Institute for Materials Research and Testing; Neville Moody, Sandia National Laboratories

Wednesday AM Room: 386
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Neville Moody, Sandia National Laboratories; Cynthia Volkert, Forschungszentrum Karlsruhe

8:30 AM Invited

Effect of Nano-Scale Twins on Work Hardening Behaviors in Pure Copper: L. Lu[1]; X.H. Chen[1]; *K. Lu*[1]; [1]Chinese Academy of Sciences, Institute of Metal Research

Work hardening is one of the most important mechanical behaviors for engineering materials. As a general trend, work hardening always decreases accompanied with strengthening of metals. In this work, we will present a novel finding that introduction of a high density of ultrathin nano-scale twins in pure Cu, its work hardening exponent can be significantly elevated relative to that of the coarse-grained polycrystalline Cu. The highest work hardening exponent achieved is about 0.66, almost twice that of conventional Cu (0.35), when the twin/matrix lamellar spacing is as small as 4 nm. The effect of nano-scale twins on work hardening exponent and rate will be demonstrated. The extra-ordinary high work hardening originates from the ultrathin twins patterns that effectively suppress the dynamic recovery of dislocations and enhance the dislocation storage ability. The results indicate that work hardening of metals can be elevated by means of proper nanostructure design.

8:50 AM

Thermal Stability and Stabilization of Ultrafine-Grained Copper: *Yujiao Li*[1]; Wolfgang Blum[1]; [1]University of Erlangen-Nürnberg

Ultrafine-grained (UFG) pure Cu produced by ECAP was subjected to thermal and thermomechanical treatment. Transmission electron microscopy and mechanical testing were combined to test the stability of the UFG structure. During deformation at temperature up to 373 K the material keeps the properties of UFG material. Annealing at 403 K for 2.5 h destroys them, indicating (partial) recrystallization of the UFG structure. This process can be retarded by a thermomechanical treatment (TMT) consisting of a series of small deformation steps at increasing temperature. The TMT leads to growth of the cell size and decrease of flow stress. The cell size-stress relation equals the well-known stress dependence of the subgrain size in steady-state deformation. Tests of mechanical properties and structure observations indicate that the homogeneous coarsening of the cell structure during the TMT affects low- as well as high-angle boundary spacings and that the TMT significantly increases the resistance against recrystallization.

9:05 AM

Fracture Toughness and Fatigue Crack Propagation Measurements in Ultrafine Grained Iron and Nickel: *Anton Hohenwarter*[1]; Reinhard Pippan[1]; [1]Erich Schmid Institute Leoben

Nanocrystalline and Ultrafine-grained (UFG) materials are commonly known as materials with extraordinary mechanical properties. Due to the limited dimensions of HPT-deformed materials little is known about their fatigue properties whereas particulary this process delivers highly refined materials with little effort. In the framework of this study pure nickel and Armco-iron were subjected to High Pressure Torsion (HPT) and in combination with different heat treatments the grain-size was varied between approximately 100 nm and 10 µm.

Afterwards crack-propagation measurements with reference to different grain-sizes were conducted and monitored. It could be shown that there is a distinctive difference in the crack path with respect to the grain size, which may has an influence on the threshold of fatigue crack propagation as well. With respect to fracture toughness measurements in the UFG regime a quite good combination of toughness and hardness was observed.

9:20 AM Invited
Fretting Fatigue of Engineering Ceramics: Thomas Schalk[1]; Karl-Heinz Lang[1]; *Detlef Loehe*[1]; [1]Universitaet Karlsruhe (TH)

Fretting fatigue may appear in the contact zone between two components when they are exposed to cyclic loading and relative motion appears between the contact surfaces. This loading condition arises often in technical applications e.g. if forces are transmitted by form-fitting connections or at forming processes. To investigate the fretting fatigue behavior of engineering ceramics a new test rig was built up. The fretting fatigue loading is realized by the superposition of cyclic four point bending with local friction loading. The friction loading is achieved by the relative motion of a fretting pad which is well defined pressed on the tensile loaded surface of the four point bending specimen and moved by an electrodynamic actuator. With this testing system fretting fatigue experiments have been carried out at temperatures up to 900°C. The lifetime behavior of alumina and silicon nitride specimens will be presented and the arising damage characterized.

9:40 AM Invited
Fatigue Behavior of Railway Wheel Steels in the Very High Cycle Regime: Vadim Wagner[1]; Frank Walther[1]; *Dietmar Eifler*[1]; [1]University of Kaiserslautern

The fatigue behavior of railway wheel steels has been investigated in the very high cycle regime under stress control at a frequency of 200 Hz on servohydraulic and resonance testing systems. Specimens machined from the rim of original wheels for high-speed passenger traffic exhibit a distinct change in the slope of the S-N curve at about 2×10^6 cycles. High-precision temperature and electrical resistance measurements influenced by deformation-induced changes of the microstructure are used to characterize the actual fatigue state under VHCF conditions. SEM investigations show crack initiation at the specimen surface as well as inside the specimen cross-section. The physically based fatigue life calculation "PHYBAL" on the basis of generalized Morrow, Coffin-Manson and Basquin equations leads to enormous scientific and economic advantages. The S-N curve calculated with temperature and resistance data measured after 10^4 cycles, i.e. 50 seconds, matches very well with the experimentally determined one until 2×10^8 cycles.

10:00 AM Break

10:10 AM Invited
Deformation Physics and Ductility of Nanostructured Materials: *Yuntian Zhu*[1]; [1]North Carolina State University, Department of Materials Science and Engineering

Nanostructured materials usually have high strength but low ductility. To improve their ductility, we need to first understand their deformation physics. Past attempts to increase the ductility usually led to a decrease in strength. In this presentation, I'll first present the deformation physics in nanostructured materials and how they affect the ductility. I'll then present several strategies to tailor the structures and deformation physics of nanostructured materials with the aim of improving the ductility without sacrificing the strength. We have been able to simultaneously increase the ductility and strength using these strategies.

10:30 AM Invited
Length-Scale Effects on Fatigue of Thin Cu Films: *Cynthia Volkert*[1]; Dong Wang[2]; Oliver Kraft[2]; [1]University of Goettingen, Institute of Materials Physics and Forschungszentrum Karlsruhe, Institute of Materials Research II; [2]Forschungszentrum Karlsruhe, Institute of Materials Research II

Fatigue lives and fatigue damage morphology of Cu films (50 nm to 3 μm in thickness) on polyimide substrates has been investigated under high cycle loading. As film thickness and grain size are decreased, characteristic fatigue damage such as extended dislocation structures and extrusions disappear and are replaced by cracks along boundaries. The transition in damage morphology with length scale is accompanied by a marked improvement in fatigue resistance. It is proposed that the transition is due to the inhibition of dislocation nucleation and motion at small length scales, and that the cracking observed in the thinnest films is evidence of a new fatigue mechanism promoted by high stresses and high boundary density. In addition, the eventual saturation of the extrusion and crack densities under total strain control allows schemes for the use of thin films in damage tolerant applications.

10:50 AM Invited
Thermal and Mechanical Stability of Nano- and Ultra-Fine-Grained Materials: *Zhirui Wang*[1]; [1]University of Toronto

The involvement and the role of inter-crystalline constituents (such as high- and low-angle grain boundaries, twin boundaries and triple points) in submicron- and nano-meter grained materials could not be ignored when the materials' mechanical and physical properties are concerned. These constituents would also increase the internal energy of the material. In this report, effects of the energy associated to these constituents in electroformed Cu and CVD-deposited Ni materials will be discussed. The main thrust of the discussion will be two folds: First, experimental results will be presented to demonstrate the evolution of the ultra-fine-twin and nano-twin structures upon thermal activation, mechanical deformation; Bauschinger effect tests and DSE tests. The discussion will then be switched to correlate specifically the internal energy of such materials with their plastic deformation - strain hardening behaviour. Based on such analysis, a model has been established and would be presented which could be employed to predict the feature of work hardening in these materials.

11:10 AM
Inhomogeniety and Size of Microstructure - A New Paradigm in Understanding Deformation Mechanism of Nanocrystalline Metals: *Taher Saif*[1]; Jagannathan Rajagopalan[1]; Jong Han[1]; [1]University of Illinois at Urbana-Champaign

We will present experimental evidence showing that plastically deformed polycrystalline free standing thin metal films with average grain size of 50-100nm recover all of the plastic strain under macroscopic stress-free condition. This recovery shows three distinct activation energies. When the grain size is increased to 100-200nm, the films do not recover plastic strain, but they show strong Bauschinger effect during unloading, while still under tension. We hypothesize, inhomogeneities in the microstructure (grain size and orientation variations) create non uniform local stress fields during loading as relatively larger or favorably oriented grain deform plastically while smaller or unfavorably oriented grains accommodate the strain elastically. Upon unloading, the elastically deformed grains, in order to reduce their strain energy, induce reverse plasticity in the larger grains leading to time dependent strain recovery in films with 50-100 nm grain size, and Bauschinger effect in films with 100-200 nm grain size.

11:25 AM
Emergence of Glass-Like Plasticity in the Finest Nanocrystalline Metals: *Jason Trelewicz*[1]; Christopher Schuh[1]; [1]Massachusetts Institute of Technology

Nanocrystalline metals exhibit a variety of unusual mechanical properties as grain size is refined towards the amorphous limit, a widely discussed peculiarity being the breakdown of the Hall-Petch relation. In this work, we study the mechanical properties of nanocrystalline Ni-W alloys, which can be produced with grain sizes spanning both the Hall-Petch and Hall-Petch breakdown regimes. We show that not only the hardness, but also its scaling with strain rate and superimposed pressure undergoes an inflection at a finite grain size coinciding with the Hall-Petch breakdown. We also illustrate how these mutually consistent inflections are a consequence of the shift to amorphous-like deformation mechanisms, with inhomogeneous shear banding taking place in samples with the finest nanocrystalline grain sizes. We finally explore how the grain boundary relaxation state affects the deformation mechanisms across the entire Hall-Petch breakdown regime.

11:40 AM
Damage Evolution and Lifetime Prediction of Thermal Barrier Coatings during Cyclic Oxidation: *Tilmann Beck*[1]; Olena Trunova[1]; Roland Herzog[2]; Rolf Willi Steinbrech[1]; Lorenz Singheiser[1]; [1]Research Center Juelich; [2]MAN Turbo AG

Thermal barrier coatings (TBCs) are applied to combustion chambers and turbine blades of gas turbines to raise efficiency by increasing maximum service temperature. For power generation units, TBC-lifetimes up to 40000hrs must be

reached with high reliability. To fulfill this condition, in-depth understanding of microstructural damage as well as reliable tools for lifetime prediction are essential. Detailed damage analyses during cyclic oxidation of a plasma sprayed ZrO_2/8 wt-%Y_2O_3 – MCrAlY – CMSX-4 TBC system showed that formation and growth of an Al_2O_3 scale on the MCrAlY bondcoat plays a major role in the initiation of delamination cracks. The onset of stable crack propagation is strongly influenced by the TBC/bondcoat interface topography. Crack propagation itself can be described by fracture mechanics approaches. Based on the quantitative evaluation of damage evolution during cyclic oxidation with several dwell times at 1050°C, a phenomenological model for life prediction was developed and validated.

11:55 AM
The Effect of Environmental Exposure on the Dislocation Structure at the Fatigue Crack Wake: *YunJo Ro*[1]; Sean Agnew[1]; Richard Gangloff[1]; [1]University of Virginia

Focused ion beam (FIB) milling was used to produce samples for transmission electron microscopy which successfully revealed the dislocation structure within 1 µm of the fracture surface of an under-aged Al-Cu-Mg alloy tested in ultrahigh vacuum and high humidity air. Both different conditions exhibited a similar layer of dislocation cells just below the fracture surface, and the cell structure abruptly changed to localized slip bands away from the fracture surface. This abrupt change confirms the presence of the high strain gradient at the crack tip. In addition, the thickness of dislocation cell layer and the intensity of the slip bands observed in ultrahigh vacuum was much larger than those observed in high humidity air. Thus, the present results suggest that less plastic strain accumulation is required to propagate fatigue cracks at high humidity air relative to ultrahigh vacuum. Speculatively, the current study supports hydrogen enhanced decohesion.

12:10 PM
Microstructure-Sensitive, Mechanism-Based Plasticity Modeling of Materials: *Yoon Suk Choi*[1]; Dennis Dimiduk[2]; Triplicane Parthasarathy[1]; Michael Uchic[2]; Christopher Woodward[2]; [1]UES Inc; [2]US Air Force Research Laboratory

The intent of microstructure-sensitive mechanism-based constitutive models is the linking of selected attributes of internal features to their corresponding plastic flow response. The micro-mechanisms that govern plastic flow should be reflected in constitutive variables and functional forms. We have recently adopted this approach to develop a constitutive model for $L1_2$ intermetallics—the primary strengthening phase in superalloys—and found that the flow stress and its evolution (strain hardening) vary dramatically with the availability of mobile dislocations. Importantly, the empirically-observed flow response could not be modeled via conventional constitutive treatments wherein forest-like obstacles are assumed to govern plastic flow (typical for FCC crystals). We will discuss major differences in constitutive treatments between conventional FCC-like forest hardening and the mobile-density controlled $L1_2$ behavior, and the corresponding microstructural sensitivity for both constitutive representations. The potential for applying the mobile-density controlled treatment to model source-controlled plastic flow of different materials will also be discussed.

Hot and Cold Rolling Technology: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Ming Li, Alcoa Inc; Juergen Hirsch, Hydro Aluminium AS

Wednesday AM — Room: 297
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Juergen Hirsch, Hydro Aluminium AS; Ming Li, Alcoa Inc

8:30 AM Keynote
Process Chain Modelling of Al Sheet Production: *Kai Karhausen*[1]; [1]Hydro Aluminium

Through Process Modelling (TPM) is a keyword in research activities with a view to optimising a process chain according to desired product properties. State variables needs to be traced through a process chain. The linking of micro scale to macro scale models is required to feed realistic process information into the microstructural modules as well as to feed the process models with correct material behaviour. While the TPM can be validated directly on measured final or intermediate properties, it is often unclear to which extent the material behaviour affects the process performance. This presentation covers an exemplary TPM calculation of hot- and cold rolling passes as well as intermediate and final annealing. Variations are tested by computing extreme material conditions for single processing operations. This provides directives on which physical mechanisms and corresponding model combinations are required in specific process situations both with respect to accuracy and computational effort.

9:10 AM
Effects of Texture Evolution on Localized Deformation of AA2024: *Soondo Kweon*[1]; Armand Beaudoin[1]; Ming Li[2]; [1]University of Illinois at Urbana-Champaign; [2]Alcoa Technical Center

An experimental and computational study is presented to examine the effects of texture evolution on localized deformation behavior in the alloy AA 2024-O. Routine monotonic mechanical testing is complemented by a novel cyclic test procedure to develop flow properties at temperatures from -100°C to 495°C and strain rates from 10^-2/sec to 10^-5/sec. A coupon test, where the stress state is similar to the slab edge, is then pursued to understand localized deformation that is taking place in the rolling process. Details of the deformation in the coupon test are developed through use of a 3D finite element model, with material response derived from the material characterization noted above. The evolution of crystallographic texture is also used as a means of drawing interpretation of the deformation history in the region of localization. The relevance of this coupon test to hot rolling is discussed.

9:35 AM
The Role of Crystallographic Texture on the Performance of Flat Rolled Aluminum Products for Aerospace Applications: *Roberto Rioja*[1]; Cindie Giummarra[1]; Soonwuk Cheong[1]; [1]Alcoa

In this paper, the evolution of crystallographic texture during hot and cold rolling of aluminum alloys is reviewed and the major texture components present in recrystallized and unrecrystallized products are discussed. In particular, the effect of the "brass" texture component on the anisotropy of mechanical properties in Al-Li alloys and the methods used to control it are presented. The influence of texture on strength, toughness and thermal stability of plate products for lower wing applications is discussed. Finally, the role of recrystallized and unrecrystallized microstructures on strength, toughness and fatigue performance of aerospace sheet is presented in light of different crystallographic textures. It is concluded that the role of crystallographic texture is necessary to optimize damage tolerant properties of Al-Li alloys for use in aerospace applications.

10:00 AM
Texture Evolution during Rolling of Aluminium Alloys: *Juergen Hirsch*[1]; [1]Hydro Aluminium Deutschland GmbH

For many applications of Aluminium alloy sheet crystallographic textures have a strong influence on specific (anisotropic) properties like strength, formability and etching behaviour, e.t.c.. For these cases textures are intentionally induced – or at least controlled – by the specific rolling conditions. Examples are given for industrial Aluminium alloy sheet processing and applications where strongly textures give superior properties to fulfill specific requirements in special applications, like deep drawing of cans, strength effects in can ends and capacity in deep etched high purity Aluminium foil for high quality electrolytic capacitors. The required control of specific processing steps during hot and cold rolling is described. The principles of microstructure evolution during processing are presented that are needed to ensure optimum material quality and product performance.

10:25 AM Break

10:45 AM
Investigation of Longitudinal Surface Cracks and Effective Metallurgical Parameters in Hot Rolling of Steels: *Anis Ghaderi Namin*[1]; Seyyed Ahmad Jenabali Jahromi[1]; [1]Shiraz University

Longitudinal surface cracks are often found on continuously cast billets. Compared to transversal cracks, longitudinal cracks are usually deeper. Often

they are located in regions close to the centre line of billet surface. They found discontinuously in all of the billet surface, too. Many parameters can effect its generation such as FeS, Pinholes, Blowholes, trace elements (Cu, Sn), Inclusions and pre-heating condition of Hot Rolling. In this paper, we mentioned all of these parameters and the ways we can prevent longitudinal surface cracks. For example the optimum ratio between Fe and S is greater than 23.

11:10 AM

Optimising the Winding of Aluminium Strip: Kai Karhausen[1]; Cremer Albert[1]; *Michael Wimmer*[1]; Stefan Neumann[1]; [1]Hydro Aluminium

The production process of Al-strip consists of a number of continuous processing steps like rolling, annealing or various strip treatments, which are interlinked semi-continuously by transport and storage operations. For these operations the strip is usually wound into a coil. Thus, the entire production may contain numerous winding and unwinding operations. Although winding is not a processing step of its own, it can change the strip properties and the performance of subsequent processing steps. Mastering the winding process is therefore mandatory to achieve a high quality level. Typical associated problems are e.g. telescoping of strip layers, coil collapse under its weight, buckling of strip layers or sticking of strip layers after annealing. Furthermore, the stress state can result in shape deviations and flatness errors of the strip upon unwinding. In this paper modeling and optimisation of the winding processes will be treated on case studies from aluminium foil production.

11:35 AM

Innovations in Surface Quality Inspection as a Cornerstone for Production Optimization: *Uwe Knaak*[1]; Elisa Jannasch[1]; [1]Parsytec Computer GmbH

Surface defects impair the quality of the manufactured aluminium strip greatly; moreover, they may lead to strip breaks or even to equipment damage: less ability to deliver usable quality to customers, less throughput, and higher costs are the consequences. The solution of this problem is Surface Quality Inspection. Parsytec offers leading-edge solutions for an outstanding surface quality inspection: Parsytec Surface Inspection Systems deliver defect information, which can be turned into quality data for a most efficient production optimization. Parsytec's latest generation surface inspection solution - *espresso SI* - entails innovations for nearly all aspects of surface inspection: accessing inspection results via the web, employment of GBit Ethernet camera technology, and standard compact PCIs. The benefits for customers comprise of highest detection sensitivity and accelerated access to relevant quality data, combined with highest availability and easiest handling and maintainability of the systems. Furthermore, based on Parsytec's unrivalled experience, *espresso SI* also adds so-called "production decision intelligence" solutions, which transform inspection data to production benefit in selected applications. Production optimization can be achieved by combining surface quality data with all other available production and process data, as well as with customer and order information. One example, which will be presented here, is a productivity solution at coil slitting lines: The inspection system not only delivers information on surface defects, it supports the grading of complete coils, but also for individual stripes or coil segments on-line. This solution helps the aluminium producers to significantly improve yield and quality of their production. All in all, Surface Inspection by Parsytec serves two aims: increasing product quality, and turning surface quality information into production excellence.

12:00 PM

Effect of Surface Engineering on Roll Bonding of the AA2024 Al Clad Plate: Jiantao Liu[1]; Simon Sheu[1]; *Ming Li*[1]; Michael Karabin[1]; Robert Schultz[1]; [1]Alcoa Technical Center

Hot roll bonding of aluminum plate and sheet currently is the primary manufacturing method of fuselage skin sheet for aircraft and brazing sheet for automotive applications. Tremendous challenges and opportunities exist to improve quality and productivity of these products. This work explored the effects of macro- and micro-surface engineering on hot roll bonding process. It was found that micro-surface engineering yielded better bonding quality than macro-surface engineering did. The critical surface roughness of the core was about 0.58 μm below which bonding quality in terms of area contact and bonding energy can be significantly improved. However, no marked improvement was observed when the surface roughness was reduced further to 0.03 μm. Also, the oxide layer on the surface of the core and local deformation on the surface of liner played a very important role. Ultrasonic test and roller peel test were employed to assess the bonding strength and quality. The quantitative ultrasonic test results are in good agreement with the roller peel test results.

Magnesium Technology 2008: Advanced Magnesium Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Wednesday AM — Room: 291
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Eric Nyberg, Pacific Northwest National Laboratory

8:30 AM

Mg-6Zn/1.5%SiC Nanocomposites Fabricated by Ultrasonic Cavitation Based Solidification Processing: *Guoping Cao*[1]; Hongseok Choi[1]; Juan Oportus[1]; Hiromi Konishi[1]; Xiaochun Li[1]; [1]University of Wisconsin

Mg-6Zn/SiC nanocomposites were successfully fabricated by ultrasonic cavitation based dispersion of SiC nanoparticles in Mg-6Zn alloy melt. As compared to Mg-6Zn magnesium alloy matrix, the mechanical properties including tensile strength, yield strength and ductility of the Mg-6Zn/1.5% SiC nanocomposites were improved significantly. In the microstructure of Mg-6Zn/1.5% SiC nanocomposites, there are still some SiC micro-clusters, but in the areas free of micro-clusters, the SiC nanoparticles were dispersed very well. The grain size of Mg-6Zn was also refined considerably by adding 1.5% SiC nanoparticles. TEM study of the interface between SiC nanoparticles and magnesium matrix indicates a good bonding and no chemical reaction between SiC nanoparticles and Mg-6Zn matrix.

8:50 AM

Microstructure Evolution and Nucleation Kinetics of Rapidly Solidified Mg7ZnxY(0.55Zr) Alloys: Shijie Zhu[1]; *Shaokang Guan*[1]; Qing Yang[1]; Mei Zhang[1]; [1]Zhengzhou University, Department of Materials Science and Engineering

The effects of solidifying condition and Y content on the microstructural evolution, phase selection and nucleation kinetics of Mg7ZnxY(0.55Zr) alloys were investigated with OM, SEM, XRD, DSC and TEM. The microstructures of as-cast alloys are dendritic morphology including preliminary α-Mg, interdendritic α-Mg/I-phase(Mg_3YZn_6) eutectic and W-phase($Mg_3Y_2Zn_3$). Microstructures of the cross section of rapidly solidified alloys ribbons are composed of fine equiaxed, columnar grain and equiaxed region. The phase composition changes as the variation of alloying component and solidified condition. Rapidly solidified Mg7Zn3Y, Mg7Zn3Y0.55Zr alloys ribbons restrain the precipitation of (Mg_3YZn_6) phase. On the basis of heterogeneous nucleation theory, the connection between incubation period(τ) and temperature was induced and the curve of τ-T was obtained. The results show the second-phase of rapidly solidified Mg7ZnxY(0.55Zr) alloy nucleate firstly, which are concord with results of experiments.

9:10 AM

Synthesis-Structure Relationship in Cast Magnesium Periodic Cellular Materials: *Samson Ho*[1]; Lukas Bichler[2]; Glenn Hibbard[3]; C. Ravi Ravindran[2]; [1]Univeristy of Toronto; [2]Ryerson University; [3]University of Toronto

Periodic cellular metals (PCMs) are weight efficient hybrids of space and metal. PCMs can deliver higher specific strengths and stiffnesses compared to conventional open cell structures like metallic foams. This study investigates the fabrication of cast magnesium PCMs using the lost foam casting (LFC) method. LFC is a near-net-shape process in which molten metal displaces a polystyrene pre-form embedded in refractory sand. The effect of mould geometry on fillability was studied with different process parameters such as foam characteristic, pouring temperature, casting section thickness and vacuum level. Micro-hardness measurements and optical microscopy were used to characterize the as-cast structure.

9:30 AM
Symmetry Control during Thixoextrusion for AZ31 Mg Alloy: *Young Ok Yoon*[1]; Shae K. Kim[1]; [1]Korea Institute of Industrial Technology

AZ31 Mg alloy was widely used in transportation industries in the field of extrusion process. However, mechanical properties of extruded AZ31 Mg alloy with symmetry were dependent on the microstructure of the material and could be characterized by their inhomogeneity, grain size distribution and crystallographic texture. Therefore, this correlation between microstructure and mechanical behavior offered the possibility to improve the mechanical properties, e.g. through optimization of the material production process. Thixoextrusion could allow symmetry with extrusion direction and reduced extrusion pressure. The present study was to characterize thixoextruded AZ31 Mg alloy in terms of its symmetric behavior through the optical microscope, electron back scattering diffraction and mechanical test. The results of thixoextrusion experiments about symmetric properties and mechanical properties were compared with conventional extrusion results. The symmetric properties of thixoextruded AZ31 Mg alloy could be obtained through the control of thixoextrusion parameters.

9:50 AM Break

10:10 AM
The Behaviors of Second Phase and Hardness of Rheo-Die Cast AZ91D during Temperature Exposure: Yongfeng Jiang[1]; *Yefeng Bao*[1]; [1]Hohai University

Solid solution heat treatment of AZ91D die cast is performed at from 300°C to 420°C in from 2h to 32h to improve strength and result in maximum toughness and shock resistance, the behaviors of second phases and hardness are investigated by optical microscopy and microhardness meters. It is indicated t that at from 300°C to 450°C, the second phases precipitated are dispersed in variation of from matrix to grain boundary, which have effects on microhardness, from 2h to 32h at same temperature, the hardness is increased firstly to peak value, and then decrease; from 300°C to 450°C peak values and valley is in advance at from 300°C to 450°C; The grain size is also varied according to temperature and time.

10:30 AM
Fracture Toughness in Magnesium Alloys by Dispersion of Quasicrystalline Phase: *Somekawa Hidetoshi*[1]; Alok Singh[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

One of the most important criteria of safety and reliability of materials is the fracture toughness. However, the fracture toughness in magnesium alloys are lower than that in aluminum alloys. The dispersion of precipitates is generally the effective method to improving the mechanical properties in metallic material, but these particles easily become the origin of fracture. On the other hand, the characteristics of icosahedral phase are different from conventional crystalline phases, because this phase has qusi-periodic structure and very strong interface with matrix. This phase has been studied as the strengthening phase in magnesium alloys, but the effect of dispersion of quasi-crystalline phase on fracture toughness has not been investigated yet. In this study, the fracture toughness in Mg-Zn-RE alloys containing quasi-crystalline phase were examined, and the fracture mechanisms were observed in detail. The fracture toughness indicated higher values, and this phase pinned many dislocation movements and prevented void nucleation.

10:50 AM
Microstructure and Mechanical Properties of Magnesium Alloys Consolidated in the Solid State from Granules: Rick Lee[1]; Xiang Li[1]; Sibashish Mukherjee[1]; *Amit Ghosh*[1]; [1]University of Michigan

Consolidation from granular stock can offer a low cost approach for preparing bulk Mg alloys in the solid state, if many steps in the rolling of plates and sheets can be avoided as practiced by wrought Mg industry. Processing of solid state consolidated Mg alloys has been studied by employing a variety of processing methods. Severe deformation techniques have been applied to the alloys to produce fine grain microstructure and superplastic properties. This paper will discuss potential for achieving high mechanical properties in Mg Alloys and its composites. (Work supported by National Science Foundation under Grants 0314218 and 0544920).

Magnesium Technology 2008: Alloy Microstructure and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Wednesday AM Room: 292
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Eric Nyberg, Pacific Northwest National Laboratory; Alok Singh, National Institute for Materials Science

8:30 AM
An Assessment of High Pressure Die Cast Mg-Zn-Al Alloys: *Mark Easton*[1]; Trevor Abbott[2]; Jian-Feng Nie[1]; Gary Savage[3]; [1]Monash University; [2]Advanced Magnesium Technologies; [3]CSIRO

Most of the current commercial Mg high pressure die casting alloys are found in the Mg-Al-Zn system, mainly based on Mg-Al alloys with small additions of Zn. More recently some alloys containing higher Zn contents have been proposed mainly for alloys with higher creep resistance with some success. This paper investigates the mechanical properties (both creep and quasi-static tensile properties) and the castability of a range of Mg-Zn-Al alloys and indicates regions where each of the properties can be optimised. The yield strength of the alloys can be related by a simple regression relationship with the aluminium and zinc contents. Above approximately 8wt% alloying elements the ductility began to decrease, with alloys with very high alloy contents having little if any ductility. The findings confirmed the castability diagram presented by Foerster in the 1970's although the data shown here presents a continuous variation of properties with alloy content rather than the discrete categories for castable and non-castable alloys as was previously described

8:50 AM
Effect of Composition and Cooling Rate on the Beta Phase Formation in Mg-Al Alloys: *Suresh Sundarraj*[1]; Mridula Bharadwaj[1]; Shashank Tiwari[1]; [1]General Motors Corporation

It is well known that the corrosion properties of Mg-Al alloys like the AM and AZ series are significantly affected by the formation of the microstructural beta phase ($Mg_{17}Al_{12}$) which forms during casting of these alloys as a result of microsegregation. In this paper we have studied the effect of the alloy composition and processing conditions which affect the amount and morphology of the beta phase formation. Binary Mg-Al alloys with varying amounts of Al content along with varying cooling conditions to mimic the sand, permanent mold, and die casting cooling rates were evaluated using this microsegregation model. The model results of the amount of beta have been verified with the in-house experimental data. Finally, a correlation was established between the corrosion behavior in these alloys with the model predictions.

9:10 AM
Effects of Composition on the Microstructure and Mechanical Properties of Mg–Al–Zn Alloys: Songmao Liang[1]; Yuequn Ma[1]; Rongshi Chen[1]; *Enhou Han*[1]; [1]Institute of Metal Research

For exploiting mechanical properties potential of low-cost Mg–Al–Zn casting magnesium alloys, the as-cast microstructure and mechanical properties of permanent-mould cast Mg–Al–Zn alloys with typical compositions within castable domain of the intermediate aluminum and zinc contents were investigated. This study disclosed the evolution rules of secondary phase type with variation in Zn/Al mass ratio. Within this domain, secondary phase changed from γ phase to ϕ phase, τ phase and quasi-crystalline phase (I phase) with the Zn/Al mass ratio increasing. By studying cooling curves acquired by thermal analysis technique, the forming temperatures of secondary phases were also determined. Mechanical property tests indicated that increasing the aluminum and/or zinc content, the Y.T.S (Yield Tensile Strength) increased and ductility decreased, while the U.T.S. (Ultimate Tensile Strength) firstly increased and then

decreased. Both the U.T.S. and elongation of the alloy with optimum composition are superior to those of AZ91 alloy prepared at the same conditions.

9:30 AM
Phase Relations, Formation and Morphologies in Mg-Zn-RE (RE=Y, Rare Earth) Alloys: *Alok Singh*[1]; [1]National Institute for Materials Science

Lattices of ternary and Mg-Zn binary phases are related to the matrix Mg phase through its basal plane. This characterizes the structural relationships among the phases and determines the orientation relationships formed by nucleation and growth of these phases over each other and the morphologies evolved, which finally affect strengthening. Orientation relationships and morphologies of icosahedral phase with ternary phases cubic W and pre-eutectic hexagonal H are described. A most important phase in this system is the binary monoclinic Mg_4Zn_7 which often nucleates over the icosahedral phase. In Mg-matrix this phase occurs in form of rods parallel to hexagonal axis, commonly known as β_1' phase, until now believed to be of structure of hexagonal $MgZn_2$ phase and the primary hardening phase in Mg-Zn alloys. Constraints to form low energy interfaces with the matrix produces a domain structure inside the rods, with four different orientations.

9:50 AM
Microstructure Development and Mechanical Behavior in High Pressure Die Cast Magnesium-Aluminum Alloys: *A. Nagasekhar*[1]; Carlos Caceres[1]; Mark Easton[2]; [1]University of Queensland; [2]Monash University

Binary Mg-Al alloys with varying content of aluminium from 0.5 to 12 mass % have been studied. The proof stress increase in two steps whereas the ductility exhibits two correlated stepwise drops, as the aluminum content increases. The first increase in strength, and attendant drop in ductility, is observed between 4 and 5 mass% Al. The second stepwise change is observed between 10 and 12 mass% Al. These effects are connected with well defined changes in the microstructure: at 4 mass% a dispersion of beta-phase intermetallic particles appears in the core region and a closed cell structure develops near the surface; at 12 mass% Al, the increased volume fraction of the beta-phase intermetallics extends the interconnected network of intermetallics to include the core region as well. The micromechanics of the strengthening and decreased ductility are discussed.

10:10 AM Break

10:30 AM
Stabilization of Mg-Ca-Zn Alloys by Zr Additons: *Dmitry Shepelev*[1]; Anton Gorny[1]; Menahem Bamberger[1]; Alexander Katsman[1]; [1]Technion - Israel Institute of Technology

The microstructure and mechanical properties of Mg-Ca-Zn alloys with Zr additions were investigated in as-cast and heat treated conditions. Different concentrations of Zr (0.5 wt% and 1.0 wt%) were used to refine the grain structure. The alloys were exposed to solution treatment at 410°C for 96 hours followed by aging at 175°C for up to 96 hours. The microstructure obtained after heat treatment had equiaxed grains with evenly distributed binary phases, Mg_2Ca and Zn_2Zr. The ternary $Mg_2Ca_6Zn_3$ phase was identified at grain boundaries surrounded by precipitate depleted zones. Thermal stability of the Zr-modified alloys was examined by microhardness measurements conducted after prolonged exposures of the alloys to elevated temperatures. It was found that Zr is a structure stabilizing factor. Its influence was associated with formation of Zr-containing phases that do not undergo coarsening at elevated temperatures used (due to low diffusivity of Zr).

10:50 AM
The Effects of Alloying Elements on Microstructures and Mechanical Properties of Mg-MM(-Sn) Alloys: *Hyun Kyu Lim*[1]; Do Hyung Kim[1]; Ju Youn Lee[1]; Won Tae Kim[2]; Do Hyang Kim[1]; [1]Yonsei University; [2]Cheongju University

In the present study, the effects of Sn addition on the microstructure and mechanical properties of Mg-MM (MM: misch metal) have been investigated. To optimize the mechanical properties of Mg-MM-Sn alloys, the effects of addition of Al or Zn have been further investigated. The secondary solidification phase in Mg-MM alloy changes dramatically with addition of Sn forming small rod-shaped phase. Although the strength of alloy is decreased with addition of Sn, the ductility is improved in Mg-rich Mg-MM-Sn alloys. To enhance the mechanical properties, a small amount of Al or Zn has been added in Mg-2MM-2Sn alloy. The rollability and mechanical properties are improved with addition of Al or Zn. From the result of conical cup test at 200°C, it has been found that the addition of alloying elements improved the formability of Mg-MM-Sn alloys.

11:10 AM
A Study on the Microstructure of AS-Cast and Homogenized Mg-Gd-Nd-Zr Alloys: Kaiyun Zheng[1]; *Jie Dong*[1]; Xiaoqin Zeng[1]; Wenjiang Ding[1]; [1]Shanghai Jiao Tong University

Microstructure of Mg-xGd-2Nd-0.5Zr (x=6, 8, 11, Wt.%) alloys in as-cast and homogenized condition were investigated in this paper, using optical microscopy, scanning electron microscopy, transmission electron microscopy, energy dispersive X-ray spectrometry and X-ray diffraction. It was observed that all the as-cast alloys contained primary a-Mg solid solution, eutectic structures, cuboid-shaped phases, rod-like precipitates and Zr-rich clusters. The eutectic phase in Mg-11Gd-2Nd-0.5Zr was characterized to be of Mg5Gd prototype with stoichiometry near Mg5(NdxGd1-x, x˜0.43). Microstructural evolution of the alloys during homogenization can be divided into three coinstantaneous processes as follows: dissolving of eutectic phases, growth of cuboid-shaped phases and coarsening of grains. After appropriate homogenization, the alloys exhibit microstructure with little eutectic phases, small amount of cuboid-shaped phases and relatively fine grains, corresponding to excellent mechanical properties. The cuboid-shaped phase with a FCC crystal structure (a˜0.56nm) was identified as a Gd-based ternary Gd-Nd-Mg phase.

11:30 AM
A Comparative Study on the Solution Treatment of Mg-Al and Mg-Al-Ca Alloys: *Lihong Han*[1]; Henry Hu[1]; Derek Northwood[1]; [1]University of Windsor

The solution treatment of permanent mold (PM) cast Mg- 5wt.% Al (AM50) and Mg- 5 wt.% Al- 2wt.% Ca (AC52) alloys was studied by Differential Scanning Calorimetry (DSC), microhardness and quantitative metallography. DSC showed that the temperature of the solution treatment was dependent on the type of the eutectic phases present in the alloys. The measurement of changes in microhardness with solution treatment time showed that it took about 10 hours for the AC52 alloy to reach a minimum value of microhardness while only 3 hours was required for the AM50 alloy. Quantitative metallography showed that the Mg17Al12 phase in the AM50 alloy dissolved quickly into the matrix at an early stage of solution treatment compared with the Ca-containing eutectics in the AC52 alloy. These observations on microstructural evolution and microhardness change suggest that these are different dissolution kinetics for the eutectic phases during solution treatment of the two alloys.

Materials for Infrastructure: Building Bridges in the Global Community: Session II

Sponsored by: The Minerals, Metals and Materials Society, Indian Institute of Metals, Chinese Society for Metals
Program Organizer: Brajendra Mishra, Colorado School of Mines

Wednesday AM — Room: 272
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Subodh Das, Secat Inc

8:30 AM Invited
Steel for Bridges: *Virendra Kumar Mehta*[1]; [1]Central Marketing Organisation, SAIL

Abstract not available.

9:00 AM Invited
The Development of High Performance Bridge Steel in China: *Chengjia Shang*[1]; Huaxin Hou[2]; Aimin Guo[3]; Xinlai He[1]; [1]School of Materials Science and Engineering, University of Science and Technology Beijing; [2]Anshan Iron and Steel (Group) Corporation; [3]Wuhan Iron and Steel (Group) Corporation

In the past decade, twenty six bridges have been constructed across Chinese longest river, Yangtze River. In the next decade, more then twenty bridges will be

built across it. The first bridge on this river is Wuhan Bridge which established in 1950's, the steel, which was imported from USSR is Q235 grade; the second Yangtze river bridge is Nanjing Bridge which was constructed by domestic produced steel, Q345(16Mn, made by An-Steel). From then on, Q420 grade 15Mn-V-N produced by An-Steel was used to construct Jiujing Bridge in Jiangxi province with main span of 216m in 1990's, despite the disadvantage of welding ability; 14Mn-Nb (370MPa grade) was developed by WISCO to construct Wuhu Bridge with main span of 312m in early 2000's. These years, high performance bridge steel is urgently demanded by super bridge projects in China. A serial of 420,460,500 and 690MPa grade low carbon microallying steel have been developed for steel bridge constructing. From the microalloying approach, low carbon or ultra-low carbon are accepted; Nb,V,Ti microalloys are used to fine the grain size, affect the TMCP and strengthen the matrix. On the other hand, the modern metallurgical technologies are applied and the steel achieves economy grade cleanness. The new developed bridge steels can achieve high strength, high toughness, good weldingability and weathering resistance. The thickness can reach 80mm by TMCP. Last year, 12000 tons of high performance 420MPa grade plate steel had been used to construct Chongqing Yangtze River Bridge. The 460MPa grade steel has been accepted to design and construct the express railway bridge across Yangtze River in Nanjing. With the demanding and developing of high strength bridge steel, the employ performance, reliability should be paid more attention and the structural design theory and specification should also be improved.

9:30 AM Invited
Materials for Infrastructural Applications: *Jonathan Martin*[1]; [1]National Institute of Standards and Technology

Recent natural (e.g., hurricanes Katrina and Rita) and anthromorphic (e.g., the World Trade Centers and Boston's Big Dig) disasters have highlighted the important roles that construction materials play in catastrophic events. Construction materials also are playing an important role in the nation's aging and overburdened infrastructure. For example, the American Society of Civil Engineers (ASCE), in their 2005 Infrastructure Report Card, projects that the cost of repairing the nation's infrastructure back to good condition has escalated to over $1.6 trillion dollars. To meet these challenges, advanced construction materials are needed. These construction materials will not only be expected to have improved initial and long-term performance properties (i.e., have longer service lives and longer mean times between repairs), but also, in many applications, will be expected to perform simultaneously multiple functions, e.g., enhancing the appearance of a structure while acting as a biocidal surface. In this presentation, field-observed construction material failures will be shown, and recent advances in the performance of construction materials will be documented.

10:00 AM Break

10:20 AM Invited
Steel for Buildings: *Supriya Das Gupta*[1]; [1]MN Dastur and Company Pvt Ltd
Abstract not available.

10:50 AM Invited
Materials for Oil and Gas Transport: *Russell D. Kane*[1]; [1]Honeywell International

The demand on materials for transporting oil, natural gas, and other fluids, including hydrogen, ethanol, etc. is severe in terms of material strength, corrosion resistance, durability and maintainability. Lower temperatures and higher pressures are key factors in enhancing this demand on materials. The status and potential for performance ability of current materials available for transporting the fluids over long distances shall be reviewed. The research and development efforts for new ferrous and non-ferrous materials, including coating options, will be discussed. The strength and corrosion performance challenges will be highlighted.

11:20 AM Invited
High Strength Corrosion Resistant Fe/9Cr/0.1C Lath Martensitic Steels for Concrete Reinforcement: *Greg Kusinski*[1]; Salem Faza[2]; [1]Clemson University; [2]MMFX Technologies Corporation

One of the major problems facing the concrete construction industry today is the corrosion of steel reinforcement in concrete structures. In addition, congestion of reinforcement in heavily reinforced elements (such as mat foundations of high-rise buildings or confinement steel in columns of high-rises in seismic zones) is a critical constructability problem. In this paper, high-strength (YS > 100 ksi and 120 ksi) corrosion resistant steel, namely Microcomposite 9Cr steel, is presented from the aspect of alloy development, property characterization, and code implementation. We present principles of steel alloy design to produce nanophase multilayered structure composed of dislocated lath martensite separated by sheets of retained austenite. Such microstructure, which is free from interlath carbides, has an optimal combination of strength and corrosion resistance. A summary of a wide range of corrosion testing in saline environments carried out by many organizations including the Federal Highway Administration and many state Departments of Transportation (DOT's), will be presented to show that the MMFX-9Cr are a cost effective solution to offer 100 year design life of concrete structures. The steels conform to the new ASTM A1035 specification and their implementation into ACI 318-08 building code will be discussed. Structural engineering calculations will be presented to show that in certain cases it is possible to achieve up 50% steel reduction as compared with conventional grade 60 ksi steel. Furthermore, these principles of high strength design could also be applied for transportation and other applications to achieve significant cost savings.

Materials in Clean Power Systems III: Fuel Cells, Hydrogen-, and Clean Coal-Based Technologies: Metallic Interconnects and Sealing in SOFCs

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizers: Zhenguo "Gary" Yang, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Ayyakkannu Manivannan, National Energy Technology Laboratory

Wednesday AM — Room: 392
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Paul Jablonski, NETL; Douglas Ivey, University of Alberta

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8:30 AM Invited
Effects of Cell Operating Conditions on Degradation by Chromium: Terry Cruse[1]; Michael Krumpelt[1]; *Brian Ingram*[1]; Gang Chen[1]; Shanling Wang[2]; Paul Salvador[2]; [1]Argonne National Laboratory; [2]Carnegie Mellon University

This research examines how the performance of solid oxides fuel cells (SOFC) degrades in the presence stainless steel bipolar plates as a result of chromium migration. The cells examined were commercially supplied anode supported cells with Ni/8YSZ cermet anodes, 8YSZ electrolytes, and LSM/8YSZ active cathodes. They were operated under varying temperatures and current densities, which influenced operating cell potential, degradation rate and chromium deposition for the cells. Several techniques were used to examine the cells after operation (e.g., 500 hours): transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive spectroscopy (with both TEM and SEM), and high energy x-ray analysis at Argonne National Laboratory's Advance Photon Source. This research will provide information and a better understanding of how degradation of SOFC's by chromium occurs which will lead to a minimization of deleterious interactions between the SOFC cathode and chromium.

9:00 AM
Development of Multi-Component Air Braze Filler Metals for High-Temperature Electrochemical Application: *K. Scott Weil*[1]; Jin Kim[1]; Jens Darsell[1]; Jung Pyung Choi[1]; John Hardy[1]; [1]Pacific Northwest National Laboratory

A silver-based joining technique referred to as reactive air brazing (RAB) has been recently developed for joining high temperature structural ceramic components of the type used in high-temperature electrochemical devices. In prior work, it was found that additions of CuO to silver have a significant effect on the wettability and joint strength characteristics of the resulting braze on

polycrystalline alumina substrates. More recently it has been found that various ternary additions can further improve the wetting characteristics of these filler metals and/or increase their potential operating temperatures. This presentation will describe the use of several different types of additions, including: (1) a titania dopant added to increase wetting, (2) a palladium alloying agent added to increase use temperature, and (3) inert particulate such as alumina and yttria-stabilized zirconia powder/fiber added to increase joint strength.

9:25 AM
Effects of Y, Co, and Y/Co Coatings on the Oxidation Kinetics of Ferritic Stainless Steels for SOFC Interconnect Applications: *Seong-Hwan Kim*[1]; Joo-Youl Huh[1]; Jae-Ho Jun[2]; Do-Hyeong Kim[2]; Joong-Hwan Jun[2]; [1]Korea University; [2]RIST

Ferritic stainless steels are considered as promising interconnect materials for SOFCs, owing to their low cost and good thermo-mechanical compatibility with other components in SOFCs. However, the continuous growth of poorly conducting oxide scales in the SOFC operating environment, which degrades the stack performance, is the major challenges to be overcome for the used of ferritic stainless steels as interconnect materials. In this study, we have investigated the effects of Y, Co and Y/Co coatings on the oxidation behavior of STS444 and Crofer 22 APU. After oxidation for times up to 1000 h in air at 800°C, uncoated and coated samples were characterized using thermogravimetry (TG), X-ray diffractometry (XRD), scanning electron microscopy (SEM), glow discharge spectrometry (GDS), and area specific resistance (ASR) measurements. In the presentation, the correlation between the oxidation behavior and electrical properties of oxide scales will be discussed in terms of the redistribution of the coating elements.

9:45 AM
Niobium-Clad Stainless Steel for Bipolar Plate Material of PEMFC: *Sung-tae Hong*[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

Niobium (Nb)-clad 304L stainless steel (SS) is currently under consideration for use as a bipolar plate material in polymer electrolyte membrane fuel cell (PEMFC) stacks. The Nb-clad SS was fabricated via roll bonding. Results from mechanical testing suggest that annealing treatment after roll bonding may be necessary to improve the ductility and reduce the springback of the material. Microstructural analysis of the specimens indicates that two failure conditions can potentially arise, dependent on the thermomechanical condition of the material. In the as-rolled condition, failure initiates via fracture through the Nb cladding. In the annealed specimens, failure can occur by brittle fracture of an interfacial intermetallic layer that forms during the annealing treatment. This generates a series of crack-induced pores along the interface between the Nb cladding and the SS core, which eventually leads to ductile failure of the Nb cladding via localized necking.

10:10 AM Break

10:25 AM Invited
Observation of Metal Nanoparticles in Oxide Scale by Synchrotron X-Ray Nano-Beam: *Zuotao Zeng*[1]; Ken Natesan[1]; Zhonghou Cai[1]; [1]Argonne National Laboratory

Oxide scale is generally considered to consist of oxides. However, our recent nano-X-ray beam analysis indicated the scale to be a mixture of oxides and metal particles. This result can have broad influence on several research areas. For example, how carbon diffuses through oxide scale and leads to carburization and metal dusting have not been well understood. If metal particles are present in the oxide scale, carbon can diffuse through the channels that established by metal particles in the scales. Currently, solid oxide fuel cell requires low resistivity for oxide scales that develop on alloys for metallic interconnects. If metal particles are present in oxide scale, it may lead to electrical short and greatly reduce the area resistance of metallic interconnects. On the other hand, if we can prevent the formation of metal particles in oxide scale, carburization can be retarded by elimination of the carbon transfer channels.

10:55 AM
Selecting the Appropriate Air Brazing Conditions for Joining Reactive Substrates: *K. Scott Weil*[1]; Jens Darsell[1]; John Hardy[1]; [1]Pacific Northwest National Laboratory

Air brazing technology is being developed at Pacific Northwest National Laboratory as means of joining and/or hemetically sealing metal and ceramic components used in high-temperature electrochemical devices such as solid oxide fuel cells, oxygen and hydrogen gas concentrators, and sensors. On several occasions, it has found that when molten, the conventional air braze filler metal (Ag-CuO) will react excessively with a given substrate; cobalt-containing oxide or metal alloys tend to be partcicularly reactive. We have developed several processing strategies that mitigate the extent of interfacial reaction and thereby avoid the associated loss in joint strength typically observed. Select examples and applications will be discussed in this presentation.

11:20 AM Invited
The Operating Characteristics of Diesel-Driven SOFC: Inyong Kang[1]; Yunhyeok Kang[1]; Sangho Yoon[1]; Gyujong Bae[1]; *Joongmyeon Bae*[1]; [1]Korea Advanced Institute of Science and Technology

The flowrates and composition of the reformate produced by the diesel autothermal reformer were changed according to the reforming conditions, such as GHSV, H2O/C and O2/C. Therefore, the reforming conditions directly affect the SOFC performance. And, although the reforming performance at a certain point is known, the performance of the autothermal reformer and SOFC often degrades steadily due to carbon deposition from lighter hydrocarbons. Fortunately, CH4 did not cause any problems when an appropriate H2O/C was determined for direct internal reforming(DIR) with a Ni-based anode. But other light hydrocarbons, such as normal butane(n-C4H10), caused severe carbon deposition, even at experimentally-proven carbon free conditions for CH4-DIR. Therefore, the maintenance of high diesel conversions is very important for the longer-term performance of the diesel-driven SOFC system. In this study, the SOFC single stacks and the 5-cell stack were provided by the Korea Electric Power Research Institute(KEPRI).

11:45 AM
Comparison of High Velocity and Quasi-Static Forming of Sheet Metals: *Kristin Banik*[1]; Scott Golowin[1]; John Bradley[2]; Keith Newman[2]; Steve Hatkevich[3]; Glenn Daehn[1]; [1]Ohio State University; [2]General Motors Corporation; [3]American Trim Corporation

The ability to manufacture sheet metal components of complex shape by stamping is limited by the ductility limits. In this study, the formability of a variety of sheet metals were evaluated both quasi-statically and with high-velocity forming. A single-sided die with narrow serpentine channels was used with a high pressure urethane pad to establish baseline quasi-static conditions. The austenitic stainless steel and Ni-based alloy exhibited adequate formability, whereas the other materials failed by splitting. Using the same die, the materials were subjected to high-velocity electromagnetic forming. This resulted in enhanced ductility. Ti and ferritic stainless steel sheets could now be formed without splitting. All samples were evaluated for hardness, microstructure, deformation depth and other properties. The improvement in formability may be due to the high strain rates (~105 s-1) or the largely compressive stress state on impact. Efforts to make high velocity processes suitable for commercial use will be discussed.

Materials Informatics: Enabling Integration of Modeling and Experiments in Materials Science: Informatics and Materials Theory and Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Krishna Rajan, Iowa State University

Wednesday AM Room: 271
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: To Be Announced

8:30 AM
Applying Informatics to Semi-Empirical First Principles Calculations: *S. Broderick*[1]; H. Aourag[2]; Krishna Rajan[1]; [1]Iowa State University; [2]Tlemcen University

In this presentation we discuss how statistical learning techniques can be used to augment more classical approaches to computational based design of materials. The role of data mining to identify dominant parameters influencing phase stability calculations is demonstrated. The use of such informatics based techniques to accelerate the computational approaches for first principle calculations is discussed. NSF International Materials Institute: Combinatorial Sciences and Materials Informatics Collaboratory (CoSMIC-IMI).

8:55 AM
Applying Information Theory to Crystal Structure Design: *Chang Kong*[1]; Changwon Suh[1]; S. Arapan[2]; R. Ahuja[2]; Krishna Rajan[1]; [1]Iowa State University; [2]Uppsala University

Data mining and informatics are becoming one of the main areas in the computational methodologies for the materials design. The classification and visualization of data structure by applying appropriate criteria enable us to readily extract useful knowledge through the discovery of the trends within the database. The concepts of information theory, which originally started from the telecommunication systems, have been widely used in various fields including materials science especially at the single atom or molecule level. However, there has rarely been a scientific search of their application for the analysis of crystal-level, yet atomically defined, materials. In this presentation, we provide the examples of the analysis using information theory for the multivariate data of inorganic crystal structures. The application of the information theory is mainly focused on the classification of structure data and the evaluation of the used criteria, as the part of the prediction procedure of inorganic crystal-structure. NSF-International-Materials-Institute:-Combinatorial-Sciences-and-Materials-Informatics-Collaboratory-(CoSMIC-IMI).

9:20 AM
Data Mining, First-Principles Calculations and Thermodynamic Modeling for Ir-W Alloys: *Tao Wang*[1]; Krishna Rajan[1]; [1]Iowa State University

Ir-base alloy has been found to be a promising material for high-temperature applications. Most studies on Ir-base systems have suffered from limited and inaccurate thermodynamic information. Our work here has focused on the construction of thermodynamic database for the Ir-W system using first-principles approach, thermodynamic modeling and data mining techniques. We have found that a combination of data mining, first-principles calculations and thermodynamic modeling can not only provide a reliable thermodynamic database but also help us to understand the role of key electronic factors.

9:40 AM
Informatics Aided Structure Maps: *C. Suh*[1]; S. Iwata[2]; Krishna Rajan[1]; [1]Iowa State University; [2]University of Tokyo, Research into Artifacts, Center for Engineering

It is shown how conventional structure maps of spinel nitrides are created from data mining techniques. It is shown how data driven analysis of structural data coupled to property calculations can also help establish structure-property relationships unlike traditional structure maps. Examples are shown for nitrides and how these results compare with classical bi-variate mapping of structure classification. The value of informatics as a powerful tool for developing new structure maps and their role in materials design is discussed. NSF International Materials Institute: Combinatorial Sciences and Materials Informatics Collaboratory (CoSMIC-IMI).

10:05 AM
Predicting Multi-Component Crystal Structures Using Data Mining and Quantum Mechanics: *Geoffroy Hautier*[1]; Chris Fischer[1]; Gerbrand Ceder[1]; [1]Massachusetts Institute of Technology

Crystal structure prediction is a fundamental problem in computational materials science. Although material property predictions can now be reliably addressed ab initio, for predictions to be relevant, they must be performed on the stable crystal structure of the compound. Predicting crystal structure is therefore an essential step in any computational materials design scheme. We have developed a method to predict the structure of multi-component systems using data mining and quantum mechanics. We demonstrate an algorithm that detects similarities between crystal structure in order to find the structure prototypes in any database. Correlations between prototypes can then be extracted from the prototyped database to assess the probability for a compound to form in a given crystal structure. The results of our prototyping and data mining algorithm on the Inorganic Crystal Structure Database (ICSD) are discussed.

10:30 AM
Representation of Complex Microstructures: A Data Perspective: *Richard Le Sar*[1]; [1]Iowa State University

One of the challenges facing those seeking to apply materials informatics methods is that properties of materials often depend critically on the three-dimensional distribution of interfaces, defects, impurities, etc., i.e., properties depend on the microstructures of the materials. The key challenge is to represent those microstructures in a way that captures the essential structural features yet is simple enough for inclusion in a searchable materials database. In this talk we will discuss ways to represent the microstructures of systems of dislocations. We will base the discussion on results from discrete dislocation simulations and will discuss various scaling relations, cluster expansions, correlation functions, etc. that have been used to capture aspects of the dislocation microstructures.

11:05 AM
Superalloy Grain Growth Model Development through Simulation-Experimentation Integration: *Eric Payton*[1]; Gang Wang[1]; Ning Ma[1]; Yunzhi Wang[1]; Michael Mills[1]; [1]Ohio State University

Grain size is an important input for prediction of mechanical behavior of structural materials. Accurate physics-based modeling of grain growth is expected to accelerate the insertion of advanced alloys. During thermomechanical processing, the variation of distributions and volume fractions of pinning particles with thermal exposure control grain growth behavior and hence determine the grain size and distribution. Various electron microscopy techniques and digital image processing methods are being used to characterize the grain size distribution and populations of pinning phases in Ni-base superalloys during thermal exposure. Characterization results are being fed into phase-field and mean-field simulations. Simulation results are being compared to experimental observations, then used to elucidate the microstructural parameters for which additional characterization is needed to increase accuracy and generality of a model for predicting grain size during heat treatment. The established feedback loop between experimentation and simulation has enabled focused experimentation and development of fast-acting predictive models.

11:30 AM
Pandat Software for Materials Property Simulation of Multi-Component Systems Linking Calphad with Materials Informatics: *Weisheng Cao*[1]; Shuanglin Chen[1]; Fan Zhang[1]; Kaisheng Wu[1]; Ying Yang[1]; Y. Chang[2]; [1]CompuTherm LLC; [2]University of Wisconsin

The Pandat, integrating PanEngine, PanOptimizer and PanStar, bridges all the functionalities for thermodynamic calculation, property optimization, and precipitation simulation of multi-component systems based on Calphad approach. This software package, in combination with thermodynamic/kinetic/thermophysical databases, provides an integrated workspace for simulating materials properties of multi-component systems. The simulation results, which include phase equilibria, thermophysical properties, and microstructure related information, are critically needed in alloy design, selection of parameters for

fabrication steps such as heat treatment, prediction of performance, and failure analysis. In addition to the functionalities provided in Pandat, its calculation/optimization engines (PanEngine, PanStar and PanOptimizer) are built as shared libraries and enable their integrations with other models for wider applications in materials science and engineering. This provides a linkage of Calphad approach with materials informatics.

11:55 AM

Virtual Candidate Spaces: Developing Design Rules for Data-Driven Exploration: *Kim Ferris*[1]; Bobbie-Jo Webb-Robertson[1]; Dumont Jones[2]; [1]Pacific Northwest National Laboratory; [2]Proximate Technologies, LLC

Traditional approaches to materials development rely upon a data point by data point approach, becoming highly dependent upon prior art and variations of current topical materials. This presentation will discuss strategies for identification of candidate materials for gamma radiation detection as prototypical examples for more efficient virtual candidate exploration. An information-based approach has been applied to ranking the alkali/alkali earth halide series on their energy conversion efficiency and luminosity response. Relevant data are sparse (25% coverage), but sufficient to complete the forward mapping of materials properties through materials signatures. These mappings are used to explore the limits of the conversion efficiency as a function of chemical composition, and examine its relationship with weight density. For scintillator luminosities, we illustrate that forward mappings are only the starting point for development of design rules, which provide both synthetic guidance for materials chemists as well as a measure of bounding materials performance.

Mechanical Behavior, Microstructure, and Modeling of Ti and Its Alloys: Microstructure/Property Correlation II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Ellen Cerreta, Los Alamos National Laboratory; Vasisht Venkatesh, TIMET; Daniel Evans, US Air Force

Wednesday AM Room: 384
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Thomas Bayha, ATI Allvac/Research and Development; M. Ashraf Imam, Naval Research Laboratory

8:30 AM

Ageing Behavior of Deep-Hardenable Ti-10V-6Cu Alloy: *Hoi Pang Ng*[1]; Colleen Bettles[1]; Barry Muddle[1]; [1]Monash University

The phase stability map proposed by Morinaga for titanium alloys indicates a potential locus for deep-hardenable alloys in the Bo vs Md space. A novel heat-treatable beta alloy Ti-10V-6Cu in the vicinity of the locus has been developed and the dependence of its isothermal ageing behaviour on initial quench rates has been studied. Transmission electron microscopy reveals distinct sizes and morphologies of omega phase in water-quenched and air-quenched samples. However, their effect on the ageing behaviours at 350°C and 500°C, respectively, is confined only to the first few minutes of ageing; after which the hardness profiles are indistinguishable, implying a deep-hardenable character of the alloy. In this work, emphasis is put on the effect of omega phase on the transformation of alpha phase occurred at 500°C ageing. A transitional rhombohedral omega phase has been characterised and its potential role as the nucleation site for alpha phase is discussed.

8:50 AM

Elevated Temperature Oxidation Resistance of Boron Modified Titanium Alloys: Deborah Sweeney[1]; *Raghavan Srinivasan*[1]; [1]Wright State University

It has been established that the addition of trace amounts (~0.1 wt%) of boron to titanium alloys refines the as-cast grain size by an order of magnitude. Reports also indicate that the room temperature corrosion resistance of the boron containing alloys may be substantially greater than conventional titanium alloys. In this study, the effects of boron addition on oxidation resistance are investigated, since conventional titanium alloys have limited corrosion resistance in air above 650°C. The mass gain per unit surface area is measured at an elevated temperature to compare the oxidation of alpha-beta and beta titanium alloys with boron and without boron additions, by exposure to air. These findings are presented in conjunction with the overall characterization of boron modified titanium alloys. Results include microstructural characterization of the oxide layer and the formation of the alpha case and also the hardness of each layer.

9:10 AM

Effect of Oxygen on Fracture Toughness and Stress-Corrosion Cracking of Ti-6211: *Peter Pao*[1]; M. Imam[1]; Harry Jones[1]; Robert Bayles[1]; Jerry Feng[1]; [1]Naval Research Laboratory

Oxygen is a potent solid solution strengthener in titanium alloys and, in relatively small concentrations, can significantly increase the tensile strength and affect the cracking resistance of these alloys. The effect of oxygen concentrations ranging from 0.075 to 0.29 wt% on the fracture toughness, stress-corrosion cracking (SCC) resistance, and fatigue crack growth of Ti-6Al-2Nb-1Ta-0.8Mo (Ti-6211) is investigated. While oxygen has little effect on the fracture toughness below 0.236 wt%, it can significantly reduce the fracture toughness at higher concentrations. Oxygen strongly affects the SCC resistance of Ti-6211. The SCC threshold decreases from more than 120 MPavm for an alloy that contains 0.075 wt% O to 40 MPavm for an alloy that contains 0.29 wt% O. The fatigue crack growth kinetics for Ti-6211 is not affected by the oxygen concentration. The interplay of SCC susceptibility, fracture paths, and fracture mechanisms in Ti-6211 with various amounts of oxygen will be discussed.

9:30 AM

The Effect of Thermal Oxidation on the Fatigue Behavior of Ti6Al4V and Ti6Al7Nb Alloys: *Onur Meydanoglu*[1]; Mehmet Cingi[1]; Murat Baydogan[1]; Huseyin Cimenoglu[1]; Eyup Kayali[1]; [1]Istanbul Technical University

Ti6Al4V alloy is the most favorable titanium alloy because of good combination of improved strength-to-weight ratio, high fatigue resistance with an excellent corrosion resistance. It covers more than 50% of total titanium usage. Recently Ti6Al7Nb alloy has been developed as an alternative to Ti6Al4V by substitution of vanadium by niobium. In this study, the effect of thermal oxidation on fatigue behavior of Ti6Al4V and Ti6Al7Nb has been examined. Based on our previous study which gives the optimum oxidation conditions, thermal oxidation of alloys was performed by holding the samples at 600C for 60h in atmospheric conditions. The fatigue tests were performed on as-received and thermally oxidized samples utilizing a rotating bending fatigue tester. Characterization of the oxide layer was made by means of optical microscopy, X-ray diffractometer and hardness measurement. The fractured surfaces of the samples were investigated by scanning electron and stereo microscopes.

9:50 AM

Effect of Heat Treatment on Microstructures and Properties of Y-Bearing TiAl Alloy: *Yuyong Chen*[1]; [1]Harbin Institute of Technology

In this article, the microstructure control and properties for various microstructures of Ti-45Al-5Nb-0.3Y have been studied using relatively large forged pancake. The canned forging pancake was successfully gained. The effect of processing and heat treatment on microstructure and properties was studied using X-ray diffraction, optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Results showed that the microstructure of forged pancake consisted of major curved lamellar, broken lamellar and minor dynamic recrystallized grains (DRX) with the size of about 1-2μm and the YAl_2 phase at grain boundaries. Three different microstructures, duplex, nearly lamellar and fully lamellar, have been obtained through heat treatment, respectively. And the microstructure evolution was also analyzed. The alloy with duplex microstructure owed the best ductility, the elongation reached 1.9%, and which with fully lamellar microstructure took on better strength, yield strength and fracture strength were 557.1MPa and 715MPa, at room temperature, respectively.

10:10 AM Break

10:30 AM
Microstructural and Mechanical Behavior Characterization of Ultrasonically Consolidated Titanium – Aluminum Laminates: *Tomoko Sano*[1]; James Catalano[1]; Daniel Casem[1]; Dattatraya Dandekar[1]; [1]US Army Research Laboratory

Multilayered hybrid metal laminates have been studied for structural applications due to their potential for higher strength, toughness, and stiffness. The goal of this study is to modify and engineer the microstructure and mechanical properties of commercial purity titanium (CP-Ti) and 1100 aluminum (Al) laminates for potential applications in mine blast mitigation. Alternating layers of 50 µm thick CP-Ti and Al layers were ultrasonically consolidated. To provide high hardness and stiffness, the consolidated material was heat treated in a variety of conditions to form intermetallic $TiAl_3$ layers. The CP-Ti/$TiAl_3$/Al layers processed by the various heat treatments were characterized by scanning electron microscopy to choose the best laminate microstructure for plate impact testing, an instrumented laboratory scale test to characterize the dynamic spall behavior of the material. From those tests, it was determined that the sample with the $TiAl_3$ layers had greater spall strength than the sample without the $TiAl_3$ layers.

10:50 AM
Microstructure and Mechanical Properties of Sponge Ti and Its Alloy Powders with Various Powder Metallurgical Processes: Haitham El Kadiri[1]; William Peter[2]; Robert Whitehorn[1]; *Seong Jin Park*[1]; Youssef Hammi[1]; Randall German[1]; Craig Blue[2]; Jim Kiggans[2]; [1]Mississippi State University; [2]Oak Ridge National Laboratory

Newly developed sponge Ti powder and its alloys have price competitiveness in automotive application. In this study, sponge Ti and several alloys with Al, V, Fe, Zn, Mo, Mn, Sn, Nb, and Cr_3C_2 were investigated in various powder metallurgical processes such as vacuum hot press and rolling process, conventional press and sinter, and powder injection molding. All samples were analyzed in term of density, microstructure, and mechanical properties. The microstructures generated by different compositions and processes were characterized and modeled to rationalize the conflicting effects of substitutional elements on the density, yield strength, ductility, and cost.

11:10 AM
The Effect of Processing Parameters on Biocompatibility of Micro-Arc Oxidized Titanium Alloys: *Mert Gunyuz*[1]; Murat Baydogan[1]; Huseyin Cimenoglu[1]; Eyup Kayali[1]; [1]Istanbul Technical University

In this study, the effect of micro arc oxidation process on surface morphology of Ti_6Al_4V alloy. Micro arc oxidation was performed by using an AC power supply operating in constant voltage mode between 450-600V using two different electrolyte. Mechanical and physical properties were evaluated on the surface including hardness, wettability, surface roughness and scratch resistance of the oxide film. Rockwell C testing was used to compare the relative adhesion characteristics of the oxide film. The surface oxidized samples were hold in simulated body fluid (SBF) to compare the biocompatibility. The surface oxide layer was examined by an X-ray diffractometer and a scanning electron microscope before and after SBF testing. Mechanical and physical properties as well as surface morphology relative biocompatibility of the samples were discussed on the basis of micro arc oxidation parameters such as applied voltage and electrolyte composition.

11:30 AM
An Investigation on Biocompatibility of Titanium Alloys: Nuray Balaban[1]; *Murat Baydogan*[1]; Huseyin Cimenoglu[1]; Eyup Kayali[1]; [1]Istanbul Technical University

Titanium alloys can successfully be used in manufacturing dental and orthopedic implants because of high biocompatibility of oxide layer formed at the surface. Thermal oxidation is the simplest way in forming an oxide layer at the surface and bioactivity of this layer can be further improved with optimizing processing parameters such as time and temperature. In this study, biocompatibility of Ti_6Al_4V and Ti_6Al_7Nb alloys was evaluated by means of holding the samples in a simulated body fluid (SBF) for seven days. The samples were subjected to acidic etching and then thermal oxidation at 400°C and 600°C for different times. Characterization of surface layers before and after SBF tests were carried out by means of surface roughness, wettability measurements, XRD and SEM equipped with EDS. Ti6Al7Nb alloy exhibited best bioactivity after acidic etching prior to thermal oxidation at 400°C for 4 hours.

11:50 AM
Thermo-Mechanical Processing of Boron Modified Beta Titanium Alloys: *Balakrishna Cherukuri*[1]; Raghavan Srinivasan[1]; Sesh Tamirisakandala[2]; Shibayan Roy[3]; Satyam Suwas[3]; [1]Wright State University; [2]FMW Composite Systems; [3]Indian Institute of Sciences

Trace Boron additions to titanium alloys have shown to reduce the grain size in as-cast condition. Two beta titanium alloys: Beta-21S and Ti-5553 with 0.1 wt% B and without boron additions were used in this investigation. The effect of Boron on the thermo-mechanical processing is investigated. Isothermal compression tests were carried out at temperatures ranging from 775 to 950 C at different strain rates ranging from 0.001/s to 1/s. The effect of boron on the processing of beta alloys will be presented in the light of micro-structural effects and the deformation behavior.

Mechanics and Kinetics of Interfaces in Multi-Component Materials Systems: Joint Session with Advances in Semiconductors, Electro Optic and Radio Frequency Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Structural Materials Division, TMS/ASM: Composite Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Bhaskar Majumdar, New Mexico Tech; Rishi Raj, University of Colorado, Boulder; Indranath Dutta, US Naval Postgraduate School; Ravindra Nuggehalli, New Jersey Institute of Technology; Darrel Frear, Freescale Semiconductor; Narsingh Singh, Northrop Grumman Corporation ES; Choong-un Kim, University of Texas - Arlington; Yanfa Yan, National Renewable Energy Laboratory; Bhushan Sopori, National Renewable Energy Laboratory; Greg Krumdick, Argonne National Laboratory

Wednesday AM — Room: 279
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Anthony Fiory, New Jersey Institute of Technology; Bhaskar Majumdar, New Mexico Tech

8:30 AM Introductory Comments

8:40 AM Invited
Morphological Evolution in Polycrystalline Films: *Ramanathan Krishnamurthy*[1]; Mikko Haataja[2]; [1]Caterpillar Inc., Technical Center; [2]Princeton University

Growth models for polycrystalline films commonly used in optoelectronic/materials applications do not consider grain size effects. We address this issue via a thermodynamics-based method that effectively handles grain grooving,lateral growth and surface fluctuations. Groove profiles formed upon annealing (zero flux) deviate considerably from the Mullins' result. Depending upon whether the annealing transport mechanism conserves mass or not, grooving stops completely at late times/small grain sizes L, or a steady state wherein the entire film is translated along its thickness is attained. The surface roughness, for all annealing transport mechanisms, varies as 1/sqrt(L) and L for large/small L, in agreement with recent STEM observations. Using electrodeposition as an example, we demonstrate that height profiles for growing films (non-zero flux) depend critically upon the ratio of the maximally unstable wavelength for growth and the grain size. We also present results for the effect of lateral grain motion on growth morphologies.

9:05 AM Invited
Engineered Interfaces for Silicon Solar Cell Applications: *Bhushan Sopori*[1]; [1]National Renewable Energy Laboratory

Fabricating high-efficiency Si solar cells requires precise control of bulk material quality and of the electrical and optical properties of interfaces. To control the optical properties, interfaces are designed to provide minimum reflection and maximum light trapping within the desired, broad, spectral range. This is accomplished by providing shaped, rough interfaces and by

antireflection/reflection coatings. These features result in optical scattering, leading to very high absorption of the light within a useable spectrum (i.e., in the 0.4–1.1 μm wavelength range) and a concomitant high photo-carrier generation. Electronically, interfaces must provide a very low carrier recombination to prevent loss of photogenerated carriers. One additional consideration for interfaces relates to process design. Solar cell fabrication requires processes such as plasma depositions that can produce surface damage, which can introduce undesirable losses. Hence, such damage must be "healed" during subsequent processing. This paper reviews methods used in fabricating solar cells to achieve interface control. In particular, we discuss interface design features such as texturing, passivation using insulating films with control charge, and minority-carrier reflecting mirrors by dopant grading.

9:30 AM
Simulations of Absorption Efficiency for Three Dimensional Carbon Nanotube Based Photovoltaics: *Jack Flicker*[1]; Jud Ready[1]; [1]Georgia Tech Research Institute

The production of cheap energy from the sun will be a major research objective in the coming years. Major strides must be made in solar cell efficiency, including increasing the absorbance efficiency of a cell by etching or texturing. In order to increase the absorbance efficiency of solar cells, we have developed a three dimensional solar cell structure by depositing a cadmium telluride thin film overtop carbon nanotube towers. These towers act as both a scaffolding and electrical interconnect. Multiple photon interactions as they reflect between these towers increase the absorption efficiency. We have developed a theoretical model and computer simulation to maximize the number of photon interactions due to the geometrical characteristics of the system (aspect ratio, spacing, size, shape, etc). Simulated modeling has shown that by optimization of parameters a three dimensional cell can obtain up to a 300% increase in power production over traditional cells.

9:50 AM Invited
Electromigration Related Effects at Hetero-Interfaces in Multi-Component Systems: *Indranath Dutta*[1]; S. Menon[1]; C. Park[1]; P. Kumar[1]; [1]US Naval Postgraduate School

In many applications with interfaces between dissimilar materials, at least one of the components adjoining the interface is subjected to a large current density and concurrent Joule heating to high homologous temperatures. Under these conditions, electromigration (EM) becomes prominent, and superposed on other thermo-mechanical effects, can cause unusual, scale-sensitive phenomena. In this paper, two instances of EM-related effects next to hetero-interfaces are discussed. First, the influence of EM-induced interfacial diffusion on diffusionally accommodated interfacial sliding in thin film structures in microelectronic devices will be discussed. Model and experiments capturing the interaction between EM and interfacial creep will be presented. Secondly, experimental observations of EM-driven liquid transport along thin film surfaces will be presented, along with potential implications for railgun technology. Based on this, a new method for conformally coating thin film structures with low melting metals, results from which will also be presented. Supported by NSF-DMR-0513874 and ONR-MURI-N00014-04-1-0599RQ-M.

10:15 AM Break

10:25 AM Invited
Interface Reliability in Microelectronic Packages: *Vijay Sarihan*[1]; [1]Freescale Semiconductor, Inc.

Microelectronics and MEMs use a very large variety of packages determined by specific application requirements. This can be from the traditional SOICs all the way to recent advanced packages like the Redistributed Chip Package (RCP). These packages are multilayered structures, use a variety of different materials in the same package and as a result have a large number of distinct interfaces. These packages during assembly, reliability testing and field applications are subjected to a wide variety of processing and environmental exposures like high humidity exposure, high temperature reflow, thermal cycling, shock and drop testing. One of the key package reliability concerns is the interfacial reliability, and interfacial integrity is crucial to the success of all packages. In this paper a number of different package types will be addressed where interfacial integrity was the primary failure mode. These include exposed pad SOIC packages, molded multi chip packages on insulated metal substrates, anisotropic conductive film packages and molded flip chip modules. The emphasis will be on the use of finite element approach with the incorporation of interfacial fracture mechanics.

10:50 AM
Magnetic Field Assisted Assembly-Theory and Experiments: *Rene Rivero*[1]; V. Kasisomayajula[1]; Shanmugamurthy Fnu[1]; Tzesar Seman[1]; Sudhakar Shet[1]; Michael Booty[1]; Anthony Fiory[1]; Nuggehalli Ravindra[1]; [1]New Jersey Institute of Technology

Principles of magnetic field assisted assembly are summarized. Experimental techniques for the assembly process with an optimal control system with feedback is described. The results of the study are analyzed in relation to applications in manufacturing of heterogeneous systems.

11:10 AM
A Fabry-Perot Interferometric Pressure Sensor with Alignment Tolerance and High Temperature Performance: *Ivan Padron*[1]; Anthony Fiory[1]; Nuggehalli Ravindra[1]; [1]New Jersey Institute of Technology

The Fabry-Perot interferometry is one of the most popular of the several optical techniques available for the fabrication of an optical sensor that can provide high degree of sensitivity, versatility and immunity to environmental noise. The Fabry-Perot Interferometric Sensor (FPIS), to be discussed in this presentation, consists of a Fabry-Perot cavity formed between two bonded surfaces: a corrugated diaphragm with a center rigid body (or boss) which deflects under external pressure and keeps a high alignment tolerance and a glass surface with an optical fiber insert. The Fabry-Perot cavity and optical fiber have been used as the sensing element and interconnect, respectively. The Fabry-Perot cavity has been fabricated using the MEMS technology. Micromachining techniques make Fabry-Perot sensors very attractive by reducing the size and the cost of the sensing element.

11:30 AM
Studies of PMD Intrinsic Dynamics of Spooled Fibers under Highly Stable Conditions: *Julio Martinez*[1]; [1]New Jersey Institute of Technology

State of polarization (SOP) and polarization mode dispersion (PMD) of optical communications fibers in an environmentally controlled atmosphere were measured at 1550 nm with a 100 nm bandwidth over extended time periods. The test fibers, which comprised various dispersion compensation module (DCM) spools, were sealed within a vibration-isolated isothermal chamber (three-sigma temperature variation less than 0.02°C). Autocorrelation function analysis, which is used to establish the average drift time associated with intrinsic fiber decorrelation, reveals slowly varying intrinsic dynamics and suggests non-constant dielectric properties of the fibers in a stabilized environment. However, the dynamical magnitudes are much smaller than in fiber-optic communications links, particularly in buried sections, which are subject to seasonal temperature changes and other environmental disturbances. The results are particularly relevant to the hinge model of PMD, which attributes most of the PMD variability in a fiber-optic link to environmental exposure of the DCMs.

Neutron and X-Ray Studies for Probing Materials Behavior: Stresses/Strains and Structure

Sponsored by: National Science Foundation, The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter K. Liaw, University of Tennessee

Wednesday AM Room: 391
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Yandong Wang, Northeastern University; Peter Liaw, University of Tennessee

8:30 AM Keynote
In-Situ Monitoring of Weld Transformations and Their Effect of Weld Residual Stresses: *Philip Withers*[1]; Harry Bhadeshia[2]; John Francis[1]; Laura Pocock[1]; Howard Stone[2]; [1]University of Manchester; [2]University of Cambridge

The volume and shear changes that occur during martensitic transformation can have a marked effect on the final weld residual stress field. Until recently it has not been possible to follow the transformation in real time for realistic weld cooling cycles. In this paper time resolved synchrotron X-ray diffraction measurements are used to follow the transformation, texture and residual stress changes taking place during weld cooling for three weld metals having different transformation characteristics and transformation temperatures. These results are used to interpret the state of residual stress, as measured by neutron diffraction, and texture, as measured by electron back scattered diffraction, found in single-pass groove welds deposited by manual-metal-arc welding using each of the three weld filler metals on 12 mm thick ferritic steel plates.

9:00 AM Invited
The Development of Internal Strains and Texture during Cyclic Deformation of Extruded Mg Az31B: *Donald Brown*[1]; Sean Agnew[2]; Ashutosh Jain[2]; Bjorn Clausen[2]; [1]Los Alamos National Laboratory; [2]University of Virginia

It has been shown that Mg can twin reversibly during cyclic deformation to relatively small strains. We have recently completed in-situ neutron diffraction measurements during strain controlled C-T cycling of extruded Mg to monitor the development of the twin volume fraction and microstructure. Twins appear with compressive deformation along the extrusion axis and withdraw with subsequent tensile deformation. The recovery (de-twinning) of the twins at the maximum tensile strain is observed to decrease with increased cycles, that is the overall twin volume fraction increases. In particular, parent grains which initially have their plane normals parallel to the straining direction cease to de-twin completely at higher cycles. Despite this, no significant repartition of the internal stresses amongst the different grain orientations was observed. However, a significant increase in peak breadth was observed with increased cycle, indicative of increased defect or dislocation density which may inhibit the motion of the twin boundaries.

9:25 AM
Elastic and Plastic Behaviors in Duplex Steel Characterized by In-Situ Neutron Diffraction and Self-Consistent Model: Nan Jia[1]; *Yandong Wang*[1]; Ru Lin Peng[2]; D. W. Brown[3]; [1]Northeastern University, Key Laboratory for Anisotropy and Texture of Materials (MOE); [2]Linköping University; [3]Los Alamos National Laboratory

For duplex alloy being subjected to thermo-mechanical treatments, the respective thermal expansion and different mechanical behaviours of each constituent phase may lead to a non-uniform partition of stresses between the phases. In addition, the grain-orientation-dependent elastic/plastic anisotropy in each phase may cause micro-mechanical interactions which modify further the microscopic load partitioning. In current work, neutron diffraction experiments on the spectrometer for materials research at temperature and stress (SMARTS) was employed on an austenite-ferrite stainless steel for tracing the evolution of micro-stresses that underlies the load sharing among different grain orientations in phases under stress and temperatures fields. Based on the diffraction measured lattice strain distributions on multiple lattice reflections, the anisotropic elastoplastic properties of the duplex steel were simulated using a Visco-Plastic Self Consistent (VPSC) model. Some modelling parameters describing the mechanical behaviours of constituent phases were derived from nanoindentation experiments in combination with EBSD technique.

9:45 AM
Cyclic Twinning-Detwinning Behavior during the Cyclic Deformation of a Wrought Magnesium Alloy, ZK60A: *Liang Wu*[1]; Ashutosh Jain[2]; Donald Brown[3]; Grigoreta Stoica[1]; Sean Agnew[2]; Bjorn Clausen[3]; Douglas Fielden[1]; Peter Liaw[1]; [1]University of Tennessee; [2]University of Virginia; [3]Los Alamos National Laboratory

The twinning and detwinning behavior in a strongly textured magnesium alloy was investigated using in-situ neutron diffraction during the cyclic deformation along the prior extrusion direction. The initial preferred orientation places the c-axis in most grains perpendicular to the loading axis, and this favors extensive {10-12}<10-11> tensile twinning under compressive loading. In contrast, the grains are not favorably oriented to undergo such twinning during monotonic tensile loading along the prior extrusion axis. This is the reason for the well-known tension-compression strength asymmetry of wrought magnesium alloys. The unique orientation relationship between the parent grains and the twin grains also favors detwinning during the subsequent loading reversal. The neutron diffraction indicates that such twinning and detwinning alternates with the cyclic loading. However, a small volume fraction of residual twins gradually increases with increasing cycles, which may be an important factor in dictating the low-cycle fatigue behavior of the magnesium alloy.

10:05 AM Break

10:15 AM Invited
Microstructure, Texture, and Residual Stresses in a Friction Stir Processed AZ31B Magnesium Alloy: *Hahn Choo*[1]; Wanchuck Woo[1]; Michael Prime[2]; Zhili Feng[3]; Bjorn Cluasen[2]; [1]University of Tennessee; [2]Los Alamos National Laboratory; [3]Oak Ridge National Laboratory

Spatial variations of microstructure, hardness, chemical composition, tensile behavior, and texture were investigated in a friction-stir processed (FSP) AZ31B magnesium alloy. Residual stresses were also measured using two different methods: neutron diffraction and contour method. No significant variations in the hardness and chemical composition were found in the FSP zones including the severely deformed stir zone (SZ), which had a finer grain size compared to the heat-affected zone and base material. On the other hand, significant changes in the yield strength, texture, and residual stresses were observed in the FSP zones. The relationship between the texture variations and yield-strength reduction; and its influence on the decrease in the residual stress near the SZ is discussed. Finally, the residual stresses measured by neutron diffraction and contour method are compared, and the effect of the texture on the neutron diffraction residual stress measurements will be discussed.

10:40 AM
Residual Stresses in LENS®-Deposited AISI 410 Stainless Steel Plates: *Liang Wang*[1]; Haitham Kadiri[1]; Sergio Felicelli[2]; Phillip Pratt[2]; John Berry[2]; Camden Hubbard[3]; [1]Mississippi State University, Center for Advanced Vehicular Systems; [2]Mississippi State University; [3]Oak Ridge National Laboratory

The residual stress in thin plate components deposited by the Laser Engineered Net Shaping (LENS®) process was investigated experimentally and numerically. Neutron and X-ray diffraction residual stress mapping was used to characterize the through-thickness stresses and surface stresses in LENS®-deposited AISI 410 stainless steel thin wall plates. Using the commercial welding software SYSWELD, a thermo-mechanical three-dimensional finite element model was developed, which considers also the effect of metallurgical phase transformations. The model was employed to predict the temperature history and the residual stress field during the LENS® process. Several simulations were performed with the geometry and process parameters that were used to build the experimental samples. The origin of the residual stress distribution is discussed based on the thermal histories of the samples, and the modeling results are compared with measurements obtained by diffraction mapping.

11:00 AM Invited
Application of In-Situ Characterization Methods in Developing the Advanced Numerical Models to Predict the Constitutive Behaviors of TRIP Steels: *Xin Sun*[1]; W.N. Liu[1]; M.A. Khaleel[1]; Z.H. Cong[2]; N. Jia[2]; Yandong Wang[2]; P.K. Liaw[3]; [1]Pacific Northwest National Laboratory; [2]Northeastern University; [3]University of Tennessee

Compared to other advanced high-strength steels, TRIP steels exhibit better ductility at a given strength level and can be used to produce complicated automotive parts. This enhanced formability comes from the transformation of retained austenite to martensite during plastic deformation. Development of an advanced quantitative microstructure-based constitutive model for capturing the deformation behavior of the TRIP steels is the first step in predicting optimum processing parameters in steel productions. To develop the micro-mechanical model accounting for the complex deformation behaviors of TRIP steels, in-situ high-energy X-ray and neutron diffraction experiments have been used in this study to quantify the lattice stress-strain relations to validate various numerical results. Some new progresses of experimental and theoretical work will be presented in this talk. The applications of those advanced models in the development of newer steel products as well as the associated manufacturing processes will be elucidated.

11:25 AM
Variation of Residual Stresses in Drawn Copper Tube: *Adele Carradó*[1]; Sebastian Brueck[2]; Laurant Barrallier[3]; A. Fabre[3]; Uwe Struhr[4]; Thomas Pirling[5]; Heinz Palkowski[2]; [1]Institut de Physique et Chimie des Matériaux de Strausbourg (IPCMS); [2]Technical University of Clausthal; [3]ENSAM Aix en Provence; [4]Paul-Scherrer-Institut; [5]Institut Laue-Langevin

Seamless tubes are used for many applications, e.g. in heating, transport gases and fluids, evaporators as well as medical use and as intermediate products for hydroforming and various mechanical applications. They are produced by cold drawing in one or several steps to reach the final dimension. The production of rough tubes is normally done by extrusion or rolling on 3-roll-mills, typically resulting in ovality and eccentricity in the tubes. Ovality and eccentricity lead to non-symmetric material flow during the cold drawing process and therewith to inhomogeneous deformation. Because of this non-axisymmetric behavior and deviations over the length of the tube caused by moving tools, inhomogeneous residual stresses are generated. To understand the interconnections between the geometrical changes in the tubes and the residual stresses, the residual strains in a copper tube were measured by neutron diffraction and compared with its changing geometry over length and circumference.

11:45 AM Invited
Neutron and X-Ray Diffraction Study of Internal Stress in Thermomechanically Fatigued Single Crystal Superalloy: *Erdong Wu*[1]; Jun Zhang[1]; Sucheng Wang[1]; Bo Chen[2]; Guangai Sun[2]; Vincent Ji[3]; Darren Hughes[4]; Thilo Pirling[4]; [1]Chinese Academy of Sciences, Institute of Metal Research; [2]Chinese Academy of Engineering Physics, Institute of Nuclear Physics and Chemistry; [3]Laboratoire d'Ingénierie des Matériaux, École Nationale Supérieure d'Arts et Métiers; [4]Institut Laue-Langevin

The relationship between internal stress and thermomechanical fatigue (TMF) in a Ni-based single crystal superalloy grown along [001] axis is studied by neutron and X-ray diffraction. The macroscopic internal stresses in the alloy generally increase with the TMF cycles until the sample is necked. However, the internal stress in the precipitate γ' phase increases more significantly than that in the matrix γ phase during TMF. The stress developed during TMF cycles in the vertical channel of the γ phase along the loading axis is compressive, whereas that in the horizontal channel is tensile. The microstrains and mosaicity in the γ/γ'phases also increase during TMF, but the trends of their developments are not consistent with that of the internal stresses. These trends and the associated TEM examination indicate that the failure by TMF is mostly caused by the development of defect related stresses in the γ'phase.

Particle Beam-Induced Radiation Effects in Materials: Carbides, Semiconductors and Other Non-Metals

Sponsored by: The Minerals, Metals and Materials Society, American Nuclear Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Gary Was, University of Michigan; Stuart Maloy, Los Alamos National Laboratory; Christina Trautmann, Gesellschaft fur Schwerionenforschung; Maximo Victoria, Paul Scherrer Institute and Lawrence Livermore National Laboratory

Wednesday AM — Room: 389
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Christina Trautmann, Gesellschaft fur Schwerionenforschung (GSI); William Weber, Pacific Northwest National Laboratory

8:30 AM Invited
The Evolution of Charged-Particle Irradiation Damage in Silicon Carbide: *William Weber*[1]; Fei Gao[1]; Ram Devanathan[1]; Yanwen Zhang[1]; Weilin Jiang[1]; In-Tae Bae[1]; [1]Pacific Northwest National Laboratory

Experimental charged-particle irradiations and multi-scale computer simulations have been used to investigate the primary damage state and evolution of radiation damage in silicon carbide as functions of temperature and charged-particle mass and energy. Atomistic simulations of energetic collision cascades indicate that single interstitials, vacancies, antisite defects, and small defect clusters are produced. Atomistic simulations have determined the activation energies for close-pair recombination and long-range diffusion, which are used in a kinetic Monte Carlo (MC) simulation model to describe isochronal annealing of defects in SiC between cascade events. At the charged-particle fluxes normally used in the laboratory to simulate neutron irradiation damage, the ratio of ionization rate to displacement rate in SiC has a significant impact on observed temperature-dependent processes. A fundamental understanding of these ionization effects is needed if charged-particle irradiation results are to be used to develop predictive models of damage evolution in SiC.

9:10 AM
Proton Irradiation Study of GFR Candidate Ceramics: *Jian Gan*[1]; Yong Yang[2]; Clayton Dickerson[2]; Todd Allen[2]; [1]Idaho National Laboratory; [2]University of Wisconsin-Madison

This work investigated the microstructural response of ZrC, ZrN, TiC, TiN and SiC irradiated with 2.6 MeV protons at 800°C to a single dose in the range of 1.5 to 3.0 dpa depending on the material. The change of lattice constant evaluated using HOLZ patterns is not observed and is small when measured using XRD for the irradiated samples up to 1.5 dpa for 6H-SiC and up to 3.0 dpa for ZrC and ZrN. In comparison to Kr ion irradiation at 800°C to 10 dpa from the previous studies, the proton-irradiated ceramics at 3.0 dpa show less irradiation damage to the lattice structure. The irradiated ZrC exhibits faulted loops which are not observed in the Kr ion irradiated sample. The irradiated ZrN shows the least microstructural change from proton irradiation. The microstructure of 6H-SiC irradiated to 3.0 dpa consists of black dot type of defects at high density.

9:30 AM
Light and Heavy Ions Effects on the Electrical and Mechanical Properties of GPC:SiC Composites: B. Chhay[1]; Claudiu Muntele[1]; *D. Ila*[1]; [1]Alabama A&M University

Glassy Polymeric Carbon (GPC) is a unique biocompatible material that can withstand high temperature and present excellent mechanical and electrical properties. When nanoparticles of Silicon Carbide (SiC) is added in the GPC matrix, the electrical conductivity of the composite increases as well as the mechanical performance. In this work, we used a molding method to prepare samples of SiC/GPC composite with 3 different concentrations of SiC and we bombarded them with light and heavy ions to study the induced changes in the electrical resistivity and in the Young's modulus of the composites. Results will be presented during the meeting. This research was sponsored by the Center for Irradiation of Materials, Alabama A&M University and by the AAMURI Center for Advanced Propulsion Materials under the contract number NNM06AA12A from NASA, and by National Science Foundation under Grant No. EPS-0447675.

9:50 AM
Magnetic Order in Proton Irradiated Graphite: Curie Temperatures and Magnetoresistance Effects: Jose Barzola-Quiquia[1]; Pablo Esquinazi[1]; *Daniel Spemann*[1]; M. Rothermel[1]; T. Butz[1]; N. García[2]; [1]University of Leipzig; [2]Consejo Superior de Investigaciones Científicas

Defect induced magnetic order is a new phenomenon in material science that refers to the triggering and manipulation of magnetic order and magnetic moments in nominally non-magnetic materials by lattice defects and/or non-magnetic add atoms. A noticeable example of this effect is the magnetic order at room temperature produced by proton irradiation of graphite. In this work we have managed to increase the ferromagnetic signal by cooling the graphite samples at 110K during proton irradiation, diminishing in this way annealing effects. SQUID measurements of the magnetization show a fluence dependent Curie temperature. The longitudinal magnetoresistance and the Hall effect show an irreversible behavior similar to that found in ferromagnetic films indicating spin/domain reorientation effects. The observed magnetoresistance effects and Curie temperatures above room temperature are promising facts that may lead to useful carbon-based devices in the near future.

10:10 AM Break

10:30 AM
Behavior of 6H-SiC Irradiated to Extremely Low Doses: *Weilin Jiang*[1]; Ponnusamy Nachimuthu[1]; William Weber[1]; Lev Ginzbursky[2]; [1]Pacific Northwest National Laboratory; [2]L.G. Tech-Link

Irradiation of 6H-SiC single crystals was performed using energetic H+ ions. The changes in lattice parameters were measured as a function of dose using high-resolution x-ray diffraction. In general, the lattice parameter changes are small and anisotropic at extremely low doses. The c-axis lattice parameter c increases monotonically with the increasing dose, which could be attributed to aligned interstitials along the c-axis. In contrast, the a-axis lattice parameter a decreases at the extremely low doses, which may originate from the irradiation-induced vacancies and the possible formation of antisite defects. As a result, an initial volumetric contraction of the unit cell occurred. To our best knowledge, this behavior of 6H-SiC has not been reported previously. As the dose increases, both parameters a and c increase with respect to the initial disordering stage. Eventually, the interstitial concentration reaches to such a level that lattice expansion becomes dominant along all directions.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VII: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; Srinivas Chada, Medtronic; Chih Ming Chen, National Chung-Hsing University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-Ho Lee, Hong Ik University; Kejun Zeng, Texas Instruments Inc; Katsuaki Suganuma, Osaka University

Wednesday AM Room: 278
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Yee-wen Yen, National Taiwan University of Science and Technology; Alexandre Kodentsov, Eindhoven University of Technology

8:30 AM Invited
Interactions in the Sn-Cu-Ni System: *Alexandre Kodentsov*[1]; [1]Eindhoven University of Technology

It was found that small additions (a few percents) of Ni in Cu significantly alter the course of the interfacial reactions. No Cu3Sn is formed at the substrate/solder interface, and the Cu6Sn5-based reaction product was found to grow almost an order of magnitude faster compared to that in the Cu/Sn binary diffusion couple. Though this demonstration is a really remarkable one, given the fact that the Sn-Cu-Ni system is, perhaps, the most important material system for Pb-free solder technology, little is yet known in a detail way on the mechanism governing this peculiar reaction phenomenon. Since the additions of the alloying element to the Cu6Sn5-superlattice results in changes of electron concentration in the system, it may be conjectured that the presence of Ni-atoms stabilizes a long-period superlattice of the low-temperature modification of the Cu6Sn5-phase.

8:50 AM
Interfacial Behavior between Bi-Ag Solders and the Ni Substrate: *Hsin-Yi Chuang*[1]; Jenn-Ming Song[1]; [1]National Dong Hwa University

This present study investigated the interfacial reaction between molten Bi-Ag alloys and Ni substrate. The kinetics of substrate dissolution and the growth of the intermetallic compounds (IMCs) were explored. The results show that the dissolution could be divided in two stages, namely, the linear stage and the steady stage. Ni dissolution obeyed the parabolic relationship in the former stage about 20 minutes, afterward the dissolution remained almost constant. Two IMCs, NiBi3 and NiBi, were observed at the interface. Based on the differences in the composition and morphology, it could be demonstrated that NiBi3 formed via interfacial reaction as well as crystallization in isothermal period and also during solidification. The NiBi layer formed between NiBi3 and Ni substrate was believed growing through a solid-state transformation. It was also found that the Ag addition accelerated the dissolution of Ni and thus the growth of Ni-Bi IMCs.

9:05 AM
Interfacial Reaction and Thermal Fatigue of Zn-4wt.%Al-1wt.%Cu/Ni Solder Joint: *Yoshikazu Takaku*[1]; Ikuo Ohnuma[1]; Yasushi Yamada[2]; Yuji Yagi[2]; Ikuo Nakagawa[3]; Takashi Atsumi[3]; Kiyohito Ishida[1]; [1]Tohoku University Graduate School of Engineering; [2]Toyota Central Research and Development Laboratory Inc.; [3]Toyota Motor Corporation

The Zn-Al-Cu eutectic alloy (Tm=381°C) is a candidate for use as a Pb-free high temperature solder as a substitute for Pb-base high solders. It is suitable for severe working environments such as the engine room of hybrid vehicles equipped with an inverter system as well as a heat engine. In this study, the interfacial reaction between Zn-Al-Cu alloys and the Ni substrate during soldering and thermal cycling was investigated. Semiconductor chips and Ni-plated Cu substrates were soldered with Zn-Al-Cu alloys at various temperatures under a nitrogen atmosphere. The soldered assemblies were then heat-treated at 200°C and 300°C to examine the microstructural evolution at the soldered interface. The effect of severe thermal cycles between -40°C and 250°C in the air on the microstructure and fracture behavior at the solder joint was

investigated. Even after a 1000-cycle test, the thickness of Al3Ni2 layer formed at the interface was quite small.

9:20 AM
Dissolution and Interfacial Reaction between Cu and Sn-Ag-Cu Solders: *Chih-Chiang Chang*[1]; C. Robert Kao[1]; [1]National Taiwan University

In electronic packaging, the dissolution of the Cu layer of the under-bump metallizations or surface finishes during reflow is one of the most important processing concerns. Therefore, the objective of this study is to investigade in depth the dissolution of Cu into SnAgCu solders with various Cu concentrations. The effect of solder volume on dissolution rate will also be studied. In this study, solder balls of different concentrations Sn3AgxCu(x=0/0.3/0.5/0.7wt.%) and different diameters, 500 or 760μm, are employed to study the influence of Cu concentration as well as the solder volume. It is found that the Cu concentration and solder ball volume have very strong effect on the Cu consumption rate. The different initial Cu concentration in the SnAgCu ternary solder changed the dissolution driving force(the Cu concentration gradient). The results of this study suggeste that a high Cu-content SnAgCu solder can be used to reduce the dissolution of Cu pad.

9:35 AM
Interfacial Reaction between Sn-Ag-Bi-In Lead-Free Solder and Copper Substrate: Ming-Hsiun Chen[1]; Che-Hsiun Huang[1]; Wei-Tang Chen[1]; *Albert Wu*[2]; [1]National Taipei University of Technology; [2]National Central University

It is still important to find a new solder alloy system for the replacement of eutectic SnPb solder. Though Sn-Ag-Bi alloy is one of the choices for lead-free solder, bismuth usually deteriorates the mechanical properties of the joints. The addition of indium can lower the melting point of the alloy but will not reduce the mechanical strength of the joints. In this study, we investigated the interfacial reaction between Sn-Ag-Bi-In solder and Cu substrate. By using different reflow conditions, the growth of intermetallic compound was conducted by measuring the thickness versus reflow time and temperature. The interfacial reaction for different contents of indium added in the Sn-Ag-Bi system was also studied. The compounds formed in the reaction are mainly Cu_6Sn_5 and Cu_3Sn at the interface. The kinetics of compound growth was also investigated in this study.

9:50 AM
Thermal Stability Improvements in Cu Metallization on Barrierless GaAs: *Wean-Kuan Leau*[1]; Jinn. P. Chu[1]; V.S. John[1]; [1]National Taiwan Ocean University

Barrierless copper metallization of GaAs device was performed using Cu/GaAs and Cu(TaNx)/GaAs structures by co-sputtering system. The pure Cu and Cu(TaNx) films were characterized by X-ray diffractometry, focussed ion beam, X-ray photoelectron spectroscopy and low angle X-ray diffractometry, which authenticates the results. The Cu films on GaAs were very stable up to 350°C for 1 hour. The Cu(TaNx) films on GaAs show better thermal stability up to 450°C for 1 hour. The thermal stability improvement by doping a minor amount of TaNx into Cu has been confirmed. The thermal property and microstructure results will be presented and discussed.

10:05 AM Break

10:20 AM Invited
Suppressing the Growth of Cu5Zn8 Intermetallic Layer in Sn-Zn-Ag-Al-Ga/Cu Solder Joints: *Kwang-Lung Lin*[1]; R. Lai[1]; Budiman Salam[1]; [1]National Cheng Kung University

The presence of the intermetallic compound (IMC) layer at the interface between solder and substrate indicated a bond actually formed between the solder and the substrate. Besides its advantage for bonding the solder and the substrate, its disadvantage is that it is generally the most brittle part of the solder joint. In this study, the growth of IMC layers was seen slower as the silver content increased in the Sn-8.5Zn-Ag-Al-Ga/Cu alloy systems. The experimental results show that the total thickness of the IMC layers in the studied solder alloy with 1.5wt.% silver content is about half that in the studied solder alloy without silver added. The reduction might be due to the formation of the (Ag, Cu)-Zn intermetallic at the interface of the silver added solder which in the no silver solder, Cu5Zn8 was normally found.

10:40 AM
Interfacial Reaction between Eutectic SnZn Solder and Thin-Film Cu Substrate: *Chih Ming Chen*[1]; Chih Hao Chen[1]; [1]National Chung-Hsing University

SnZn eutectic alloy is a promising lead-free solder. Copper is a common metallic substrate in electronic devices. Thus, interfacial reactions at the SnZn/Cu solder joints are studied extensively. However, the Cu is usually the bulk type; interfacial reaction regarding the thin-film Cu substrate is not studied yet. In this talk, we will present that the interfacial reaction of thin-film Cu is different from that of bulk Cu. Reaction product and interfacial morphology will be discussed and compared between the bulk and thin-film Cu types.

10:55 AM
Interfacial Reactions between Pb-Free Solders and In/Ni/Cu Substrates: *Yee-wen Yen*[1]; Wei-kai Liou[1]; Hong-yao Wei[1]; Chiapyng Lee[1]; [1]National Taiwan University of Science and Technology

This study investigated the interfacial reactions between Pb-free solders-Sn-3.0 wt%Ag-0.5 wt%Cu (SAC) and Sn-0.7 wt%Cu (SC), and In/Ni/Cu multilayer substrates by the diffusion bonding method. The reflow temperatures were at 160, 180 and 200°C for various lengths of time, and then samples were annealed at 100°C for 100 to 500 hours. The experimental results show that the scalloped Cu6Sn5 phase was formed and spread over the SAC/In/Ni/Cu and SC/In/Ni/Cu interface. The ternary Ni-In-Sn intermetallic compound (IMC) was formed when the samples were aged at 100°C. At the reflow temperature was increased to 200°C, only one Cu6Sn5 phase was formed on the solder/substrate interface even the reaction couple was aged at 100°C for 500 hours. According to mechanical test, the formation of the Ni-In-Sn ternary IMC cause the mechanical strength of the solder joint decrease.

11:10 AM
Local Resistance Measurements via Four Points Probe (4pp) Method in Eutectic Solder Assembly after Temperature Cycling Test by SEM Internal Probing Technique: T.I. Shih[1]; Yung-Chi Lin[1]; *Jeng-Gong Duh*[1]; Tom Hsu[2]; [1]National Tsing Hua University; [2]United Microelectronics Corporation

Interfacial reaction for the flip chip solder bump of Sn63-Pb37 structure and electrical characterization for lead-free materials in the bump technology under TCT of environmental tests were investigated. In the industry, there is very limited success in understanding the precision of electrical resistance via four points probe method, as well as in reducing resistance errors due to inaccuracy in the positions of the probes. The present study was aimed to provide an innovative attempt to cast additional light on this critical subject through local nano-probing in a high-resolution scanning analytical electron microscope (FEI XL-30). The microstructure, elemental distribution and electrical characterization of IMCs were thoroughly examined using an integrated combination set-up of FE-EPMA, SEM nano-probing system and SELA-EM2. This allows us to obtain a well-documented correlation between the electrical properties of the specific IMCs and their morphology as well as microstructure.

11:25 AM
Modelling Sn-Cu Interfacial Reactions with the Phase Field Method: *An Serbruyns*[1]; Nele Moelans[1]; Ingrid De Wolf[2]; Bart Blanpain[1]; Patrick Wollants[1]; [1]Katholieke Universiteit Leuven; [2]IMEC

During soldering and subsequent thermal cycling, an intermetallic compound (IMC) layer is formed between the molten solder and the solid substrate, which is required for a strong bond. However, thick IMC layers are detrimental for mechanical properties of the joint. Therefore, their growth has to be controlled. The behaviour of the IMC layers in Sn-Cu systems is studied using a phase-field model. In the phase field method, microstructures are represented by a set of phase field variables that are continuous functions of the spatial coordinates and time. The model uses a diffuse interface approach, which allows the consideration of several concurrent microstructural processes for complex morphologies, without a considerable increase of the mathematical complexity. Influence of diffusion coefficient ratios, start concentrations, elastic properties and other parameters are investigated.

Pyrometallurgy - General Sessions: Pyrometallurgy

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Boyd Davis, Queens University

Wednesday AM Room: 283
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Michael Potesser, University of Leoben

8:30 AM
Enhanced Reduction of Self-Reducing Pellet of Chromites with Fe-Si Addition in the Reductant: *Cyro Takano*[1]; Adolfo Zambrano[1]; Marcelo Mourao[1]; [1]University of Sao Paulo

Fe-Cr-C production is a very high electrical energy consuming process. When self-reducing agglomerates are used it is expected to decrease up to 10% of this electrical energy. This research aims to understand how the synergetic effects are when stronger reductant, like Fe-Si, is substituted for coke. Brazilian chromites containing 41.2%Cr2O3 were mixed with petroleum coke, small amount, 1%, 2% and 4% of Fe-75%Si and agglomerated with cement as the binder. The mixture was pelletized, dried and submitted at temperatures 1773K up to complete the reactions. The results showed that the reduction reactions were 1.4 and 2 times faster with 1% and 2%Fe-Si addition, respectively, than with no addition. The microscopic analysis showed that a liquid phase formed but the pellet did not collapse and indicated that the coalescence of the metallic phase depends on the dissolution of the pre-reduced particles of the chromites into slag.

8:55 AM
Improvements Carried out at Huelva Smelter in the 2007 General Shutdown: A Review: *Rafael Fernandez-Gil Bando*[1]; Carlos Ortiz[1]; Juan Moreno[1]; [1]Atlantic Copper SA

The Huelva Flash Smelting Furnace was commissioned in September 1975. The Smelter and the Acid Plant was expanded in 1995 to its present capacity of 300,000 tpa new copper equivalent and one million tonnes of concentrate per year. During these years of operational experience, most of the initial practices have been reviewed, process control has drastically improved and main process equipment has been modernised. The fourth Flash Furnace campaign lasted 9 years (1995-2004) during which 8 million tonnes concentrate were smelted. Increasing in sulphur content in concentrates and new environmental requirements imply new challenges for smelters to keep production and operation costs. This paper is an overview of the modifications and improvements carried out in the 2007 general shutdown in Smelter and Acid Plants. Also environmental improvements carried out on Smelter gasses treatment systems are described.

9:20 AM
Impurities Behavior under Oxidization and Reduction Conditions of Non-Ferrous Pyrometallurgical Process: *German Wastavino*[1]; [1]IM2 Codelco

In recent years the amount of the impurities such as As and Sb in the smelting process has increased because of the quality of the copper concentrate, producing a high variation of the concentration of impurities in the anode. This behavior create an additional problem in the electrolytic refining because it affects the ratio As/(Sb+Bi), increasing the concentration of As and Sb in the cathode. The objective of this study is to clarify the effects of oxidization and reduction conditions in different processes.Teniente Converter, Pierce Smith Converter and Slag Cleaning Electric Furnace were evaluated under different industrial sceneries to achieve a high impurities volatilization. The present work describes the main results and conclusions of this study. It was found that the behavior of impurities in electric furnace might limit the process, because the recovered metal contains high concentration of As and Sb which is usually recirculated to conventional converting process.

9:45 AM Break

10:05 AM
New Pyrometallurgical Bullion Lead Refining Process: *Michael Potesser*[1]; Burkhardt Holleis[2]; Helmut Antrekowitsch[1]; [1]University of Leoben; [2]Messer Austria GmbH

After the primary or secondary lead production the bullion lead is remelted and charged into a vessel with following refining process. The removing of arsenic, antimony and tin takes place by salt slags or oxidizing gases like air or pure oxygen. Due to the increasing lead price Messer Austria GmbH and in cooperation with the institute of Nonferrous Metallurgy at the University of Leoben developed a new process named OXIPOT for the refining of Sn, As and Sb. The OXIPOT process bases on the bottom purging technology with a ceramic or steel purging device. This leads to an increased retention time of the refining products due to the deeper bath level and an optimized specific reaction volume concerning the bubble size. This paper describes the process, pilot plant, thermodynamic calculations for the optimisation concerning the selectivity of element removal and an economic calculation.

10:30 AM
The Investigation of Domestic Iron Ores for Sponge Iron Production: *Ercin Ersundu*[1]; Nuri Solak[1]; Süheyla Aydin[1]; [1]Istanbul Technical University

In the present work, three different domestic iron ores; Sivas Divrigi B Head high grade hematite, Malatya-Hekimhan-Hasançelebi medium and low grade magnetite lump ores reduced with Soma-Kisrakdere lignite coal to investigate their suitability for sponge iron production. In the experimental studies different operation parameters were selected being Cfix/Fetot ratio, temperature in the range of 1100-1250°C and time, to determine their effects on metallization. Metallization degree of 88% was calculated in the reduction experiments realized by using Sivas Divrigi B Head lump ore, whereas for Malatya-Hekimhan-Hasançelebi magnetite lump ores 65-70% metallization degrees were obtained. Additionally, kinetic calculations have been performed and it was found that the reduction reactions obey the Ginstling – Brounshtein type diffusion controlled model. Activation energy values were determined in the range of 55–150 kJ/mol for different types of iron ores, which is in agreement with the literature data.

10:55 AM
Practice of Separating Precious Metals from Base Metals in Gold-Antimony Alloys by Selective Chlorination Leaching under Controlling Potential: Liu Weifeng[1]; Yang Tianzu[1]; *Dou Aichun*[1]; Chen Fangbin[1]; Liu Yong[1]; [1]Central South University

Owing to the existing problems in the electrolysis of gold-antimony alloys process, the new hydro-technology of separating precious metals from base metals have been studied in china. In this paper, the new hydro-technology of separating precious metals from base metals has been introduced in detail. The technology of selective chlorination leaching under controlling potential has been applied to treat the gold-antimony alloys. The impurity metals were removed effectively since the leaching efficiencies of copper, nickel and antimony are over 99%, with direct recovery of gold being 99.83%. Silver, antimony, copper and nickel were recovered effectively. The crude gold powder containing 94% gold can be obtained from the residue. The direct recovery of gold can be improved from 70% to 99.83%. This technology has been applied successfully in the treatment of gold-antimony alloys since September 2005. Many former procedures have been cancelled, such as electrolysis, Anode slime leaching by nitric acid, smelting in crucible and blowing in muffle.

Recycling: Light Metals

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Christina Meskers, Delft University of Technology; Greg Krumdick, Argonne National Laboratory

Wednesday AM Room: 280
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Christina Meskers, Delft University of Technology

8:30 AM
Dynamic Processes for the Recycling of Low-Quality Aluminum Charge Materials: *Simon Lekakh*[1]; David Robertson[1]; Sergey Rimoshevsky[2]; Leonid Tribyshevsky[2]; Vladimir Tribyshevsky[2]; [1]University of Missouri-Rolla; [2]Belarusian National Technical University

Melting processes may be classified into two groups – stationary and dynamic. In stationary processes the melt and solid charge are immovable or listlessly moving by natural convection. In dynamic processes there is active motion of the solid charge and melt inside the furnace by external mechanical or electromagnetic forces. Features of these processes were analyzed from the point of view of metal recovery and energy efficiency when charge materials with different metallurgical quality were used. The dynamic melting processes have the possibility of increased aluminum recovery from low-quality scrap with oxidized surface, such as turnings and dross, by effective separation of melt from slag with using special fluxes. The melting parameters in a dynamic rotary furnace were experimentally studied and computationally modeled. Technical results will be presented of using a small size (0.5 t) rotary furnace for recycling locally-sourced low-quality aluminum charge materials over a period of several years.

8:50 AM
Fundamental Study of the Decoating of Aluminium Beverage Can: Xiangjun Zuo[1]; *Lifeng Zhang*[2]; [1]Norwegian University of Science and Technology; [2]Missouri University of Science and Technology

The first step of the recycling of aluminium beverage can is decoating, removing surface layer of plastic (polyester coat on the inwall of can) and paint (on the ectotheca). In the current study, Small pieces of Carlsberg beer can were heated in TG/DTA and MS machines at different heating rates and different carrying gas flow rates. SEM is used to observe the structure of the samples before and after decoating. The activation energy of reactions at different temperature is deduced according to the experimental data. A model has been developed to describe the decoating process.

9:10 AM
The Recycling of Aluminum from Building and Construction Applications: *Subodh Das*[1]; John Green[2]; John Kaufman[1]; [1]Secat Inc; [2]JASG Consulting

The aluminum industry is a leading proponent of global sustainability and has for many decades strongly advocated recycling of the metal. Recycled aluminum is a key and growing component of the U.S. metal supply and in recent years has actually exceeded the aluminum supplied from primary production. The use of recycled aluminum saves ~95% of both the energy and emissions associated with the smelting of aluminum and reduces reliance on foreign imports. This paper explores the recycling of aluminum from the building and construction (B and C) markets, taking advantage of a European study by Delft Technical University. Significant potential savings from recycling during building demolition with aluminum recoveries averaging ~95% are demonstrated. A rudimentary on-site alloy sorting scheme is proposed and a pathway forward is suggested to validate the sorting approach and confirm the recycling potential. Further, the potential for reuse of the resultant alloy content is considered.

9:30 AM
Microstructures Evolution and Mechanical Properties of Rare Earth Magneium Alloy Machined Chips Recycled by Severe Plastic Deformation: *Tao Peng*[1]; Qudong Wang[2]; [1]Shanghai Jiaotong University, School of Materials Science and Engineering; [2]Shanghai Jiaotong University, State Key Laboraroty of Metal Matrix Composite

GW103K Rare earth magnesium alloy machined chips were recycled by CEC (cyclic extrusion compression) technology. In this work, the total recycling course mainly included sintering technology of Machined chips and CEC of the sintered billets. Machined chips were sintered at the temperature of 673K, 723K and 773K with a pressure of 250 Mpa in air. Those sintered billet were then extruded at 773K. CEC not only made machined chips excellent metallurgical bonding, reduced the porosity, but made uniform dispersion of oxide contaminants and grains refinement with the incressing of extrusion passes. The extrusions processed from machined chips showed a good combination of high ultimate tensile strength of 400~460Mpa, high 0.2% proof stress of 300~350 Mpa and elongation to failure of 5~10% at the room temperature which had higher strength and equivalent elongation compared with those of extrusions processed from the cast ingot.

Refractory Metals 2008: Processing

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Refractory Metals Committee
Program Organizers: Todd Leonhardt, Rhenium Alloys Inc; Jim Ciulik, University of Texas at Austin

Wednesday AM Room: 388
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Todd Leonhardt, Rhenium Alloys, Inc; James Ciulik, University of Texas at Austin

8:30 AM Introductory Comments

8:35 AM
Transitions from Tool Steels (H-13) to Refractory Metals (Mo, Ta, W) through Laser Powder Deposition: *James Sears*[1]; [1]South Dakota School of Mines and Technology

Laser powder deposition of refractory metals, such as tantalum, molybdenum, rhenium and tungsten, is being evaluated for manufacturing of permanent molds for titanium casting, as internal cladding for gun barrels and shot liners for Mg and Al die casting. This work is focused on transitions from tool steel (H-13) to these refractory metals via an intermediary material by successive layering by Laser Powder Deposition. Issues related to transition materials used, powder type and morphology, clad quality and dilution control that were encountered during this project are discussed. This work was performed under a grant from the U.S. Department of Energy (DOE), Office of Industrial Technology under contract DE-PS07-03ID14425: Industrial Materials for the Future Program.

9:00 AM
Molybdenum and Molybdenum Alloy Seamless Tubing: *Donald Mitchell*[1]; Todd Leonhardt[1]; [1]Rhenium Alloys Inc

An increase in demand for longer length and thinner wall tubing, along with technological advancements in design and applications have resulted in requirements of higher quality and greater precision of molybdenum and molybdenum alloy seamless tubing. Several methods of production will be discussed; including plug drawing and mandrel drawing. Mechanical properties and microstructures will also be illustrated and discussed. Several of the industries benefiting from these materials are temperature measurement, medical, nuclear, and aerospace.

9:25 AM
The Response of an NbSi Alloy to Net Shape HIPping: Qiang Li[1]; Roger Morrell[2]; *Xinhua Wu*[1]; [1]University of Birmingham; [2]National Physical Laboratory

Hot Isostatic pressing (HIPping) has been used to consolidate atomised Nb-16Si-25Ti-8Hf-2Cr-2Al using a three stage HIPping process to produce a near net shape component. The creep and oxidation behaviour of the as-HIPped powder have been assessed. The consolidation during HIPping has been monitored using analytical scanning and transmission electron microscopy and X-Ray diffraction. The powder is HIPped in mild steel tooling to define the shape after which it is de-canned and HIPped at higher temperatures in order to remove the remaining porosity. Young's modulus and the shear moduli of the powder-HIPped samples have been measured and are similar to those found in cast samples. The oxidation properties are similar to those of cast samples but the creep properties are worse, presumably because of the fine scale of the microstructure. These observations are discussed in terms of using HIPping of powder to produce Net Shape components of NbSi-based alloys.

9:50 AM
Development of Irradiation Hardening of Unalloyed and ODS Molybdenum during Neutron Irradiation to Low Doses at 300C and 600C: *Brian Cockeram*[1]; Lance Snead[2]; Richard Smith[1]; [1]Bechtel Bettis Inc.; [2]Oak Ridge National Laboratory

Unalloyed molybdenum and Oxide Dispersion Strengthened (ODS) molybdenum were neutron irradiation at nominally 300C and 600C in the high fluence isotope reactor (HFIR) at relatively low fluences that correspond to nominal displacement doses of 0.01, 0.1 and 1.3 dpa. Following Irradiation hardening occured at neutron fluences of 1.3 dpa with an increase in yield stress, a significant decrease in uniform elongation, and elevation of the Ductile to Brittle Transition Temperature (DBTT). The hardening behavior was evaluated by comparing the observed defect and defect cluster formation and accumulation with indirect measures of defect density obtained from electrical resistivity measurements, hardness measurements, and tensile properties. The influence of temperature on the mobility of defects and resulting properties also was evaluated. Additionally, the role of microstructure and grain size on irradiation hardening was evaluated by comparing the results for unalloyed molybdenum and ODS molybdenum.

10:15 AM Break

10:25 AM
Consequences of Crystal Orientation on Fabricating Single or Multi-Crystal Nb SRF Cavities: *Derek Baars*[1]; Thomas Bieler[1]; Chris Compton[1]; Terry Grimm[1]; [1]Michigan State University

Manufacturing superconducting radio frequency (SRF) cavities from single crystal niobium is being investigated as an alternative to polycrystalline niobium for several reasons: 1) single crystal sheets may be cut directly from the purified ingot, eliminating the cost of rolling into polycrystalline sheet, 2) polycrystalline sheet texture varies among manufactures and even between batches, leading to variability in forming and surface finish, 3) a single crystal cavity has already been tested and matches the capability of polycrystalline cavities. The effect of various crystal orientations on dislocation density, surface quality, and recrystallization after plastic deformation similar to that anticipated for normal cavity assembly steps such as forming operations, chemical etches, and welding will be presented.

10:50 AM
Small-Scale Resistance Spot Welding of 50Mo-50Re Thin Sheet: *Jianhui Xu*[1]; Williams Umstead[2]; John Farrell[2]; Michael Effgen[2]; Tongguang Zhai[1]; [1]University of Kentucky; [2]Semicon Associates

A 50Mo-50Re alloy, in a form of thin sheet (0.127 mm thick), was jointed by small-scale resistance spot welding (SSRSW). The effects of six important welding parameters, such as hold time, electrode, ramp time, weld current, electrode force and weld time, were studied systematically, in an attempt to optimize the welding quality. It was found that the commonly used criterion that the suitable nugget penetration was 80% of the thickness of the welded sheet in large-scale resistance welding was not applicable to SSRSW of the 50Mo-50Re sheet, since a penetration larger than 80% found in this study did not deteriorate the strength of the weld. The diameter of a nugget was only 30-40% of the electrode diameter in SSRSW due to the relatively low electrode force, compared with the high electrode force employed in large-scale resistance welding where the diameter of nugget was almost 100% of the electrode diameter.

11:15 AM
High Temperature Mo-Si-B Alloy Designs and Microstructures: *J. Perepezko*[1]; R. Sakidja[1]; [1]University of Wisconsin-Madison

The multiphase microstructures that can be developed in the Mo-Si-B system offer useful options for high temperature applications. Alloys based upon the coexistence of the high melting temperature (>2100C) ternary intermetallic Mo_5SiB_2 (T_2) phase with Mo(ss) and Mo_3Si phases allow for in-situ toughening and offer favorable oxidation resistance. A focal point of the microstructural designs is the T_2 phase which exhibits a range of solubility. Selected refractory metal substitutional alloying has been examined to alter the solubility of the T_2 phase and the relative phase stability as a method to develop multiphase microstructure design options. The observed alloying trends also highlight the fundamental geometric and electronic factors that influence the relative stability of the T_2 phase. Similarly, the systematic investigation of reaction kinetics involving the T_2 phase has a direct application to the analysis of oxidation behavior and to the design of effective coating systems with self-healing and gradient characteristics.

11:40 AM
Influence of Texture and Velocity on the Dynamic Tensile Extrusion Response of Tantalum: *Fang Cao*[1]; Ellen Cerreta[1]; George Gray[1]; [1]Los Alamos National Laboratory

The mechanical behavior and damage evolution of tantalum are influenced by texture, strain rate, and grain size. The effects of original rolling texture and grain size on the large-stain dynamic tensile extrusion process in a high purity Ta have been investigated and quantified in this research. The post-extrusion macroscopic evolution of the samples was captured using high-speed photography in a modified Taylor Gun Facility. Post-mortem characterization of the microstructure, surface topography, texture, and substructure evolution was conducted using optical metallography, scanning electron microscopy (SEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM), respectively. The dynamic tensile extrusion behavior and microstructure evolution in Ta have been compared to its responses under quasi-static and controlled high strain rate shear conditions to evaluate "path dependency" effects on defect storage processes.

Structural Aluminides for Elevated Temperature Applications: Phase and Microstructure Evolution

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Young-Won Kim, UES Inc; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Rui Yang, Chinese Academy of Sciences; Christoph Leyens, Technical University of Brandenburg at Cottbus

Wednesday AM — Room: 394
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Rui Yang, Chinese Academy of Sciences; Michael Loretto, University of Birmingham

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8:30 AM Invited
Modulated Microstructures: A Novel Approach for the Design of TiAl Alloys: *Fritz Appel*[1]; [1]GKSS Research Centre

Phase decomposition and ordering reactions in beta/B2-phase containing TiAl alloys were utilized to establish a novel, previously unreported, type of in-situ composite. The characteristic constituents of this composite are laths with a modulated substructure that are comprised of several stable and metastable phases involving gamma, alpha-2, beta/B2, and orthorhombic B19. The modulation occurs at the nanometre-scale and thus leads to a significant structural refinement. Interfacial dislocations and ledges accommodate the lattice mismatch between the different phases and provide a fine dispersion of nucleation sites for mechanical twins. The phase transformations involved in the formation of the microstructure were characterized by high-resolution electron microscopy. Microstructural control of the alloys can be achieved by

conventional thermo-mechanical processing, which however has to be tightly tailored to the alloy composition. Potential applications will be discussed by addressing the strength, creep resistance and fracture toughness of the material.

9:00 AM

Strengthening of Lamellar TiAl Alloys by Dynamic Precipitation of Beta Phase during Creep: Hanliang Zhu[1]; *Kouichi Maruyama*[2]; [1]University of Queensland; [2]Tohoku University

Further improvement of their strength at elevated temperatures is necessary for widespread applications TiAl alloys. In TiAl alloys, alpha2 Ti3Al phase transforms to gamma TiAl phase during high temperature exposure. This transformation results in disappearance of gamma/alpha2 boundaries, and the consequent weakening of the alloys. Recent TiAl alloys often contain beta stabilizing elements to have their better formability. In such alloys, precipitation of beta particles takes place during the alpha2 to gamma transformation, and precipitation zones of dense beta particles are formed in place of prior alpha2 plates. The precipitation zones can act as barriers to dislocation motion, and can bring about strengthening. Microstructural changes during creep exposure were studied up to 9000 h at 760C with Ti-47Al-2W-0.2Si alloy. Dynamic precipitation and dissolution of beta particles were observed during the creep tests. It will be discussed how to design the beta precipitation to improve its creep strength.

9:20 AM

In-Situ Characterization of Phase Transformations and Microstructure Evolution in a γ-TiAl Based Alloy: *Klaus-Dieter Liss*[1]; Andreas Stark[2]; Arno Bartels[2]; Helmut Clemens[3]; Thomas Buslaps[4]; Dominic Phelan[5]; LaReine Yeoh[1]; [1]Australian Nuclear Science and Technology Organisation; [2]Hamburg University of Technology; [3]Montanuniversität Leoben; [4]European Synchrotron Radiation Facility; [5]University of Wollongong

Phase diagrams and microstructures of titanium aluminides are rather complex and, so far, little data were observed in-situ at elevated temperatures. We report on two-dimensional high energy X-ray diffraction and complementary laser scanning confocal microscopy to characterize the appearing phases and to follow the phase evolution in-situ in real time. As an example, the microstructure evolution of a quenched γ-TiAl alloy, consisting of α2-Ti3Al grains at RT, has been followed in both reciprocal and direct space as a function of temperature up to 1400°C. At 600-800°C extremely fine γ-laths are formed in α2-grains occurring through an oriented rearrangement of atoms. Streaks linking reflections of both phases testify from coherent lattice and orientation gradients in the transforming crystallite. At temperatures above the eutectoid temperature recrystallization effects and the γ->α phase transition take place leading to grain refinement.

9:40 AM

Evaluation of Experimental Beta Gamma Alloys Produced via Powder Metallurgy: *Charles Yolton*[1]; Jean Stewart[1]; Young-Won Kim[2]; Alojz Kajinic[1]; [1]Crucible Materials Corporation; [2]UES

Beta gamma alloys are a new class of gamma titanium aluminide alloys that are under continued exploration to offer refined cast structures and improved hot-workability and machine-ability over conventional gamma alloys. The improved process-ability is associated with beta solidification and solid-state phase transformation pathways that yield significant amounts of finely dispersed stable beta at processing temperatures. While beta gamma alloys offer improved cast-ability, melting and casting of large ingots may still present challenges. Powder processing of beta gamma alloys can offer certain advantages over ingot metallurgy processing and the only size limitation is the volume of the vessel used for consolidation by hot isostatic pressing (HIP). Two experimental beta-gamma alloys, Ti-44Al-7(Nb, Mn) at% and Ti-44Al-4(Nb, Mo) (at%), were selected for the present work and their powders were produced by gas atomization. This paper will present the initial findings on production of beta gamma alloys via a pre-alloyed powder route.

10:00 AM Break

10:10 AM Invited

Alloy Design Concept for Development of Wrought Gamma Titanium Aluminides - Roll of Interstitial and Substitutional Elements in Microstructural Control: *Masao Takeyama*[1]; [1]Tokyo Institute of Technology

Cast TiAl based alloys are in practical use for automobile and jet engine components. In contrast, the wrought alloys are currently under progress. This talk gives the alloy design concept and thermo-mechanical controlled processing (TMCP) methods for the development of wrought gamma alloys, based on the phase equilibria, phase transformations and kinetics of precipitation in Ti-Al-M ternary systems. Emphasis will be placed on the role of substitutional and interstitial elements in microstructure control. The appropriate addition of substitutional β stabilizer M (V, Nb, Cr, Mo) creates the novel transformation pathway of β->α->β+γ. This transformation makes it possible to impart both hot deformability in process and toughness in service by dispersing the β particles within lamellae. . The combined addition of an interstitial element, carbon, stabilizes γ phase at lower temperatures, allowing us to control a fine-grained fully lamellar microstructure with fine dispersion of p-type carbide (Ti_3AlC) within lamellae.

10:40 AM

Thermal and Stress Induced Formation of Ordered ω-Phase in Nb-Rich γ-TiAl Based Alloys: Andreas Stark[1]; *Arno Bartels*[1]; Frank-Peter Schimansky[2]; Rainer Gerling[2]; Helmut Clemens[3]; [1]Hamburg University of Technology; [2]GKSS Research Centre; [3]Montanuniversitaet Leoben

The occurrence of ordered ω-phase $B8_2$ in γ-Ti-45at%Al with 5-10at% Nb and with or without small carbon additions was investigated. For the preparation of the alloys a powder metallurgical approach was used. The formation of ω-phase was observed and analyzed by X-ray diffraction and SEM/EBSD. In Ti-45at%Al-10at%Nb the B2-phase transforms into ω-phase during cooling after HIP at 1280°C. With ≤7.5at% Nb the ω-phase occurs during isothermal compressive deformation at temperatures around 800°C. Here the supersaturated α_2-phase after HIP transforms into ω-phase. Under compression stress at 800°C the transformation is fast and needs only some minutes to take place. Without applied stress also a transformation of α_2 to ω-phase is observed at 800°C, but it takes several 100 hours. The amount of ω-phase increases with content of Nb. Otherwise, carbon additions stabilize the α_2-phase and thus hamper its transformation into ω-phase.

11:00 AM

A Numerical Model for the Effect of Nb on the Lamellar and Massive Transformation in Ti-Al-Nb Alloys: *Amin Rostamian*[1]; Alain Jacot[1]; [1]Computational Materials Laboratory, Ecole Polytechnique Fédérale de Lausanne, Switzerland

A phenomenological modelling approach has been developed for the description of the massive and lamellar microstructures which form during cooling of TiAl-based alloys from the α phase region at high and moderate cooling rates, respectively. The nucleation of both massive and lamellar γ is described by classical heterogeneous nucleation theory which takes into account that nuclei are formed predominantly at α grain boundaries. The growth rate of the massive phase is calculated with an expression for interface-controlled reactions, while the thickening rate of the lamellae is described with a modified multi-component Zener model of precipitation. The model permits to investigate the influence of nominal composition and cooling rate on the proportion of massive and lamellar γ in the microstructure. The effect of Nb additions on the transformation kinetics is discussed and calculated CCT diagrams for different Nb contents are compared with experimental data published in the literature.

11:20 AM

Understanding the Massive Transformation in TiAl Alloys Using End Quenching: *H. Saage*[1]; D. Hu[1]; H. Jiang[1]; Michael Loretto[1]; X. Wu[1]; [1]University of Birmingham, IRC in Materials

The influence of composition and of other variables such as cooling rate and grain size on the massive transformation in a wide range of TiAl alloys has been studied with the aim of developing alloys that can transform massively on air cooling rather than on quenching. The main experimental tool which has been used is end quenching using the Jominy technique. Examination of longitudinal samples taken from different alloys reveals in a particularly simple way the factors which influence the transformation. Optical and analytical scanning and transmission electron microscopy have been used to characterise the microstructures of TiAl alloys with limited additions of alloying elements and alloys with significant additions of Ta and Nb with a range of oxygen contents and different grain sizes. The observations made on the Jominy samples allow an understanding of the sensitivity of the transformation behaviour to all of these experimental variables and it has been shown that 25mm diameter samples of Ta-containing TiAl which have low oxygen contents (below 500wtppm)

can transform through thickness when air-cooled. These observations will be discussed in terms of the possibility of using the massive transformation to refine the microstructure of cast components of TiAl.

11:40 AM

Microstructure and Compressive Property in Aluminum-Titanium-Vanadium Ternary Alloys with Phase Constitutions Containing γ Intermtallics and β Phase: *Tohru Takahashi*[1]; Koshiro Otsuka[2]; Yohji Kojima[2]; Yukiko Toyoda[2]; Daisuke Ashida[2]; [1]Department of Mechanical Systems Engineering, Tokyo University of Agriculture and Technology; [2]Graduate Student, Tokyo University of Agriculture and Technology

Microstructure and compressive behavior have been investigated on several aluminum-titanium-vanadium ternary alloys whose chemical compositions are varied along a conceivable conjugate line spanning between γ single phase and β single phase. The $Al_{50}Ti_{40}V_{10}$ alloy (V10) with the subscript numbers showing the atomic fraction of each component, contained a small amount of β besides the γ phase. The $Al_{40}Ti_{30}V_{30}$ alloy (V30) showed a fine-grained dual phase microstructure of about a half-and-half mixture, and the $Al_{35}Ti_{25}V_{40}$ alloy (V40) was a coarse-grained β material. At an ambient temperature the V30 and V40 alloys were much stronger than the V10 alloy. But the harder phase intechanged from β to γ at around 1000K as the temperature increased. Accordingly, the compressive creep rate at elevated temperatures became much lower in V10 alloy.

Ultrafine-Grained Materials: Fifth International Symposium: Deformation Mechanisms

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Yuri Estrin, Monash University and CSIRO Melbourne; Terence Langdon, University of Southern California; Terry Lowe, Los Alamos National Laboratory; Xiaozhou Liao, University of Sydney; Zhiwei Shan, Hysitron Inc; Ruslan Valiev, UFA State Aviation Technical University; Yuntian Zhu, North Carolina State University

Wednesday AM — Room: 395/396
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Zhiwei Shan, Hysitron Inc; Hans Fecht, Ulm University; Tongde Shen, Los Alamos National Laboratory; Igor Alexandrov, UFA State Aviation Technical University

8:30 AM Invited

Mechanisms of Deformation Behavior of Ultrafine-Grained Ti: *Igor Alexandrov*[1]; Roza Chembarisova[1]; Vil Sitdikov[1]; [1]Ufa State Aviation Technical University

A grain size is known to be one of the factors which define mechanical properties of metallic materials. At the same time the mechanisms which regulate the deformation behavior of bulk ultrafine-grained (UFG) metals produced by the severe plastic deformation method are still a subject for intensive study and fixed ambiguously. The report presents the developed model and the results of its application for kinetic modeling of the deformation behavior of UFG CP Ti. Conservative slip and non-conservative climb of dislocations, deformation twinning, dynamic strain ageing are considered as potential deformation mechanisms. A wide temperature-rate range of deformation modes is considered. Conclusions about the role of the investigated mechanisms in the appearance of the peculiarities of the deformation behavior of UFG CP Ti are made.

8:50 AM

Microstructural Refinement of Bismuth-Antimony Alloy by Severe Plastic Deformation Processing: K. Hartwig[1]; *Jae-Taek Im*[1]; Jeff Sharp[2]; [1]Texas A&M University; [2]Marlow Industries, Inc.

Cast bismuth (Bi) antimony (Sb) alloy was deformed by equal channel angular extrusion (ECAE) to refine the microstructure. The material under study is used in thermoelectric cooling applications, and the objective of the study was to improve the thermoelectric figure of merit and mechanical properties by grain refinement. In the work reported, twelve millimeter diameter bars of Bi10Sb were encapsulated in square cross section aluminum 6061 alloy containers and heated to the processing temperature. The composite bars were then placed in a warm tool and extruded through a 90 degree angle die isothermally. Processing variables included punch speed, extrusion temperature, multipass route and exit channel area reduction ratio. Post extrusion material characterizations included optical microscopy, x-ray diffraction, energy dispersive spectroscopy, wavelength dispersive spectroscopy and scanning electron microscopy. Texture evolution was analyzed using the {006} reflection plane to identify the orientation of the basal poles. The cast microstructure was equiaxed, had a nominal grain size of one to three millimeters, and showed substantial microsegregation. Severe plastic deformation above the recrystallization temperature is shown to break down the cast grains into a bimodal microstructure consisting of fine-grained (5-30 micron) and coarse-grained (50-300 micron) regions. It is noteworthy that further grain refinement and the elimination of the bimodal microstructure do not progress rapidly beyond the first pass. Texture results show that route C processing gives a stronger texture than route A processing, and that in both cases, the basal-plane poles become aligned with the shear direction. Reducing the exit channel area is seen to encourage a much stronger texture than ECAE processing alone.

9:05 AM Invited

Deformation and Toughening Mechanisms of Nanograins – Lessons from Nature: *Xiaodong Li*[1]; [1]University of South Carolina

Seashells are natural nanocomposites with superior mechanical strength and toughness. What is the secret recipe that Mother Nature uses to fabricate seashells? What roles do the nanoscale structures play in the inelasticity and toughening of seashells? Can we learn from this to produce seashell-like nanocomposites? Here we will limit our focus to nacre (mother-of-pearl). The presentation summarizes the recent discovery of nanoparticles "nanograins" in nacre and elucidates the roles that these nanograins play in nacre's toughness. It was found that rotation and deformation of aragonite nanograins are the two prominent mechanisms contributing to energy dissipation in nacre. The biopolymer spacing between nanograins facilitates the grain rotation process. The digital image correlation technique was used to map local, nanoscale strains to elucidate deformation and fracture mechanisms. This presentation also presents future challenges in the study of nacre's nanoscale structure and mechanical properties.

9:25 AM

Multiple Deformation Mechanisms in Bulk Nanocrystalline Silver: *Tongde Shen*[1]; Can Aydiner[1]; Donald Brown[1]; [1]Los Alamos National Laboratory

We monitored the stress-strain relations of nanocrystalline silver in tension and simultaneously recorded the synchrotron x-ray diffraction patterns. From the diffraction data, we deduced the grain sizes (GS), dislocation densities (DD), and deformation texture. Our results suggest that the deformation of nanocrystalline silver can be divided into three stages. In stage 1 (before yielding), strain increases GS but decreases the density of glissile dislocations. In stage 2 (after yielding but before stage 3), strain largely increases the stress, but only slightly decreases GS and increases DD. The change in both GS and DD is completely reversible during unloading, suggesting that resistance for the plastic deformation mainly comes from the rotation of grains, as evidenced by the fact that no deformation texture is observed. In stage 3, strain increases both the dislocation density and the deformation texture, indicative of the pile-up of dislocations, similar to the plastic deformation of coarse-grained metals.

9:40 AM

Orientation Splitting and Its Contribution to Grain Refinement during Equal Channel Angular Extrusion: *Yan Huang*[1]; Philip Prangnell[1]; [1]University of Manchester

Mechanisms of grain refinement during ECAE of a single-phase aluminium alloy have been studied using the high resolution EBSD technique. It was found that, in addition to the formation of shear-plane cell bands (CBs) and shear bands (SBs) by "simple shear", the development of deformation bands due to orientation splitting contributed significantly to the refinement of microstructure during the early stages of ECAE processing. "Regular" slab-like deformation bands and "irregular" transitional bands were observed after 1st pass; both developed boundaries of high misorientations. During 2nd pass deformation, moderate orientation splitting took place within the deformation bands, although new deformation bands were not detected. With increased strains, orientation splitting tended to occur within small volumes of material, assisting the evolution of the shear plane CBs and SBs into a fibre structure dominated by boundaries of high misorientations. The crystallographic features of the orientation splitting were examined.

9:55 AM
Plastic Deformation of Fully Dense Nanocrystalline Material: *Hans Fecht*[1]; Youlia Ivanisenko[2]; [1]Ulm University; [2]Research Centre Karlsruhe

We present results on the fundamentals of plastic deformation in fully dense bulk nanocrystalline metals with a mean grain size between 10 and 30 nm prepared by an innovative and new combination of Inert Gas Condensation (IGC) and subsequent Severe Plastic Deformation (SPD) technique. The model system investigated is Pd (fcc) at the first stage and several single-phase Pd alloys. The experiments focus on mechanical tests using new testing equipment for miniaturized specimens. The aim is to obtain a significantly improved database of materials behaviour for these alloys at this very small grain size, since this has previously only been explored sporadically, and to elucidate and describe the microscopic mechanisms that mediate the deformation. The obtained results are important not only for fundamental material science, but also allows to elaborate the principles of development of technically feasible nanostructured materials providing the highest level of strength and ductility of these materials.

10:10 AM Break

10:25 AM Invited
Mechanical Properties and Deformation Mechanisms in Ultrafine-Grained Metals: *Heinz Werner Höppel*[1]; Johannes May[2]; Doris Amberger[2]; Mathias Göken[2]; [1]Institute of General Materials Properties WW I; [2]University of Erlangen-Nuernberg

Ultrafine-gained (UFG) metals exhibit very promising mechanical properties under monotonic loads: High specific strength is paired with a relatively high ductility. Although, some authors reported a strongly increased ductility (elongation to failure), others observed a reduced ductility compared to the CG counterparts. The enhanced ductility has been found so far for UFG Cu and Al and Al-alloys. In all these cases, the ductility increases as the strain rate decreases. Unfortunately, the deformation mechanisms behind are currently unclear and are in the centre of the scientific discussion. Grain boundary sliding, Coble creep as well as thermally activated annihilation processes are the proposed mechanisms. By reviewing relevant results published in literature on SPD-processed and PED-materials and also analysing new results obtained on UFG Aluminium alloys the objective has been to clarify that point. The proposed mechanisms are also discussed with respect to the cyclic deformation behaviour of UFG materials.

10:45 AM
In Situ TEM Compression Tests on FIB Fabricated Single Crystal Ni Pillars: *Zhiwei Shan*[1]; Raj Mishra[2]; S.A. Syed Asif[1]; Oden Warren[1]; Andy Minor[3]; [1]Hysitron Inc; [2]General Motors; [3]Lawrence Berkeley National Laboratory

Single-crystal Ni pillars of nominal diameters between 150 and 400 nm were micromachined using focused ion beam (FIB). TEM observations showed that the pillars had a high density of defects after FIB processing and a side wall taper angle of about 4.5 degree. In-situ TEM compression tests using a diamond flat punch were performed on pillars of several different sizes. Our observations showed that: i) dislocation density always decreased dramatically during the deformation test and in some cases a dislocation-free pillar was the end result; ii) the high resolution load vs. displacement curves exhibit extremely irregular profile and were very sensitive to the loading conditions and the sample size; iii) Unlike those bulk materials, the strain hardening observed in our work resulted from the dislocation starvation. The underlying physical mechanisms controlling the nanoscale pillars during deformation will be discussed in light of the above findings.

11:00 AM Invited
Dislocation Dynamics in Nanocrystalline Nickel: *Scott Mao*[1]; Zhiwei Shan[1]; Eric Stach[2]; Jorg Wiezorek[1]; David Follstaedt[3]; James Knapp[3]; [1]University of Pittsburgh; [2]Purdue University; [3]Sandia National Laboratories

It is believed that the dynamics of dislocation processes during the deformation of nanocrystalline materials can only be visualized by computational simulations. Here we demonstrate that observations of dislocation processes during the deformation of nanocrystalline Ni with grain sizes as small as 10nm can be achieved by using a combination of in situ tensile straining and high-resolution transmission electron microscopy. Trapped unit lattice dislocations are observed in strained grains as small as 5 nm, but subsequent relaxation leads to dislocation recombination.

11:20 AM
The Role of Twinning in UFG Fcc and Hexagonal Metals Determined by X-Ray Line Profile Analysis: Levente Balogh[1]; *Tamas Ungar*[1]; [1]Eotvos University

The appearance of stacking faults or twin boundaries in coarse grain materials is determined by the stacking faults energy (SFE). In most of the pure fcc or bcc metals the SFE is so large that plastic deformation is carried almost solely by dislocation activity. In submicron or nanometer grain size materials the creation and/or mobility of dislocations can be so difficult that faulting or twinning can become an alternative channel for carrying of plastic deformation. Faulting and twinning has recently been incorporated into the line profile analysis procedure: CMWP. The extended procedure, eCMWP is applied to HPT processed Al alloys, pure Ni, Cu and Cu-Zn alloys and ECAP processed Ti. The frequency of twin boundaries is determined and discussed in correlation with dislocation density, crystallite or subgrain size, size-distribution and mechanical properties.

11:35 AM
Dynamic Deformation Behavior of Ultrafine Grained Materials Produced by ARB and Subsequent Annealing: *Naoki Takata*[1]; Yoshitaka Okitsu[2]; Nobuhiro Tsuji[1]; [1]Osaka University; [2]Honda R&D Company, Ltd.

For automobile applications, dynamic deformation behaviors of materials are important to guarantee the safety in collision. In the present study, we fabricated the ultra-low carbon steel and pure aluminum sheets with various grain sizes ranging from 0.2 to 10 micrometer through accumulative roll bonding (ARB) and subsequent annealing at various temperatures. Mechanical properties of these materials were examined at various strain rates ranging from 10-2 s-1 to 103 s-1 for clarifying the dynamic deformation behaviors of the ultra-fine grained materials. The flow stress in the dynamic deformation was much higher than that in the quasi-static deformation. It was found that yield-drop phenomenon of the ultra-fine grained materials significantly depended on the strain rate. It could be concluded that the dynamic deformation behavior of the ultra-fine grained materials was attractive compared with that of the same materials with conventional grain size.

11:50 AM
Strain Rate Sensitivity of Ultrafine-Grained Copper with Nano-Scale Twins: *Lei Lu*[1]; Ming Dao[2]; Suresh Subra[2]; [1]Chinese Academy of Science, Institute of Metal Research; [2]Massachusetts Institute of Technology

We have conducted systematic experimental studies of the effects of nano-scale twin concentration, at a fixed ultra-fine grain size, on the deformation and ductility of ultrafine-grained copper prepared by pulsed electrodeposition technique. It is demonstrated that increasing the nano-scale twin density, while keeping the grain size fixed, results in significant increases in strength and strain-rate sensitivity. It is also found that the activation volume associated with this deformation process significantly decreases with increasing twin volume fraction. These results point to the trend that refining structural dimensions through the introduction of nano-scale twins has the same effect on strength and hardness as grain refinement. In addition, nano-scale twins are also found to provide enhanced ductility. Results of uniaxial tension, strain-rate jump, and instrumented nanoindentation tests will be reported in this presentation and some related modeling results will be briefly referred to with respect to the possible deformation mechanisms.

12:05 PM
Effect of Initial Morphology on Low-Temperature Superplasticity of Submicrocrystalline Ti-6Al-4V: *Young Gun Ko*[1]; Dong Hyuk Shin[2]; Chong Soo Lee[3]; [1]Massachusetts Institute of Technology; [2]Hanyang University; [3]Pohang University of Science and Technology

A present study was made to investigate the superplastic deformation behavior of ultra-fine grained (UFG) Ti-6Al-4V in relation to its initial morphology of alpha/beta phase. Ultra-fine grained microstructures were produced via equal-channel angular (ECA) pressing at 873 K using two different initial microstructures, i.e., equiaxed and lamellar microstructures. After imposing an effective strain up to 4, alpha and beta grains in both microstructures were significantly refined to ˜ 300 nm in diameter, and however, beta-phase grains were more uniformly distributed in the initial lamellar microstructure resulting in higher area fraction of alpha/beta interface than the initial equiaxed structure. The flow behavior of UFG Ti-6Al-4V alloy was well described by the constitutive equations which consisted of grain matrix deformation and grain/phase boundary sliding. It was found that higher superplasticity can be obtained in the UFG microstructure when using the fine lamellar microstructure as an initial material.

12:20 PM Award Ceremony for Posters

2008 Nanomaterials: Fabrication, Properties, and Applications: Characterization and Theory

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Nanomaterials Committee
Program Organizers: Wonbong Choi, Florida International University; Seong Jin Koh, University of Texas at Arlington; Donna Senft, US Air Force; Ganapathiraman Ramanath, Rensselaer Polytechnic Institute; Seung Kang, Qualcomm Inc

Wednesday PM Room: 273
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Raju Ramanujan, Nanyang Technological University; Ganapathiraman Ramanath, Rensselaer Polytechnic Institute

2:00 PM Invited
Understanding Mechanisms of Growth of Nanostructures during Wet Chemical Synthesis: *N. Ravishankar*[1]; [1]Indian Institute of Science

In spite of their simplicity, most wet chemical synthetic methods are too reagent-specific and there is very limited fundamental understanding of nanostructure growth. While control of shape during synthesis is reasonably well understood, understanding the general mechanisms of shape control are still elusive. In this talk, I shall illustrate two examples of anisotropic shapes (1-D nanowires and 2-D platelets) and discuss the mechanisms of their formation. In particular, I will illustrate that the mechanism of shape control for the formation of 2-D crystals is directly related to the driving force available for interface motion and is far more general that imagined earlier. The proposed mechanism is applicable for a variety of situations ranging from wet chemical synthesis, vapor phase methods and the formation of inorganic phases by biomineralisation. Such an analysis provides predictive capabilities and enables rational synthesis of 2-D nanostructures.

2:30 PM Invited
Antenna Effects in Semiconducting Nanowires: *Peter Eklund*[1]; Jian Wu[2]; Q. Xiong[3]; G. Chen[4]; [1]Pennsylvania State University, Department of Materials Science and Engineering; Department of Physics; [2]Pennsylvania State University, Department of Materials Science and Engineering; [3]Harvard University, Department of Chemistry and Chemical Biology; [4]Honda Research America

Novel antenna effects have been observed in polarized Raman and Rayliegh backcattering experiments on individual GaP nanowires. The results we observe in GaP should be perfectly general and should appear in all semiconducting nanowires studied under similar conditions. They are fundamentally important for electrooptic applications of nanowires. The polarized Rayleigh experiments allow us to measure directly the internal electric field effects in the nanowire, whereas the polarized Raman scattering intensity is a more complicated interplay between the symmetry of the phonon scattering and the dipolar nature of the antenna fields. As a result of the antenna effects, and consistent with very high internal electric fields, we can observe stimulated backscattering that exhibits a nonlinear dependence on the laser intensity. Our analysis of the data involves calculations of the internal electric field using the Discreet Dipole Approximation (DDA). From experiment and theory, we show that the antenna effects dominate for small diameter nanowires (d<40 nm), but isolated higher order resonances at much larger nanowire diameters are also possible.

3:00 PM
Chemical Synthesis and Magnetic Properties of Cubic Morphology Fe<3>Pt Nanoparticles: *Mohammad Shamsuzzoha*[1]; Madhuri Mandal[1]; David Nikles[1]; [1]University of Alabama

Fe<3>Pt nanoparticles of cubic crystal morphology with sizes in the order of 40 nm were prepared by the use of TX-100 micelles template during the reduction and decomposition of platinum acetylacetonate and iron pentacarbonyl respectively. The nanoparticles thus prepared have an ordered simple cubic structure and are supermagnetic. However, upon annealing with progressively increasing temperature, the nanoparticles showed a drastic drop in the value of magnetic moment at 47°C. This anomalous magnetic behavior is attributed to the occurrences of the Curie temperature for the particles. These cubic morphology particles owing to their facted morphology as well as of magnetic properties appear a suitable candidate for the hyperthermia therapy in cancer treatment.

3:15 PM
Structural and Thermodynamical Study of CoPt Nanoparticles: *Yann Le Bouar*[1]; Christian Ricolleau[2]; Damien Alloyeau[3]; Cyril Langlois[2]; Annick Loiseau[3]; T. Oikawa[4]; [1]Laboratoire d'Etudes des Microstructures, Centre Nationals de la Recherche Scientifique; [2]LMPQ, Université Paris 7; [3]Laboratoire d'Etudes des Microstructures, Office National d'Etudes et Recherches Aérospatiales; [4]Jeol Ltd.

The present work is focused on the study of the size effect on the thermodynamical behavior of CoPt nanoparticles. Pulsed laser deposition technique (PLD) is used to synthesize nanoparticles of controlled composition and size, on amorphous carbon or alumina. To characterize the particles, we use conventional TEM, nanoprobe electron diffraction, High Resolution Transmission Electron Microscopy (HRTEM) and Energy Dispersive X-ray Analysis (EDX). The crystal parameters of the particle, determined by electron diffraction, is found proportional to the composition of the particle. The 3D morphology of the nanoparticles has been determined using focal series and electron tomography experiments. The particles (from 2 nm to 12 nm) are slightly faceted and exhibit ellipsoidal shapes elongated in the plane of the substrate. Finally, we study the order-disorder transition in the nanoparticles as a function of their size by annealing either ex situ or in situ in the microscope.

3:30 PM
Grain Growth Behavior and Consolidation of Ball Milled: *Rajeev Gupta*[1]; Raman Singh[1]; Carl Koch[2]; [1]Monash University; [2]North Carolina State University

Nanocrystalline iron-chromium alloys may provide considerable corrosion resistance, even at low chromium contents. However, processing of such alloys could be a challenge. This paper describes successful synthesis of nanocrystalline Fe-10%Cr alloy by ball-milling route. In the absence of suitable hot compaction facility, the alloy powder could be successfully compacted close to the desired density, by employing a step of prior annealing of the powder. Grain growth behaviour of Fe-10%Cr nanocrystalline alloy was investigated at 500, 600 and 700°C. At 500°C, no appreciable grain growth was observed, after the initial grain growth. However, sudden and rapid grain growth was observed after 90 at 600°C, and 30min at 700°C. The paper will present the grain growth kinetics and successful processing of nanocrystalline iron-chromium alloys, and preliminary results of their corrosion tests.

3:45 PM
Theory of the Large Melting Point Hysteresis of Ge Nanocrystals Confined within Silica: *Daryl Chrzan*[1]; I. Sharp[1]; Q. Xu[1]; C. Yuan[1]; C. Liao[1]; J. Ager[2]; E. Haller[1]; [1]University of California, Berkeley and Lawrence Berkeley National Laboratory; [2]Lawrence Berkeley National Laboratory

Recent experiments demonstrate that Ge nanocrystals confined within a silica matrix display an extremely large (470 K) melting-point hysteresis centered (approximately) on the bulk melting point of Ge. This behavior stands in marked contrast to bulk solids that, in general, cannot be superheated. Further, since the silica is amorphous, one cannot invoke lattice registry effects to explain the observations. We show that a classical nucleation theory is able to explain quantitatively experimental observations. The theory properly reflects the geometry of the confined nanocrystals, and the impact of this geometry on the nucleation kinetics. Further, the theory relies on generally accepted values for interface energies and latent heats. Thus, there is no need to assume that interface energies depend on nanocrystal radii, nor to invoke atomic scale effects to explain the observations. This work is supported by the U. S. Department of Energy under contract No. DE-AC02-05CH11231.

4:00 PM Invited
Understanding of Ultra-Low Thermal Conductivity Layered Crystals and Nanolaminates: *Pawel Keblinski*[1]; [1]Rensselaer Polytechnic Institute

Using molecular dynamics simulations and vibration mode analysis we investigate the mechanism underlying ultra-low thermal conductivity of layered crystals thin films observed in recent experiments (Chiritescu at al, Science 315, 351-353, 2007). In particular, we compare film size dependence of thermal conductivity of ordered and out-of-plane disordered layered crystals. The disorder, indeed, significantly reduces thermal conductivity. It also makes the film thickness dependence weak by comparison with that characterizing

ordered layered crystals, however, the film thickness dependence it is not completely eliminated. We will also discuss the potential of designing low thermal conductivity nanolaminates involving alternating layers of soft and hard materials.

4:30 PM
Specific Heat and Thermal Conductivity Measurements Parallel and Perpendicular to the Long-Axis of Cobalt Nanowires: *Nihar Pradhan*[1]; Huanan Duan[1]; Jianyu Liang[1]; Germano Iannacchione[1]; [1]Worcester Polytechnic Institute

We present specific heat (Cp)and thermal conductivity (κ) measurements on cobalt nanowires (CoNWs) parallel and perpendicular to the nanowire long-axis from 300 to 400 K. Parallel measurements were done by growing the nanowires within the highly ordered, dense packed, array of parallel pores in aluminum-oxide. Measurements perpendicular to the long-axis of CoNW were done by depositing a thin film of CoNWs within the calorimetric cell. The specific heat both parallel Cp and perpendicular Cp deviates at higher temperatures strongly from bulk, amorphous,powder cobalt. The perpendicular thermal conductivity follows a similar a bulk-like behavior,exhibiting a maximum value near 365 K indicating the onset of boundary-phonon scattering. The parallel thermal conductivity does not display any maximum but rather is a smooth increasing function of temperature over the range studied and appears dominated by phonon-phonon scattering.

4:45 PM
Nanoscale Properties Resulting from d States in Complex Metallic Alloys: Esther Belin-Ferré[1]; *Jean-Marie Dubois*[2]; [1]National Center of Scientific Research; [2]Institut Jean Lamour

Many complex compounds exist in alloys like Al-Mg, Al-Mg-Zn or Al-Metal and Mg-Metal. The electronic structure of their valence band was investigated using X-ray emission spectroscopy, which provides separately the electron energy distribution around each chemical species in the solid, averaging the data over all sites. We discuss the nanoscale properties originating from Al 3s,d and Mg 3s,d states in several complex alloys comparatively to the pure metals as well as to other simpler structures, thus evidencing the importance of Al and (or) Mg d-like states at the Fermi level. By comparison to simple alloys containing a transition metal, it is suggested that Al or Mg d-like states play a significant role in the formation of a pseudo-gap at the Fermi level and therefore contribute to select and stabilize complex structures based on icosahedral clusters of nanometer size.

5:00 PM
Structure and Hardness Evolution in Nanocrystalline LIGA Fabricated Ni-12at%W Alloy at 873 K: *A.S.M.A. Haseeb*[1]; Klaus Bade[2]; [1]University of Malaya; [2]Forschungszentrum Karlsruhe

Nanocrystalline Ni-W alloys have good potential in LIGA fabricated MEMS. Thermal stability of Ni-12 at%W alloy prepared on silicon wafer using PMMA resist irradiated by synchrotron x-ray, as well as on copper is investigated. The grain size of Ni-12 at%W alloy increases from 21 nm to 31 nm upon heating at 873 K for 48 hours. Grain growth exponent is found to be 2.25, which indicates an insignificant role of grain boundary diffusion. The microhardness of the alloy decreases from 705 to 420 HV. Under the same heating condition, the hardness of sulphamate Ni decreases from 324 to 92 HV. The initial cross sectional banded microstructure of LIGA test microcomponent persists during heating at 873 K for 48 hours. A comparison with literature data on nanocrystalline Ni and Ni base alloys reveals that Ni-12 at%W alloy possesses a much more stable structure at 873 K.

5:15 PM
Phase Formation in (Co0.95Fe0.05)89Zr7B4 Nanocrystalline Alloys during In-Situ Heating: *Ramasis Goswami*[1]; Matthew Willard[2]; [1]SAIC/Naval Research Laboratory; [2]Naval Research Laboratory

Nanocrystalline soft magnetic alloys are interesting for inductor applications due to their balanced magnetic properties resulting, in part, from their unique microstructure. Understanding the microstructure formation is an important for optimization of the magnetic properties. In this study, phase transformations in a (Fe0.05Co0.95)89Zr7B4 melt spun alloy have been investigated by in-situ heating in the transmission electron microscope. The sample in the as spun condition consists of fine BCC crystallites dispersed in amorphous matrix. At higher temperatures, above secondary crystallization, the amorphous matrix dissociates into Zr-intermetallics and the grains coarsen significantly. HRTEM on samples annealed at 550°C showed 8-10 nm grains, both BCC and FCC, are surrounded by with the residual amorphous matrix, forming a barrier for diffusion between grains that prevents the grain growth at intermediate temperatures. Z-contrast imaging showed the nanocrystalline pockets containing higher Zr, which stabilizes the amorphous region.

5:30 PM
Mechanical Properties of Nanocrystalline Cu-Sn Alloys Produced by Hot Rolling of Ball-Milled Powders: *Hee-Tae Jeong*[1]; Jae-Hyuk Shin[1]; Hyun-Joo Choi[1]; Donghyun Bae[1]; [1]Yonsei University

During the past decade, lots of interests have been given to the fabrication of bulk nanostructured metallic materials for structural applications. In the present study, the mechanical properties of hot-rolled Cu100-xSnx, x=5, 10, 15, and 22 wt.%, alloys were investigated. The powders for the consolidation process were produced by mechanical alloying. With a proper milling condition, the produced powders, in the range of Sn content up to 22 wt.%, were found to have single phase of α-(Cu,Sn) solid solution and the grain size of the powder was below 50 nm. Hot-rolling process was employed to fabricate the plates. The microstructures, mechanical properties, such as tensile strength and ductility, and strain rate effects, of the as-rolled and heat-treated plates and their deformation mechanism will be presented.

3-Dimensional Materials Science: Modeling and Characterization across Length Scales III

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Michael Uchic, US Air Force; Eric Taleff, University of Texas; Alexis Lewis, Naval Research Laboratory; Jeff Simmons, US Air Force; Marc DeGraef, Carnegie Mellon University

Wednesday PM — Room: 286
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Eric Taleff, University of Texas; Jaimie Tiley, US Air Force

2:00 PM Invited
3D X-Ray Microscopy Investigation of Materials Microstructure: *Bennett Larson*[1]; [1]Oak Ridge National Laboratory

X-ray structural microscopy using polychromatic x-ray microbeams now provides the capability to measure local crystal microstructure and strain in deformed materials with submicron spatial resolution in three-dimensions. 3D x-ray microscopy measurements performed on 3D grids lead to dislocation density tensors for characterizing deformation in terms of geometrically necessary dislocation (GND) densities. 3D x-ray microscopy measurements made on single crystal copper indented with a 100 micron radius spherical indenter will be presented in terms of 3D spatially resolved rotation maps and dislocation density tensors. These measurements will be compared with (collaborative) computer simulations for spherically indented copper to illustrate the connection between experiment and computations. Exploiting the confined deformation volumes associated with indentation provides a direct and absolute linkage between 3D x-ray microscopy measurements and computer simulations on mesoscopic length scales. Research sponsored by the DOE BES-DMS&E and performed at the DOE supported APS at ANL and SHARE at ORNL.

2:30 PM
Non-Destructive 3-D X-Ray Diffraction Imaging at the Nanoscale: *Barry Muddle*[1]; Rouben Dilanian[2]; Nadia Zatsepin[2]; Andrei Nikulin[2]; [1]Monash University, Centre for Design in Light Metals; [2]Monash University, School of Physics

Approaches to non-destructive, 3-D x-ray imaging with nanoscale (10-30nm) spatial resolution typically fall into two categories: (i) those employing refractive x-ray lenses to deliver 3-D microscopy in scanning or full-field transmission modes, and (ii) those using an iterative oversampling algorithm to retrieve phase from multiple 2-D maps of diffracted intensity to deliver lens-

free, coherent diffractive imaging. Both approaches require highly coherent x-rays and tomography is necessary for full 3-D imaging. The present alternative employs a crystal analyser to collect diffracted intensity as a function of spherical coordinates in reciprocal space and an iterative algorithm is used to reconstruct the 3-D form of the average diffracting object with a spatial resolution of a few nanometres. The technique is insensitive to the coherence of the x-ray beam, and 3-D reconstruction is possible without tomographic synthesis. Application to characterisation of nanoparticulate in a range of dispersed-phase nanocomposite structures will illustrate the approach.

2:50 PM

Reconstructing 3D Microstructure from X-Ray Diffraction Microscopy Data: *Robert Suter*[1]; Chris Hefferan[1]; Frankie Li[1]; Robert Moore[1]; Brian Tieman[2]; Ulrich Lienert[2]; [1]Carnegie Mellon University; [2]Argonne National Laboratory

X-ray diffraction microscopy makes it possible to non-destructively measure volumes of microstructure. As such, it is possible to perform dynamic measurements of the response of ensembles of grains to stimuli such as heat or stress. Forward modeling methods for analyzing the data use computer simulation to adjust simulated local crystallographic parameters to match observed diffraction. This method is flexible since multiple phases and complex scattering can be incorporated but it is also computationally intensive. We describe successful reconstruction of an aluminum polycrystal. We interpolate layer-by-layer reconstructions onto a cubic array of voxels, mesh boundaries with triangles, and use smoothing algorithms to remove steps and optimize grain boundary locations. The resulting map includes grain geometries, boundary types, and internal structure such as orientation gradients. The feasibility of extending this type of analysis to large volume data sets and the necessity of parallel computing architectures will be discussed.

3:10 PM

NanoCT: Analysis of Internal Microstructures with High Resolution Computed Tomography: *Kathleen Brockdorf*[1]; David Lehmann[1]; [1]Phoenix X-ray

High-resolution X-ray tomography allows the visualisation and spatial analysis of internal microstructures of small samples. The nanotom of phoenix|x-ray is the first 180 kV nanofocus computed tomography system tailored specifically to the highest-resolution applications in the fields of material science, micro electronics, geology etc. with exceptional voxel-resolutions down to <0.5 microns. By granting the user the ability to navigate the internal structure of an object slice-by-slice in a non-destructive manner, the system creates new analysis capabilities thus far have been unreachable: Any internal detail showing a contrast in material, density or porosity can be visualized and distances can be measured. By this way different metal phases of alloys or the texture of fibres in composite materials with only a few microns diameter may be analysed. This opens a new dimension of the 3D-microanalysis and will partially substitute destructive methods like traditional slicing – saving costs and time per sample inspected.

3:30 PM Break

3:50 PM

Atom Probe Tomography: Reconstructing and Quantifying Microstructural Features from Atoms: *Michael Miller*[1]; [1]Oak Ridge National Laboratory

Major advances have occurred in atom probe instrumentation of over the last few years. Datasets containing the atomic coordinates and the mass-to-charge state ratios of almost a billion atoms have been generated. Although standard visualizations methods such as atom maps and isoconcentration surfaces provide valuable information on the phase distribution and morphology, it is difficult to extract quantitative information on the distribution of solutes and sizes of the microstructural features present directly from these methods. Therefore, statistical based methods have been developed to perform these types of analysis. The concepts underlying these methods and the issues of implementing them at the atomic level will be discussed. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy.

4:10 PM

Subnanoscale Characterization of a Multicomponent Steel by 3-Dimensional Atom-Probe Tomography: R. Prakash Kolli[1]; *David Seidman*[2]; [1]Northwestern University, Department of Materials Science and Engineering; and Nalco Company; [2]Northwestern University, Department of Materials Science and Engineering and Northwestern Center for Atom Probe Tomography (NUCAPT)

We describe briefly the atom-probe tomography (APT) methodology, which possesses a resolution of ca. 0.2 to 0.5 nm, and the compositional and morphological results that can be obtained using it. We then present results from a precipitation hardened, multicomponent, high-strength low-carbon (HSLC) ferritic steel being developed for naval applications. We illustrate that the mean radius, $R(t)$, and number density, $N_r(t)$, of the precipitates can be measured directly thereby characterizing their morphology. Furthermore, we demonstrate that the elemental concentration profiles, solute partitioning, phase compositions, and supersaturations can be obtained; yielding detailed compositional information about the precipitates, matrix, and interfacial region. The acquired results are correlated to bulk mechanical properties (yield stress, ultimate tensile stress, plasticity at failure, Charpy V-Notch values) and strengthening mechanisms, precipitate growth and coarsening models, or can be used as input or to refine computational models such as the commercially available *Thermo-Calc* or *Precipi-Calc* programs.

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Microanalysis and Modeling of TiNi-Based Shape Memory Alloys with 3-D Local Electrode Atom Probe Tomography: *Matthew Bender*[1]; Gregory Olson[1]; [1]Northwestern University

3-D local electrode atom probe (LEAP) tomography is utilized to support the computational design of nanoscale precipitation-strengthened TiNi-based shape-memory alloys. Using LEAP tomography, the otherwise unattainable B2-L21 phase relations for Ni-Ti-Al-Zr alloys at 600°C are mapped. Results indicate that Zr addition has the unintended effect of stabilizing unwanted metastable Ni_4Ti_3 phase; therefore, phase relations of Ni-rich precipitates are also studied and optimized alloy compositions are designed to avoid them. Precipitate growth and coarsening of aluminide precipitates is investigated with LEAP tomography, revealing that optimal microstructures occur in the early stages of precipitation rather than at equilibrium. Using experimentally calibrated models, a superelastic biomedical Ni-Ti-Al-Zr alloy for the application of stenting a superficial femoral artery is designed to have low-misfit coherent precipitation strengthening, desired transformation temperatures, and enhanced radiopacity absorption, within the constraint of practical processing temperatures. Mechanical testing validates the expected high-performance properties of designed alloys.

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Pulsed-Laser (3D)Atom Probe Tomography of Spinodal Decomposition and Transition Carbide Formation during Tempering of Low C-High Si-Low Ni Martensitic Steel: *Padmanava Sadhukhan*[1]; Don Sherman[2]; Gregory Olson[3]; [1]Northwestern University; [2]Caterpillar Inc; [3]Northwestern University, Questek Innovations LLC

Pulsed-Laser Atom-Probe(AP) tomography has been used to study kinetics of spinodal decomposition/transition e'-carbide formation in 0.29C-1.52Si-0.19Ni low-alloy tough-steel. The as-quenched martensite specimens were (1)aged at room-temperature for prolonged period, and (2)tempered at 215C for various times. Carbon segregation was analyzed using (3D) Local-electrode AP(LEAP™) carbon distribution maps, with an average of 35-40 Million ions collected. Composition of carbon-enriched regions,modulations and transition-carbides have been analyzed using both cylindrical 1D-composition profile method along different axes and proximity-histogram concentration profile method. The latter separates the interfacial regions from the precipitate/concentrated cores, with the precipitates binned in 1nm3 volume increments. Analysis of concentration-profiles of these carbides have been compared to earlier observed modulations/transition carbides using APFIM/TEM in high-Carbon Fe-Ni-C martensites. Steel specimens were provided by AMT, Caterpillar. Financial support from ONR(N00014-01-1-0953) is acknowledged. Atom-probe analyses were performed at Northwestern-University Center for Atom-Probe Tomography(NUCAPT). LEAP™ tomograph was purchased with funding from NSF-MRI(DMR-0420532) and ONR-DURIP(N00014-0400798) programs.

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Microstructure Evolution of the High Carbon Pearlitic Steel Wires by Using LA-3DAP: *Yang Yo Sep*[1]; Park Chan Gyung[1]; Bae Jong Gu[2]; [1]POSTECH MSE; [2]KISWIRE Research and Development Center

A three dimensional knowledge of the atom-scale structure is necessary for improving the design and understanding of the modern materials. Laser assisted atom probe is used to obtain atom by atom 3D reconstruction to characterize three versions of several alloys in high carbon steel wires. Since steel wires experience the heavy drawing process, the carbon atoms in cementite moved into ferrite matrix. It is worthy to find the reasons why carbon dissolution is occurred, since the cementite dissolution is strongly related with mechanical properties, especially fatigue resistance. To identify the carbon movement in the cementite and ferrite, the steel wires were fabricated depending on the carbon content and drawing condition. The carbon atoms moved into ferrite with increase of drawing strain, regardless carbon contents. It revealed that the fatigue resistance of all steel wires decreased with increase of carbon content and drawing strain resulted from the increase of cementite dissolution.

Alumina and Bauxite: Operations

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Sringeri Chandrashekar, Rio Tinto Aluminium Limited; Peter McIntosh, Hatch Associates

Wednesday PM — Room: 296
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Milind Chaubal, Sherwin Alumina Company

2:00 PM Introductory Comments

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The Kinetics of Quartz Dissolution in Caustic Solutions and Synthetic Bayer Liquors: Bingan Xu[1]; Christine Wingate[1]; *Peter Smith*[1]; [1]CSIRO

The Oku-Yamada (O-Y) equation (1971) has successfully estimated quartz attack for many bauxites. However it is often used outside its development conditions (particularly temperature). This paper examines the kinetics of quartz attack for quartz in pure caustic and synthetic Bayer liquor, and for an Australian bauxite in synthetic liquor. Under typical conditions, the attack of quartz in pure caustic was well modeled by the O-Y equation but it tended to over-estimate attack in synthetic liquor. The equation also over-estimated attack on coarse particles and under-estimated attack on fine particles, suggesting quartz surface area (not a factor in the O-Y equation) is important. Quartz attack on bauxite was modeled well suggesting that the quartz size distribution was comparable to that in Oku and Yamada's bauxite. Measured quartz attack at higher temperatures (240-280°C) showed the same trends as lower temperatures, suggesting the equation can be used for bauxite in this temperature range.

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Potential Improvements of Bauxite Atmospheric Digestion Circuit: *Alexander Suss*[1]; Nikolay Ivanushkin[2]; Irina Paromova[1]; Andrey Panov[1]; Anatoly Lapin[1]; Tatiana Gabrielyan[1]; [1]Russian National Aluminium-Magnesium Institute; [2]Russian Aluminum Company

The paper describes the work on elaboration of two - stages counter current digestion circuit for pure gibbsite bauxites from Fria deposit in Guinea. With the alumina recovery at the same level as for single stage digestion the transition to the 2 stages allows to obtain higher RP in the liquid phase for non-autoclave digestion in agitations. The expected production growth after implementation of 2 stages counter current digestion circuit at Friguia Alumina Refinery will be ~ 50 ktpy. The higher RP ratio increases the agglomeration capacity of green liquor with stronger hydrate crystals. It will allow to reduce the attrition index from ~ 33-35 to ~ 20%.

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Process Diagnosis Based on Neural Network Expert System for Gas Suspension Calcinations of Aluminum Hydroxide: *Dai fei Liu*[1]; Jie Li[1]; Feng qi Ding[1]; Zhong Zou[1]; Xiang tao Chen[1]; [1]Central South University

Stable and effective operations in the gas suspension calcination of aluminum hydroxide can save energy, increase yield and reduce exhaust emission. But this goal hasn't been reached because the roasting process is too complex. Until now there is few process diagnosis models discussed or built in the published literatures. This paper presents a process diagnosis system, which uses neural network and expert system, and comprises four parts, namely state analysis module, energy evaluation module, parameter optimization module and control guidance module. In the system the state analysis module is used to analyze process state, the energy evaluation module is to compute energy costs and the parameter optimization and control guidance module are to deduce other useful results and optimization parameters. The application of this system indicates that the identifying accuracy for abnormal process achieved 90% and temperature error was within ±5°C, real-time evaluation and optimization provided good guidance for practice.

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Review on the Quality of Alumina: Wangxing Li[1]; Zhanwei Liu[1]; Dalin Fan[1]; *Zhonglin Yin*[1]; [1]Zhengzhou Research Institute of Aluminum Corporation of China Limited

After China entered WTO, the important tasks facing our alumina industry are actively participating in international cooperation and competition in a global economy. With the development of aluminium smelting technology and greater attention given to the environmental protection, the quality requirements of alumina are more strictly. The effects of alumina quality on the aluminium smelting are discussed in this paper. The differences between domestic and foreign alumina products in chemical components and physical properties are also presented in this paper. The measures of improving the quality of alumina are explored, so the alumina products can meet the requirements of aluminium smelting.

Aluminum Alloys: Fabrication, Characterization and Applications: Composites and Foams

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials

Wednesday PM — Room: 293
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Subodh Das, Secat Inc; Weimin Yin, Williams Advanced Materials; Shridas Ningileri, Secat Inc

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Development of High-Strength Hypereutectic Al-Si Alloys by Nano-Refining the Constituent Si Phases: *Mohammad Shamsuzzoha*[1]; Frank Juretzko[1]; Anwarul Haque[1]; [1]University of Alabama

Among the structural light weight materials, Al-Si alloys have established itself as the most widely used Al-alloys for aerospace and automotive applications. Our recent efforts have demonstrated that it is possible to develop primary Si-phase free hypereutectic Al-Si alloys by a casting method. In the continuation of this effort, an Al- 17wt%Si alloy that has a microstructure in which silicon phase assumes nano sized fibrous morphology has been developed by directional casting. The fabrication and characterization of this unique Al-Si alloys are presented. The mechanical properties of this alloy based on first principle, are also discussed.

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Fabrication of Aluminum Composites with Gradient Cladding Interface by Continuous Casting: *Weiwen Zhang*[1]; Datong Zhang[1]; Yuanyuan Li[1]; [1]South China University of Technology

A method named double-stream-pouring continuous casting (DSPCC) was proposed to produce bimetallic composites with gradient cladding interface. The

characteristics of DSPCC and the differences between DSPCC and conventional continuous casting were highlighted. According to the trials in Al-Zn-Mg, Al-Cu-Mg, Al-Mn and Al-Mg-Si alloy systems, it is found that the interfacial zone dimensions can be controlled by adjusting the alloy composition and solidification processing parameters, such as the temperature of the melts, the casting speed and the depth of the submerged nozzle in the mold. A 2024/3003 composite having the equivalent corrosion resistance to 3003 alloy was prepared by DSPCC. After plastic deformation and T6 heat treatment, the tensile strength and yield strength of the composite are 2.56 and 4.05 times of that of 3003 alloy, respectively and the elongation of the composite is still as high as 21.6%.

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Influence of Magnesium and Silicon Content on Microstructures of In-Situ Mg_2Si/Al Composites: *Jing Qingxiu*[1]; Zhu Yinglu[1]; [1]Institute of Material and Chemistry Engineering, Jiangxi University of Science and Technology

In-situ Mg_2Si reinforced Al alloy composites were fabricated by common weight casting technique. Effects of different Mg and Si content and heat treatments on microstructures of materials were studied. The results indicate that effect of combined modification of rare earths and Strontium salt on primary a-A1 is very good, while not obvious on eutectic α-A1. Eutectic Mg_2Si nucleates at crystal boundaries, with shaping undeveloped Chinese characters-like in a hypoeutectic Al-Mg_2Si system which contains more Si element, while eutectic Mg_2Si is surrounded by α-A1 and grows to integrated Chinese characters-like shape in a nearly eutectic Al-Mg_2Si system in which little Si contained. Eutectic Mg_2Si is fined to particle or short cosh in a heat treatment process, and the size of primary Mg_2Si is also reduced at some extent, from about 15μm to 8μm.

3:00 PM

Viscosity Control of Foam Aluminum Melt and Study on Bubble's Stability: *Li Wei*[1]; Li Cao[1]; Shumei Li[2]; [1]Shenyang Ligong University, Materials Science and Engineering College; [2]Dandong University, Computer College

This is a study on preparing foam aluminum by powder metallurgy. In this research the raw materials are pure aluminum powder, pure aluminum powder added respectively carborundum particles, soot powder and carbon fiber. The microstructure of cell wall of foam aluminum, the viscosity and surface tension of melt are investigated. The bubble stabilities which are controlled through adding SiC particles, soot powder and carbon fiber are discussed, and the mechanism of bubble's stability is analyzed. The results showed that the homogeneity of bubble holes of foam aluminum is better which is obtained by adding SiC practical and carbon fiber into the raw materials. Observing SEM can indicate that SiC practical and carbon fiber distribute evenly on cell wall of foam aluminum. It is means that during foaming a lot of SiC practical and carbon fiber distribute diffusively in liquid aluminum melted. It increases the viscosity of melt so that stability of bubble is increased.

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Study on the Control of Foam Gradient by Foam Melting: *Wang Yong*[1]; Guangchun Yao[1]; Li Bing[1]; Cao Zuo-kun[1]; [1]Northeastern University

The solidification modes of foamed Al and its influence on the cell structure are studied, and the changes of volume and porosity of foamed melt during solidification process are discussed. It is demonstrated that the evolution of the cellular structure is influenced by different solidification mode in the process. The relation of the foam growth height with time is researched. The foam evolvement of the melt in the solidification phase is discussed. Some conclusions are obtained: the evolution of cellular structure is influenced by many processing factors such as viscosity, foaming time and solidification mode. Al foam melt is prone to solidify with planar front, using special solidification mode can control the gradient of the cellular structure effectively, and the bubble size has a certain relation with the cooling time.

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The Effect of Complex Modification and Melt Mixing Treatment on Microstructure in In-Situ Mg_2Si/Al-Si Composite: *Jing Qingxiu*[1]; Huang Xiaodong[2]; [1]Jiangxi University of Science and Technology, Institute of Material and Chemistry Engineering; [2]Jiangxi University of Science and Technology

The effect of $SrCl_2$ and RE complex modification and melt mixing treatment on microstructure in in-situ Mg_2Si/Al-Si composite was investigated in this paper. It is found that primary Mg_2Si particles have been greatly refined from 25μm to below 8μm with morphology changing from polygonal to quadrangle in the optimum melt mixing condition and with the optimum content of $SrCl_2$ and RE addition. The result also indicates that, with the increase of $SrCl_2$ and RE content, the pseudo-eutectic Mg_2Si is changed gradually from agglomerated coarse tablet to dispersed spots or slim bars. The growth of Mg_2Si follows not only the twin plane re-entrant edge (TPRE) mechanism, but also layer mechanism as well.

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Study on Sound Insulation Performance of Al Foam Sandwich Board: *Guoyin Zu*[1]; Guangchun Yao[1]; Hong Li[1]; [1]Northeastern University, School of Materials and Metallurgy

Sound reduction index of Al foam bare board and sandwich board was tested using standing wave method, the effect of density and thickness on acoustical behavior of Al foam sandwich board was studied. The results showed that, sound reduction index of Al foam sandwich board increased with the increase of sound wave frequency, sound reduction index during difference frequency was controlled by difference mechanism, it could be divided into three regions: rigidity and damp control region, quality control region and mixing influence region. With the increase of density and thickness of Al foam sandwich board, the sound insulation performance enhanced gradually, sound reduction index increased with the increase of sound wave frequency.

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Effect of Cold-Rolling Bonding Process on Microstructure and Properties of the Sandwich Aluminum Foil for Automobile Heat Exchanger: *Guoyin Zu*[1]; Ning Wang[1]; Jiuming Yu[1]; [1]Northeastern University, School of Materials and Metallurgy

The cold-rolling bonding process of sandwich aluminum foil for automobile heat exchanger was investigated, and the effect of deformation rate in the first pass, the clad thickness and the final annealing schedule on microstructure and properties of the sandwich foil was analyzed. The results showed that under deformation rate of 30%-50% in the first pass, the initial bonding can be gained between the clad A4045 and the core A3003, the upper clad thickness of the product was probably the same as the lower one. The final clad radio was influenced by deformation rate and initial thickness of the rolled piece; it increases with the increasing of deformation rate and initial thickness. The cladding foil had the best sagging resistance when the cold-rolling deformation rate was 25%-35%.

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Study on Powder Metallurgy Foaming Process of Rolled Precursor: *Guoyin Zu*[1]; Guangchun Yao[1]; Hong Li[1]; Liang Hao[1]; [1]Northeastern University, School of Materials and Metallurgy

The powder metallurgy foaming process of rolled precursor was investigated in this paper, evolvement process of high tightness core to foam structure was analyzed, efficiency approach to increase stability of foam structure was studied, and the optimal foaming process was obtained. The results showed that cracks and drainage during foaming were inhibited by heat treatment of foaming agent TiH_2 and proper quantities addition of Mg to body system. The optimum heat treatment way of TiH_2 was that heat preserving at 450°C for 1 hour, and Mg addition was 1wt.%. The optimum foaming temperature of rolled precursor was 620~630°C, ideal core foam structure was obtained when at 620°C for 4~6min.

Aluminum Reduction Technology: Fundamentals, Low Melting Electrolytes, New Technologies

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Aluminium Tech

Wednesday PM Room: 298
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Victor Buzunov, United Company «Russian Aluminum»

2:00 PM
Technical Contributions of the Late Warren Haupin – A Review: *Jay Bruggeman*[1]; [1]Alcoa Inc.

From his pioneering work in pot heat balance measurements and modeling to his fundamental studies on anode effects and electrode processes, the late Warren Haupin often laid a foundation of understanding for others to build on. This paper reviews Haupin's major technical contributions as a tribute to the man who impacted the aluminum industry so much.

2:25 PM
Top Heat Loss in Hall-Heroult Cells: *Xian Chun Shen*[1]; Margaret Hyland[2]; Barry Welch[3]; [1]CITIC International Cooperation Co., Ltd.; [2]University of Auckland; [3]Centre for Electrochemical and Mineral Processing, University of New South Wales

The top heat losses were measured in industrial Hall-Heroult cells, including heat losses from different cover materials, anode assemblies and off-gas. The effect of the top heat loss on cell energy balance was discussed.

2:50 PM
Electrolysis of Aluminum in the Low Melting Electrolytes Based on Potassium Cryolite: Yurii Zaikov[1]; Andrey Khramov[1]; Vadim Kovrov[1]; Vasily Kryukovsky[1]; Alexey Apisarov[1]; *Olga Tkacheva*[1]; Oleg Chemesov[1]; Nikolay Shurov[1]; [1]Institute of High Temperature Electrochemistry

The aluminum electrolysis in electrolytes with composition (mas.%): KF(47,37)-AlF3(52,63) with CR=1,3 and KF(49,0)-AlF3(49,0)-LiF(2,0) with CR=1,58 at temperature 750°C was carried out in the lab scale cell with liquid aluminum cathode. As anodes, metallic Al-Cu and Ni-Cu-Al-Fe or cermet Cu-Cu2O or ceramic SnO2 materials were used. The alumina concentration was kept on the 4,5 mas.% level. The region of anode current density was 0,4-0,6 A/cm2. Duration of experiments reached 96 hours. Temperature, voltage and current on the cell during electrolysis, voltage at the moment of current interruption were recorded. The most steady anode materials have proved to be Cu-Al(3 or 5 mas.%) and Cu2O-Cu(15 mas.%).

3:15 PM
Influence of CaF2 on the Properties of the Low-Temperature Electrolyte Based on the KF-Alf3 (CR=1,3) System: Alexander Dedyukhin[1]; Alexey Apisarov[1]; Alexander Redkin[1]; *Olga Tkacheva*[1]; Yurii Zaikov[1]; [1]Institute of High Temperature Electrochemistry

The low melting electrolyte based on the KF-AlF3 (CR=1,3) system is very prospective for development of the new energy saving aluminum production technology. The low electrical conductivity of the KF-AlF3 (CR=1,3) system can be increased by the LiF addition. Calcium fluoride is always brought into the electrolyte as an impurity of the alumina. The influence of CaF2 on the properties of the low melting electrolytes at temperature below 800°C is not studied properly. Therefore electrical conductivity, melting point and calcium fluoride solubility in the KF-AlF3 (CR=1,3) and KF-LiF-AlF3 (CR=1,3) systems within temperature range 700-800°C have been studied. The CaF2 solubility increases with LiF additions. The electrical conductivity dependence varies not linearly with the content of dissolved CaF2. The melting point of the electrolytes under study rises with the calcium fluoride concentration increase.

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Temperature of Primary Crystallization in Part of System Na3AlF6-K3AlF6-AlF3: Wang Jiawei[1]; *Lai Yanqing*[1]; Tian Zhongliang[1]; Li Jie[1]; Liu Yexiang[1]; [1]Central South University

Methods for testing temperature of primary crystallization in aluminum electrolyte melts were summarized, a classical thermo-analysis method was chosen, an experimental setup was built and its reliability was proved detailly. To get base datum of electrolytes and provide theoretical guidance for low temperature aluminum electrolysis, temperature of primary crystallization in part of system Na3AlF6-K3AlF6-AlF3 were tested, the ratio of K3AlF6 to K3AlF6+Na3AlF6 was 0~20wt.%, and AlF3 would occupy 0~30wt.% of total electrolytes weight. Results showed that, when AlF3 was below 20wt.%, adding of K3AlF6 decreased temperature of primary crystallization, but when above 20wt.%, the effect of K3AlF6 on it was not obvious. To the samples with ratio of K3AlF6 to K3AlF6+Na3AlF6 0, 10, 20, when AlF3 was below 18wt.%, every adding 1wt.%AlF3 would decrease approximate 3.49, 2.33 and 1.59 respectively, but when above 18wt.%, the effect of AlF3 on it became notable, every adding 1wt.%AlF3 would all decrease about 10.

4:15 PM
Electrical Conductivity of (Na_3AlF_6-40 wt%K_3AlF_6) - AlF_3 wt% Melts: Huang Youguo[1]; Lai Yanqing[1]; Tian Zhongliang[1]; *Li Jie*[1]; Liu Yexiang[1]; Yi Qingyu[2]; [1]Central South University; [2]Guangxi Normal University, School of Chemistry and Chemical Engineering

The electrical conductivity of Na3AlF6-K3AlF6-AlF3 melts was studied with a Continuously Varying Cell Constant technique (CVCC). The reliability of experiment device was validated by testing the electrical conductivity of KCl solution, molten KCl, and molten cryolite. To be used as a low temperature electrolyte for inert anodes, the melts contained 0, 20, 24, and 30 wt % AlF3 with 40 wt%K3AlF6 in Na3AlF6 + K3AlF6 mixture. The superheat was 20, 40, 60, and 80°C. The electrical conductivity data in the molten mixtures can be described by a simple equation of the Arrhenius type: $Ln\kappa = A + B/T\kappa = A + B/T$ where κ represents electrical conductivity (S•cm-1), T is temperature (K), A and B are constants for the measured system.

4:40 PM
Chemical and Electrochemical Reactions in the Al-Al2O3-Na2SO4 System at 927°C: *Xiao Yan*[1]; Marshall Lanyon[1]; [1]CSIRO

Solubilities of a-Al2O3, ZrO2, and Y2O3 in Na2SO4–based melts and aluminum interaction with molten Na2SO4 were studied using an equilibration technique. The Al2O3 solubility was determined to be 0.012 wt% in a pure Na2SO4 melt in air, increased with increasing Na2O content over 0.2–4 mol% Na2O. The ZrO2 and Y2O3 solubilities in the melts were below the detection limits. An Al2O3 layer formed on the solidified aluminum surface after immersion in fused Na2SO4 in argon for 24 hours, whereas Al-Na-S mattes were not detected due to their dissolutions into molten Na2SO4. Cathodic reactions at platinum, gold, and aluminum cathodes in the melts were also investigated using electrolytic cells with a graphite rod or SOFC-type anode. The results indicated that both aluminum and sodium were electrochemically deposited from the α-Al2O3 saturated electrolytes. The possibility of using molten Na2SO4 as an alternative solvent for Al2O3 in aluminum electrowinning is discussed.

5:05 PM
Effect of LiF on Liquidus Temperature, Density and Electrical Conductivity of Molten Cryolite: *Hongmin Kan*[1]; Zhaowen Wang[2]; Zhongning Shi[2]; Yungang Ban[2]; Bingliang Gao[2]; Shaohua Yang[2]; Xiaozhou Cao[2]; Xianwei Hu[2]; Shaoxian Ma[3]; Youwei Wu[3]; [1]Northeastern University/Shenyang University; [2]Northeastern University; [3]Northeastern University, Design and Research Institute

Effect of LiF on physicochemical properties of molten Cryolite-based aluminum electrolyte system was investigated. Liquidus temperature, density and electrical conductivity of melts in the Na3AlF6-AlF3-Al2O3-CaF2-LiF system were measured. The activation energy of conductance was obtained on the basis of the electrical conductivity data. The results showed that the liquidus temperature and the activation energy of conductance were reduced and the electrical conductivity was increased greatly by adding LiF. However, the density was increased with increasing LiF content in the acidic electrolytes. It is disadvantageous for the operation of industry cell. The effects of LiF content

on physicochemical properties of molten Cryolite-based aluminum electrolyte were analyzed in theory.

5:30 PM
The Development of 85kA Three-Layer Electrolysis Cell for Refining of Aluminum: Hengchao Zhao[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics and Astronautics

After studying the thermal-electric distribution, magnetic field of the 85kA three-layer electrolysis cell deeply and systematically, some influence regulations of the operating parameters to the cells have been disclosed. Based on these works, a series of 85kA three-layer electrolysis cells with the Gadeau process electrolyte system for capacity of 20,000t/y refined aluminum have been designed optimally; the operating parameters of the cells have been optimized; some improvements have been applied on the 85kA three-layer electrolysis cells in order to operate the aluminum refining course automatically. The aim of high current efficiency 98.5% and low DC power consumption 14000kWh/t has come true.

Aluminum Reduction Technology: Reduction Cell Modelling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Aluminium Tech

Wednesday PM Room: 297
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Dagoberto Severo, PCE Ltd.

2:00 PM
Optimization in Magnetic Modeling: *Stéphane Wan Tang Kuan*[1]; Denis Jacquet[2]; Thierry Tomasino[1]; Carlos Canutas De Wit[2]; [1]Alcan; [2]GIPSA-Laboratory, Centre National de la Recherche Scientifique

The stability of the electrolysis cells represents a major challenge in terms of safety and current efficiency. In order to control stability, models are used to determine the magnetic field in the cells but also in the potrooms with final outcome being an optimal cell busbar circuit. They also can be used to determine the current intensity and the position of a compensation loop if required. The method we used allows a rapid optimization of the induced magnetic field distribution at the various parts of a production line. It is based on a static and nonlinear optimization technique. The paper presents a practical case of magnetic optimization on a plant of AP30 technology integrating cost aspects. The constraints and limits of such an approach are also mentioned.

2:25 PM
Shallow Water Model for Aluminium Electrolysis Cells with Variable Top and Bottom: *Valdis Bojarevics*[1]; Koulis Pericleous[1]; [1]University of Greenwich

The MHD wave instability in commercial cells for electrolytic aluminium production is often described using 'shallow water' models. The model is extended for a variable height cathode bottom and anode top to account for realistic cell features. The variable depth of the two fluid layers affects the horizontal current density, turbulent horizontal velocity circulation and the resulting wave development. Instructive examples for the 500 kA demonstration cell are presented.

2:50 PM
Impact of the Vertical Potshell Deformation on the MHD Cell Stability Behavior of a 500kA Aluminum Electrolysis Cell: *Marc Dupuis*[1]; Valdis Bojarevics[2]; Daniel Richard[3]; [1]GeniSim Inc; [2]University of Greenwich, School of Computing and Mathematics; [3]Hatch

In previous publications, it was rationalized that a large vertical potshell deformation may have a negative impact on the operations of very high amperage cells. The MHD-Valdis non-linear Magneto-Hydro-Dynamic model was therefore extended to take into account the displacement of the potshell. The MHD cell stability behavior of a 500 kA cell with a 17.3 meters long potshell was then studied.

3:15 PM
Comparison of Various Methods for Modeling the Metal-Bath Interface: *Dagoberto Severo*[1]; Vanderlei Gusberti[1]; Andre Schneider[1]; Elton Pinto[1]; Vinko Potocnik[2]; [1]PCE Ltd.; [2]Vinko Potocnik Consultant

Correct evaluation of the stationary metal-bath interface in aluminum reduction cells is still a source of discussion and controversy. The objective of this paper is to present some calculations of the interface performed by different methods, used in software packages and to compare them with measured metal-bath interface profile in a real cell. Our comparison includes shallow layer approach and 3D multiphase methods, such as homogeneous and inhomogeneous volume of fluid (VOF). Different boundary conditions on the top of bath channels and different turbulence models are tested. Finally, a simple generic magnetic field is proposed which could be used for future software benchmarking of this problem.

3:40 PM Break

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Modelling and Measurement of Metal Pad Velocity in a 208 Ka End to End Prebaked Cells: Mohamed Doheim[1]; Abdel Fatah El-Kersh[2]; Nabil Kotb[3]; Mohamed Ali[4]; *Mohamed Mahmoud*[4]; [1]Assiut University; [2]El Taif University; [3]El Minia University; [4]Egyptalum

Measurements and modeling of flow characteristics are presented for turbulent flow subjected to current-magnetic field interaction in 208 kA end to end prebaked cells. The numerical calculations of incompressible turbulent flow have been carried out by solving full time –averaged Navier-Stokes equation using finite difference method. The k-epsilon turbulence model is modified to account for near wall function. In this part, the electromagnetic force is introduced in the governing equations as source term. Velocity measurements in the metal pad were established using iron rod method. The calculated and measured velocities showed a good agreement and the model can be used to predict the velocity profiles inside the molten melt regions at different operating conditions.

4:15 PM
Dynamics of the Gas Emission from Aluminum Electrolysis Cells: Laszlo Kiss[1]; *Klára Vékony*[1]; [1]Universite du Quebec a Chicoutimi

The evolution of gas has a strong impact on the operation of the aluminum reduction cell. The dynamics of the bubble related phenomena involves the nucleation travel and coalescence of the bubbles under the anode, and finally their escape from the bath. Generally, it is assumed that the bubbles escape freely and easily as they arrive to the upper horizontal surface of the bath. In the present paper, we analyze the case when the escape of the gas is hindered above the bath, for example by the presence of a crust layer. If the gas accumulates and forms a cushion under certain zones of the crust, its escape can provoke oscillations. This frequency is independent of the nucleation and even of the formation of big coalesced bubbles under the anode. The dynamic character of this phenomenon, which depends strongly on the geometry, will be illustrated by a simple mathematical model.

4:40 PM
Freeze Thickness in the Aluminum Electrolysis Cells: *Laszlo Kiss*[1]; Véronique Dassylva-Raymond[1]; [1]Universite du Quebec a Chicoutimi

The presence of a frozen electrolyte layer (ledge) on the sidewall is vital for the aluminum reduction cell. The thickness of the ledge is the result of the complex interaction between heat balance, amperage, bath chemistry, cell resistance, bath movement etc. In the present paper, the general tendencies like the effects of increasing the amperage, the correlation between bath-ledge heat transfer and freeze thickness are analyzed. The analysis is based on the mathematical modeling of the heat balance of an aluminum reduction pot. The tendencies extracted from the results of the numerical simulations can be cast into simple functional or graphical forms that characterize the given cell design.

5:05 PM
Two-Dimensional Model of Melt Flows and Interface Instability in Aluminum Reduction Cells: *Mehdi Kadkhodabeigi*[1]; [1]Sharif University of Technology

We derive a new non-linear two dimensional model for melt flows and interface instability in aluminum reduction cells. This model is based on non-linear de St.Venant shallow water equations and contains the main features of

an aluminum reduction cell. In this model we consider linear friction terms but in a new way that has not been considered in previous works. Our results are in good agreement with the results of simulation of viscous flow. This model is applicable both in determination of melt flows in molten aluminum and cryolite layers and also in finding the extreme limit for stability of interfacial waves in an aluminum reduction cell.

Biological Materials Science: Mechanical Behavior of Biological Materials II

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

Program Organizers: Ryan Roeder, University of Notre Dame; Robert Ritchie, University of California; Mehmet Sarikaya, University of Washington; Lim Chwee Teck, National University of Singapore; Eduard Arzt, Max Planck Institute; Marc Meyers, University of California, San Diego

Wednesday PM — Room: 390
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: John Nychka, University of Alberta

2:00 PM Invited
Effects of Cytoskeletal Dynamics on Cell Deformation and Function: *Subra Suresh*[1]; Ju Li[2]; George Lykotrafitis[1]; [1]Massachusetts Institute of Technology; [2]Ohio State University

All living matter such as tissues and cells differ fundamentally from traditional engineering materials in that they require the input of energy (ATP) to function. Such metabolism introduces interesting non-equilibrium effects, which fundamentally alter their mechanical behavior. A general framework for simulating cellular cytoskeletal dynamics (CD) (PNAS 104 (2007) 4937; Biophys. J. 88 (2005) 3707) has been developed so as to model the self-assembly of cytoskeleton of human red blood cells (RBC). The questions addressed include: (a) how do applied mechanical forces and ATP input influence cytoskeletal reorganization? (b) how does such cytoskeletal reorganization facilitate cell shape changes and motility through small blood vessels? and (c) how does the cytoskeleton recover its original shape upon release of the mechanical or chemical energy? The CD simulation results resolve some long-standing questions regarding RBC equilibrium shape and deformation response.

2:30 PM
Surface Phase Separation and Flow in Vesicle Bio-Membranes: Shuwang Li[1]; *John Lowengrub*[1]; [1]University of California, Irvine

Recent experiments of giant unilamellar vesicles (GUV) reveal that lipid bi-layer membranes exhibit rich shape transition behaviors, such as bud formation. Physical processes like phase separation and bending are usually coupled with morphology changes in membranes. Because of the presence of high-order nonlinear terms in these physical processes, it is highly challenging to simulate phase decomposition and motion of phase boundaries on a moving surface. In this talk, we present a mathematical model describing the nonlinear coupling among the flow, vesicle morphology and the evolution of the surface phases. Our numerical method combines the immersed interface method to solve the flow equations and the Laplace-Young jump conditions, the level-set method to represent and evolve the surface, and a non-stiff Eulerian algorithm to update the mass concentration on the surface. Numerical results show detailed dynamics of phase separation and shape transformation (e.g. bud formation).

2:50 PM
Coupled Composition-Deformation Phase-Field Simulation of Multicomponent Lipid Membranes: *Chloe Funkhouser*[1]; William Jaquinde[1]; Francisco Solis[2]; Katsuyo Thornton[1]; [1]University of Michigan; [2]Arizona State University

We investigate the morphological evolution of binary lipid membranes via a phase-field simulation. We consider membranes in which the local composition and shape are coupled through composition-dependent spontaneous curvature. The evolution of the composition field is described by a Cahn-Hilliard-type equation, while shape changes are described by relaxation dynamics. Our method explicitly treats the nonlinear form of the geometrical scalars, tensors, and differential operators associated with the shape of the membrane. We study morphological evolution and stability of lipid membranes initialized in a variety of compositional and geometric configurations. Nearly planar membranes and spherical vesicles are considered. We discuss the effects of spontaneous curvature, bending rigidity, and line tension on the dynamics and final configurations. For the nearly planar case, microstructures are characterized quantitatively using average domain size, shape anisotropy, and other statistical measurements.

3:10 PM
On Affinity in the Deformation of Biopolymer Networks: *Erik Van der Giessen*[1]; Patrick Onck[1]; [1]University of Groningen

Networks of (usually cross-linked) biopolymers play a key role in various biological functions, in particular in mechanotransduction. Well-known examples are the cytoskeleton and collagen. The peculiar strain stiffening exhibited by many of these materials has been the topic of intense investigations. The current explanations can be classified in two groups. The first attributes strain stiffening to the stiffening of individual filaments caused by entropic effects. The second explanation charges the non-affine distortion of the network, allowing for a transition from a soft, bending-dominated response to one dominated by stretching of the filaments. By contrast, the entropic stiffening theory available to date presumes affinity of the network distortion. Affinity or not has thus become a critical issue. Theoretical and simulation considerations, first in two and more recently in three dimensions, are slowly getting supplemented with experimental observations. This paper aims at presenting a comprehensive, state-of-the-art view on affinity.

3:30 PM Break

3:40 PM
Evaluation of the Elastic Properties of Human Corneas via Nanoindentation: Rut Rivera[1]; *David Bahr*[2]; Julie Last[3]; Christopher Murphy[3]; [1]University of Puerto Rico at Humacao; [2]Washington State University; [3]University of Wisconsin

The assessment of the mechanical properties of the cornea requires the ability to probe spatially unique regions of the tissue, both laterally and through the thickness. Nanoindentation allows for these properties to be measured, but brings specific challenges due to the material's compliance and environmental requirements. Utilizing a nanoscale dynamic mechanical analysis method we have carried out indentations in human corneas and hydrogel based contact lenses in a phosphate buffer solution. For the cornea the target layers of interest were the basement membrane and stroma. The elastic response of these surfaces for depths between 50 and 5000 nm is on the order of 10-1 MPa as a function of depth, with a viscoelastic response that appears to be relatively constant with depth. Challenges related to quantification of properties are discussed, particularly issues of sensitivity to surface detection in solution with a material that exhibits significant adhesion to mechanical probes.

4:00 PM
Evidence of Van Der Waals Adhesion Found in Abalone Foot: *Albert Lin*[1]; Ralf Brunner[1]; Po-Yu Chen[1]; Frank Talke[1]; Marc Meyers[1]; [1]University of California, San Diego

The attachment of abalone to surfaces was investigated and the stress for separation was measured and determined to be 0.15 MPa. The surface of the abalone foot in contact with surfaces was characterized and is found to be composed of fibers (setae) terminated by spatulae with a diameter of 200 nm in a similar fashion to gekko feet. The areal density of the spatulae is 1.9 x 106/mm^2. Atomic force microscopy measurements show that the spatulae show bonding to surfaces and that the bonding force per spatula is 3 μN. This is characteristic of van der Waals forces in a similar way to gekko feet. This corresponds to a theoretical attachent stress of 0.18 MPa, in accordance with the experimental measurements. Other effects, such as capilarity and suction are also being investigated. Research funded by the National Science Foundation Grant DMR 0510138.

4:20 PM
Mechanics of Permanent Biological Attachment Systems: *Tina Steinbrecher*[1]; Deane Harder[2]; Thomas Speck[2]; Oliver Kraft[1]; Ruth Schwaiger[1]; [1]Forschungszentrum Karlsruhe; [2]University of Freiburg

Climbing plants have developed a variety of mechanical strategies to permanently attach to supporting structures thereby achieving vertical growth. Boston ivy (*Parthenocissus tricuspidata*) has specialized attachment pads that exhibit excellent mechanical performance. However, they have not yet been investigated systematically with respect to interface, microstructure or mechanical behavior. In this study, the mechanical properties of attachment pads on various substrates as well as the interface were investigated. Boston ivy was found to attach to many different substrates, e.g. to very smooth inorganic as well as to rough organic substrates. In contact, the attachment structures lignify and stay attached even under extreme environmental conditions. Our tensile tests show that a pad withstands stresses up to 4 MPa; failure never occurred at the interface. Based on microscopical investigations, which indicate an almost perfect plant/substrate form closure, the strategy of the plant to achieve this strong attachment is discussed.

4:40 PM
Growth and Mechanical Behavior of Hydroxyl-Carbonate Apatite Layer on 45S5 Bioactive Glass: *Ding Li*[1]; Fuqian Yang[1]; John Nychka[2]; [1]University of Kentucky; [2]University of Alberta

Bioactive glasses have been known for applications in bone replacement implants. The formation of hydroxyl-carbonate apatite (HCA) layer and the mechanical behavior of the HCA layer potentially determine the bioactive response of bioglasses in bone implants. In this work, the growth behavior of the HCA layer over 45S5 bioglass is evaluated in a simulated body fluid at a temperature of 37°C. After 24 hours immersion, the HCA layer fully covers the surface of the 45S5 bioglass. The thickness of the HCA layer increases with the immersion time, following a parabolic relation. The localized mechanical behavior of the HCA layers is studied by using the indentation technique. The effect of the HCA layer thickness on the mechanical response is probed.

5:00 PM
Microstructure and Mechanical Behavior of Biocompatible TiO_2/Ti: *Grant Crawford*[1]; Nikhilesh Chawla[1]; Jack Houston[2]; Jan Ringnalda[3]; Tony Carpenter[3]; [1]Arizona State University; [2]Sandia National Laboratory; [3]FEI Company

Titanium oxide coatings have been shown to exhibit desirable properties as biocompatible coatings. In this talk we report on mechanical properties and deformation behavior of TiO_2 nanotubes grown on pure titanium substrates through anodic oxidation. By adjusting the processing conditions, the coating thickness, tube diameter and tube wall thickness were varied. Characterization of the as-processed coatings was conducted using scanning electron microscopy, Focused Ion Beam milling, and transmission electron microscopy. Nanoindentation, using an Interfacial Force Microscope, was employed to accurately probe the Young's modulus of the nanotubes. This technique was also used to study the inelastic deformation behavior of the nanotubes. The relationship between the nanostructure of the tubes and the mechanical response will be discussed.

5:20 PM
The Design of Material and Mechanical Properity of Stabilization Systems and Special Medical Tools for Alloplastic of Hip Joint: *Szota Michal*[1]; Malgorzata Lubas[1]; Jozef Jasinski[1]; Bogdan Stodolnik[2]; [1]Czestochowa Technical University; [2]ZWM of Bialysto University of Technology in Suwalki

There are different reasons for which the results of hip alloplasty become worse with time. Aseptic slackening of endoprosthesis is the main reason affecting a growth in dysfunction of the limb. In such cases the most effective method of repair the bone defects occurring in the vicinity of endoprosthesis is an application both tightly stuffed allogenic bone grafts and metallic stabilizers. Secondary hip arthroplasty needs the massive and frozen bone grafts and metal stabilizers in the form of nets and baskets, which strengthen and fasten the bone defects. Stabilizers protect the graft against excessive overload occurring in its surrounding, especially during heal process. In order to complete the bone defects a prototype batch of: -the bridging baskets in 12 type dimensions, -strengthening baskets in 2 diameters, -strengthening meshes shaping by bending and stamping.

5:40 PM
Microstructure and Property of Titanium Using for Implants: *Szota Michal*[1]; Jozef Jasinski[1]; Bogdan Stodolnik[2]; [1]Czestochowa Technical University; [2]ZWM Bialystok University of Technology in Suwalki

Oxidation is one of the most employed methods to improve titanium and its alloys properties especially due to medical application. This process like most of the thermo-chemical treatment processes substantially influences on the characteristic of surface layers and the same on its mechanical and useful properties. Oxide coatings produced during titanium oxidation were examined due to their composition identification. Titanium was oxidized in fluidized bed in temperature range between 500-700°C. Microstructures of titanium with a visible oxide coating on its surface after thermo-chemical treatment and changes of grain size in core of titanium samples are described. Moreover X-ray phase analysis of obtained oxides coatings was made as well as microhardness measurements of titanium surface layers after oxidation process. Finally, the surfaces of titanium after oxidation in fluidized bed were measured by Auger electron spectroscopy.

Bulk Metallic Glasses V: Structures and Modeling II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter K. Liaw, University of Tennessee; Wenhui Jiang, University of Tennessee; Guojiang Fan, University of Tennessee; Hahn Choo, University of Tennessee; Yanfei Gao, University of Tennessee

Wednesday PM — Room: 393
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Katharine Flores, Ohio State University; Takeshi Egami, University of Tennessee

2:00 PM Invited
Atomistic Mechanism of Relaxation and Deformation in Metallic Glasses: *Takeshi Egami*[1]; [1]University of Tennessee

The mechanism of mechanical deformation and relaxation will be discussed from the atomistic point of view. The majority of the experimentalists working on metallic glasses use the free-volume theory. In the field of liquids and glasses, on the other hand, the main-stream theory today is the energy landscape theory. Both of them are essentially phenomenological theories, in the sense that the parameters in the theory cannot be directly computed from the first-principles. Thus the connection of these theories to the microscopic reality is not totally clear. What we need is a theory for the intermediate steps that link these theories to atomistic processes. While this is a challenging problem since metallic glasses are atomic glasses, they may be simple enough to allow some progress. We discuss the theory of topological fluctuations in the atomic connectivity network that may shed some light into this difficult problem.

2:20 PM Invited
New Generation of Structural Materials: Solid Solution Alloys with High Entropy of Mixing and Bulk Metallic Glasses: *Zhang Yong*[1]; [1]University of Science and Technology Beijing

According to Gibbs phase rule, a great number of intermediate phases will form for the multi-principal components materials (also high-entropy alloys, HEAs), which are located at the center of the phase diagram, and solid solution phase generally forms at the terminal side of the phase diagram. However, it is recently reported that some HEAs form simple solid solution rather than many intermediate phases. In this paper, phase-formation rule of HEAs was obtained in terms of the atomic-size factor (δ) and enthalpy of mixing (ΔH_{mix}) by summarizing the literature data reported, and its validity was also verified by our experimental results. The mechanical properties of the HEAs were compared with those of the bulk metallic glasses.

2:40 PM Invited

The Influence of Shear on the Viscosity and Crystallization of Bulk Metallic Glass Forming Liquids: *Ralf Busch*[1]; [1]Saarland University

The shear rate and temperature dependence of the viscosity and the onset of crystallization in several bulk metallic glass (BMG) forming liquid has been measured in the liquid and undercooled liquid state. The alloys show pronounced shear thinning especially close to the liquidus temperature. With increasing temperature this behavior gets less pronounced and in certain alloys a transition to a Newtonian liquid is observed, which is associated with the change from strong to fragile liquid behavior. In general, we find that the shear rate effects are more pronounced in strong liquids than in fragile liquids. Shearing the liquid accelerates the crystallization process and shifts the time-temperature transformation diagrams to shorter times. We discuss the influence of shearing not only on the kinetics but also on the thermodynamic driving force that leads to faster crystallization.

3:00 PM Invited

Novel Applications of Metallic Glass Composites Using Their Functional Properties: *Nobuyuki Nishiyama*[1]; Nozomu Togashi[1]; Yasunori Saotome[2]; Akihisa Inoue[3]; [1]Research and Development Institute of Metals and Composites for Future Industries; [2]Tohoku University, Institute for Materials Research; [3]Tohoku University

Metallic glasses (MGs) generally exhibit superior mechanical properties such as high-strength, low Young's modulus and high elastic elongation limit. These excellent mechanical properties are used for production of prototype industrial products. For instance, precision MG gear parts for micro geared motor and MG diaphragms for pressure sensor are successfully developed and these products exhibit an outstanding performance due to unique mechanical properties. In a few years, some applications will be on the market. This fact encourages further progress of fundamental researches and industrial developments. Unfortunately, there are several issues that have to solve among industries. That is, lack of plastic elongation under tensile load, high electrical resistivity or only exhibiting soft magnetic properties. If the issues could be solved, applications of MG will be drastically broadened. In this paper, we propose the way to solve the issues by applying MG composites and novel applications of MG composites will be discussed.

3:20 PM

Characterization of Glass and Nanocrystalline Composite Structure in Highly Driven Marginal Glass Forming Alloys: *Eren Kalay*[1]; Scott Chumbley[1]; Iver Anderson[1]; [1]Ames Laboratory/Iowa State University

Al-Rare Earth (RE) based alloys are important marginal glass forming alloys, where glass formation is often accompanied by the presence of nanocrystals during solidification. Among these alloys, the Al-Sm system has attracted interest due to its wide glass formation range. Thus, Al(100-x)Smx (x=8,10,12, and 14) binary alloys were produced using Cu-block single-roller melt spinning. The as-quenched phase was studied by high resolution electron microscopy (HRTEM) in combination with energy loss (EELS) and energy dispersive spectroscopy (EDS)and by x–ray diffractometry (XRD). The structure of the as-quenched nanocrystalline phase is sensitive to initial composition of liquid and has a typical length scale of 5 nm. The appearance of pre-peaks on XRD diffraction patterns and low calculated activation energies for the decomposition of amorphous product phases at different compositions indicate some local ordering on quenching.

3:35 PM Invited

Amorphous Structure with Inhomogeneity for Enhancement of Plasticity: *Do Hyang Kim*[1]; Hye Jung Chang[1]; Eun Soo Park[2]; Won Tae Kim[3]; [1]Yonsei University, Department of Metallurgical Engineering; [2]Harvard University, School of Engineering and Applied Sciences; [3]Chongju University, Division of Applied Science

Design of amorphous structure with inhomogeneity, which includes inhomogeneous distribution of chemical bonding, clustering and free volume is suggested as one of the possible ways for enhancement of plasticity in monolithic metallic glasses. In the present study, the possible routes and mechanisms for the enhancement of plasticity in monolithic glass alloys will be presented. Ti-Zr-Ci-Ni-Be and Zr-Ti-Cu-Ni-Nb-Al metallic glass alloys exhibit enhanced compressive plasticity due to the presence of quenched-in icosahedral nuclei in as-cast state. While Ni-Nb and Cu-Zr based alloys exhibit enhanced plasticity with the addition of elements having positive heat of mixing with constituent elements. The detailed structural analysis implies that the degree of preferential bonding increases with increasing the difference in the heat of mixing between the elements and with increasing the amount of additional element. The inhomogeneous amorphous structure might induce more sites for initiation of shear bands and finally can distribute the localized stress.

3:55 PM Break

4:00 PM Invited

Characterizations of Mechanically Alloyed Ti-Based Bulk Metallic Glass Composites Containing Carbon Nanotube: *Chih-Feng Hsu*[1]; Pee-Yew Lee[1]; [1]National Taiwan Ocean University

This study explored the feasibility of preparing Ti50Cu28Ni15Sn7 bulk metallic glass composite by powder metallurgy route. The CNT / Ti50Cu28Ni15Sn7 metallic glass composite powders can be formed by mechanical alloying process. The DSC result shows that the thermal stability of the Ti50Cu28Ni15Sn7 amorphous matrix is affected by the presence of the CNT particles. The bulk metallic glass composite was successfully prepared by vacuum hot pressing the as-milled CNT / Ti50Cu28Ni15Sn7 metallic glass composite powders. A significant hardness and fracture strength increase with the CNT additions was observed for the consolidated composite compacts. The compressive fracture strength and elastic strain of the composites reaches 1937 MPa and 13 %, respectively, for the 12 vol. % CNT specimen. The effects of the CNT addition on the thermal stability and mechanical property of CNT/Ti50Cu28Ni15Sn7 bulk metallic glass composites were discussed.

4:20 PM

A Simple Model to Predict the Temperature Dependence of Elastic Moduli of Bulk Metallic Glasses: *Zhiying Zhang*[1]; Veerle Keppens[1]; Takeshi Egami[1]; [1]University of Tennessee

In the past few years, significant research efforts have been devoted to the study of the elastic moduli of bulk metallic glasses. These studies are partially triggered by the reported correlation between the energy of fracture and the Poisson ratio for bulk metallic glasses, with brittle glasses displaying a low Poisson ratio. However, reliable measurements as a function of temperature on high-quality materials are often time-consuming. We report a simple model to predict the temperature dependence of elastic moduli of bulk metallic glasses from room temperature measurements, using the Varshni Equation and the basic assumption that between absolute zero and the melting point, the shear and bulk moduli change resp. by 45% and 22%. This model has been tested using experimental data obtained on a large variety of bulk metallic glasses, and the predicted values are found to be in very good agreement with the experimental results.

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Ni and Cu Free Vitreloy: *Aaron Wiest*[1]; Gang Duan[1]; Annelen Kahl[1]; Joseph Schramm[1]; Landon Wiest[2]; Andrew Peck[3]; Georg Kaltenboeck[1]; Marios Demetriou[1]; William Johnson[1]; [1]Caltech; [2]Brigham Young University; [3]Pasadena City College

Investigation of the ZrTiBe system revealed three regions of interest. One region contains bulk glass forming alloys having critical casting thicknesses as high as 6mm and densities as low as titanium. Another region forms in-situ beta phase composites that exhibit moderate ductility. The third region contains alloys exhibiting large supercooled liquid regions and eutectic crystallization. We will show that partial substitution of Be in a base ternary glass with various late transition metals (excluding Ni and Cu) yields a glass with properties drastically improved over the ternary base alloy. This suggests that Vitreloy alloys derive their properties from their ternary counterparts. Using our ternary map as an alloy development tool, Ni and Cu free compositions with casting thicknesses in excess of 1cm, yield strengths > 1.7GPa, ΔT=150C, and compressive ductility in excess of 2% were found.

4:50 PM

Structural Evolution and Phase Analysis of Bulk Amorphous $Zr_{56.6}Cu_{17.3}Ni_{12.5}A_{l9.6}Ti_4$ Alloy: *Dawei Xing*[1]; Jun Shen[1]; Jianfei Sun[1]; Gang Wang[1]; Yulai Gao[1]; Demin Chen[1]; Peter K. Liaw[2]; [1]Harbin Institute of Technology, School of Materials Science and Engineering; [2]University of Tennessee

The structural evolution of bulk amorphous $Zr_{56.6}Cu_{17.3}Ni_{12.5}Al_{9.6}Ti_4$ alloy in a as-cast wedge ingot was studied by OM, SEM, and TEM to clarify the competing relationship between the crystalline phases and amorphous phase during cooling. It was found that there were three clear zones; amorphous, partially-crystallized, and crystallized areas, from the thin to thick ends of the wedge ingot. These three zones were dominated by the amorphous, the intermetallic phase $CuZr_2$+$NiZr_2$, $Cu_{10}Zr_7$+Zr_3Al, and Zr_2Ni, respectively. The precipitation of these phases was dependent on the cooling rate. The observations were discussed in terms of a schematic Time Temperature Transition(TTT) curve of $Zr_{56.6}Cu_{17.3}Ni_{12.5}Al_{9.6}Ti_4$. This finding may gives a direct evidence to that Zr-Cu has a larger glass-forming tendency than that of Zr-Ni, and GFA may be improved by restraining Zr_2Ni phases through removing all the Ni elements in a Zr-based multi-component alloy.

5:05 PM

A New Criterion for Predicting the Onset of Glass-Like Temperature Dependent Behavior in Simple Liquids: *Rachel Aga*[1]; James Morris[1]; Valentin Levashov[2]; Takeshi Egami[2]; [1]Oak Ridge National Laboratory; [2]University of Tennessee

We present simulations on the temperature-dependence of the shear viscosity and diffusion coefficient of a monatomic glass-forming liquid. Changes in temperature-dependent properties include deviation from Arrhenius behavior, and temperature-dependence of the effective diameter from the Stokes-Einstein relation. We introduce a definition for the term "liquid-like" based on experimental viscosities from various atomic liquids at their melting temperatures. The above changes in dynamical behavior occur when the system has departed the liquid-like dynamical regime, and is now exhibiting glass-like temperature-dependent behavior. In the glass-like regime, there is an increasing spread in the values of time correlations for shear stress components. In contrast, the spread is constant when the system is liquid-like. The development of a double-peaked g(r) is strongly correlated with the transition from liquid-like to glass-like temperature-dependent dynamics. This research has been sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy under Contract DE-AC05-00OR-22725 with UT-Battelle.

5:20 PM

A Thermodynamic Description of the Metallic Glass State Using the Central Atoms Model: *Eric Lass*[1]; Gary Shiflet[1]; Aiwu Zhu[1]; Joseph Poon[1]; [1]University of Virginia

The thermodynamic properties of the metastable amorphous phase in metallic glass systems have been studied using the central atoms model. By consideration of the shell or "cage" of nearest neighbors (NN) surrounding a given atom, an understanding of short range order, its temperature dependence, and the effects on the thermodynamics of the system are obtained. Certain NN combinations are more stable relative to others, depending on bulk composition, atomic size, chemical heats of mixing between elements, etc. The resulting expressions for the thermodynamic functions are dependent on the probability distribution of the possible NN cages. This model allows for a more accurate description of the supercooled liquid state by the introduction of such irregularities as a variable number of NN between atoms of like and unlike species. It is shown the even in a simple binary system the local structure around the different species varies dramatically.

5:35 PM

Diffusion Mechanism in Metallic Glasses from Bond Defects Perspective: *Aiwu Zhu*[1]; Gary Shiflet[1]; S. Poon[1]; [1]University of Virginia

Diffusion in metallic glasses and deeply undercooled liquids is analyzed from the perspective of structural defects induced by the bond deficiencies (BD). The elementary process is considered to be thermally activated hopping of diffusing atoms between the first-neighbor and the second neighbor positions at the BD defects that requirescooperative movement of multiple adjacent atoms. The activation energy Q will then depend on the bond strengths, size of diffusing atoms, the elastic constant of the matrix and the effective number of matrix atoms that are involved. Application to the Zr-Ni and Ti-Ni systems shows the atomic size effect of diffusing atoms agrees with experimental measurements. Most of the other observations including the effects of isotopes, of pressure and the shape slope of the Q versus pre-exponential factor can also be explained accordingly.

Cast Shop Technology: Casting Processes and Quality Analysis

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Hussain AlAli, GM Casthouse and Engineering Services, Aluminium Bahrain Company (ALBA); David DeYoung, Alcoa Inc

Wednesday PM
March 12, 2008
Room: 295
Location: Ernest Morial Convention Center

Session Chair: Neil Hall, BHPBilliton - Bayside Aluminium

2:00 PM

Characterization of the Microstructure of the AA8006 Alloy Sheets and Foils: *Carmen Stanica*[1]; Petru Moldovan[2]; Gheorghe Dobra[1]; Cristian Stanescu[1]; [1]ALRO S A; [2]Polytechnic University of Bucharest

The paper presents the evolution of the 8006 alloy microstructure in the plastic deformation process; the paper's purpose is to set the causes for the defective conditions of the different size sheets and foils. The experiments put in evidence the defective conditions caused by the presence of iron, manganese and silicon compounds in the 8006 alloy structure, as well as the conditions occurred in the rolling process. The slab microstructures before and after homogenization were compared to the microstructures of foils and sheets obtained by plastic deformation. The techniques of optical quantitative microscopy, electronic microscopy SEM/EDS and electronic microscopy at the JXA micro analyzer to highlighting the process and material factors responsible for the presented defective conditions.

2:20 PM

Determination of TiB_2 Inclusions in Al-Billets by Single Spark Emission Spectrometry: *Marcel Rosefort*[1]; Frank Urbanek[1]; Joerg Niederstrasser[2]; Hubert Koch[1]; [1]Trimet Aluminum AG; [2]OBLF Gesellschaft fuer Elektronik- und Feinwerktechnik mbH

Aluminum safety parts and aluminum bright shining components, especially for automotive applications, have to be nearly free from inclusions due to their influences on mechanical properties and surface quality, respectively. On the other hand grain refinement is essential. For this purpose Titanium/Boron based master alloys are used, which can lead to TiB_2 agglomerations in the billet. The usual method for quality control is microscopic examination of the specimen. The growing demand for high quality billets has created a need for faster and cost-efficient methods of quality control. Single spark spectrometry promises fast determination of inclusions, especially of TiB_2-particals or agglomerations. Statistical evaluation of single spark spectrometry data allows the determination of distribution and size of TiB_2-agglomerations. This paper covers the application of single spark emission spectrometry to Al-billets, the graphical and statistical analysis and an outlook on the quality control using this new technique.

2:40 PM

Mastering the Strip Geometrical Tolerances with the Twin-Roll Continuous Strip-Casting Technology: *Frederic Basson*[1]; Marc Bosch[1]; Philippe Charlier[1]; [1]Novelis PAE

From Pechiney to Novelis, and today with Hindalco in the Aditya Birla Group, Novelis PAE has been a major actor in developing twin-roll continuous strip-casting technology for more than five decades. In the recent years, driven by the higher speed capabilities of the latest generation rolling mills, customer requirements and technological developments have both been focused on tightening the strip geometrical tolerances, in order to improve the consistency of the strip cross profile and longitudinal gauge variations. To meet these demanding requirements, the highly proven Jumbo 3CM® continuous casting

technology includes advanced technological features addressing each casting process parameter impacting the solidification and thus the strip geometry : metal feeding system with accurate metal level regulation and temperature control, homogeneous roll water cooling system, highly reactive hydraulic gap control, improved shell coating control, together with an in-line cross and longitudinal strip profile monitoring.

3:00 PM
On the Formation of Interdendritic Cracking Phenomena in Direct Chill Casting of Aluminum Alloy Slabs: *Mostafa El-Bealy*[1]; [1]Ain Shams University

The formation mechanism of interdendritic cracking phenomena of continuously cast aluminum alloy slabs has been studied by metallographically examining slab sample and by performing a set of mathematical analyses of heat flow, solidification and interdendritic strain phenomena. The model also combines an interdendritic cracking model with concept of the effect of different dendritic solidification behaviors on Bealy's interdendritic cracking susceptibility function "BICSF" and elementary interdendritic area "EIA". The study has revealed that the interdendritic cracking phenomena can be classified principally into macro and micro cracks. The type of interdendritic crack depends mainly on the degree and pattern of macrosegregation. The analysis of interdendritic cracking by mathematical modeling indicates that Bealy's interdendritic cracking susceptibility function (BICSF) measures qualitatively the susceptibility of interdendritic micro or macrocracking during the interdendritic coherent region in the mushy zone. However, the elementary interdendritic area EIA is to simulate qualitatively crack initiation and propagation.

3:20 PM
Novelis Fusion™: A Novel Process for the Future: Todd Bischoff[1]; Lawrence Hudson[2]; *Robert Wagstaff*[1]; [1]Novelis; [2]Novelis, Specialty Products Business Unit

Production of multilayered products in the aluminum industry has entered a "new birth" period in the flat rolled aluminum sheet industry as a result of the recent introduction of the Novelis Fusion™ process. This paper analyzes the interplay in the liquidus temperature and the freezing ranges of each alloy as well as potential problems and possible solutions encountered when producing some clad products.

3:40 PM Break

3:50 PM
Wrinkling Phenomena to Explain Vertical Fold Defects in DC-Cast Al-Mg4.5: *J. Lee Davis*[1]; Patricio Mendez[2]; [1]Novelis; [2]Colorado School of Mines

Some Al-Mg4.5 aluminum ingots cast by the direct chill method are subject to surface defects which appear to originate on the molten surface while others are not. Defects – commonly called "vertical folds" – are frozen into the solid ingot and must be removed prior to rolling. Vertical folds are found on top of the molten ingot surface where areas of thin oxide skin are (1) bounded by physical constraints and (2) stretched. The mechanism of wrinkling is suggested for the formation of vertical folds. Wrinkling behavior is described by physical and mathematical expressions for an elastic sheet in tension. The frequency and amplitude of vertical folds in DC-cast aluminum is found to obey wrinkling laws based on the thickness, length, Young's modulus, and Poisson's ratio of the thin, elastic oxide sheet.

4:10 PM
Investigation of Hydrogen Measurement Technique for Molten Aluminum: *Juan Moreno Exebio*[1]; Daniel Larouche[1]; Dany Paquin[2]; Jasmin Proulx[2]; Claude Dupuis[3]; [1]Laval University; [2]ABB; [3]Alcan International Limited

On-line measurement of hydrogen content in molten aluminum alloys based on the Sievert's law is the most widely used method in the aluminium industry. For more than 15 years, the AlSCAN analyzer has been recognized as the industry reference for hydrogen measurement because of its good reproducibility and its high robustness. It is well known that ambient conditions surrounding the molten aluminium are the main factors affecting the concentration of dissolved hydrogen. This paper presents the results of a detailed investigation of the key components and operating parameters of the AlSCAN technique on the measurement of dissolved hydrogen in molten aluminium alloys. Impact of ambient humidity in the vicinity of the measurement circuit is highlighted. Improvement to the current technology is proposed.

4:30 PM
Ab-Initio Predictions of Interfacial Heat Flows during the High Speed Thin Strip Casting of Metals and Alloys: Jinsoo Kim[1]; Donghui Li[1]; *Mihaiela Isac*[1]; Roderick Guthrie[1]; [1]McGill University, McGill Metals Processing Centre

The purpose of this study was to develop ab-initio mathematical and computational models aimed at predicting instantaneous heat fluxes when a liquid metal or alloy contacts a colder substrate. Semi-analytical and fully computational models have been developed to determine whether measured instantaneous heat fluxes associated with the strip casting of aluminum and magnesium alloys on copper, steel, and variously plasma-coated steel substrates, can be inferred from first principles. It is shown that the thermal properties of the melt and substrate are critical in determining the peak heat fluxes achieved during metal/mould contact, a cold copper substrate being more effective than steel. However, the net amount of heat extracted tends to favor a steel substrate. The surface texture of a belt and presence of gas, and or liquid/solid films among the "valleys" between the peaks where perfect thermal contact is assumed, is also shown to be of great significance. The way in which the liquid metal first contacts the substrate (i.e. the liquid metal delivery system), is similarly important in determining the shapes of the instantaneous heat flux curves, as are the rates at which the metal strip decouples from the substrate. The computational models developed, allows various scenarios to be effectively studied, and experimental curves matched with "predicted" curves based on reasonable assumptions. The use of such models can help guide operators of near-net shape casting machines in their quest to effectively control interfacial heat fluxes during the formation of sheet products.

4:50 PM Panel Discussion

Characterization of Minerals, Metals, and Materials: Characterization of Microstructure and Properties of Materials IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Materials Characterization Committee
Program Organizers: Jian Li, Natural Resources Canada; Toru Okabe, University of Tokyo; Ann Hagni, Intellection Corporation

Wednesday PM
March 12, 2008
Room: 284
Location: Ernest Morial Convention Center

Session Chairs: Toru Okabe, University of Tokyo; Yoshitaro Nose, Kyoto University

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2:00 PM
Formation of Silicon Carbide Crystals on Porous Silicon Substrate by Reaction Diffusion at Low Temperatures: *Yoshitaro Nose*[1]; Kohei Hosokawa[1]; Tetsuya Uda[1]; Yasuhiro Awakura[1]; [1]Kyoto University

We tried the formation of silicon carbide crystals by reaction diffusion of silicon and carbon at low temperatures. In general, silicon carbide crystals form on a silicon substrate by chemical vapor deposition at temperatures above 1200°C since atomic diffusion is very slow in this system; however, the formation at low temperatures is desirable. We thus considered utilizing porous silicon substrate, which has many pores of the nano-micron size on the surface, to promote carbon diffusion. Porous silicon substrates were fabricated by electrochemical method and its porosity and pore size were controlled by the kind of bath, current density and time. When the substrate was annealed at 1000°C for 10h after carbon deposited, the formation of silicon carbide crystals was observed on the porous silicon by Raman spectroscopy, while it was not observed on the single crystal substrate.

2:20 PM
Effects of Microalloying on an Innovative Quenched and Tempered Plastic Mold Steel: *Donato Firrao*[1]; Paolo Matteis[1]; Pasquale Russo Spena[1]; Giovanni Mortarino[1]; Maurizio Chiarbonello[1]; Maria Pinasco[2]; Maria Ienco[2]; Matteo Fabbreschi[2]; Giuseppe Silva[3]; Barbara Rivolta[3]; Riccardo Gerosa[3]; Maria Tata[4]; Severino Missori[4]; Roberto Montanari[4]; Andrea Ghidini[5]; [1]Politecnico di Torino; [2]Università di Genova; [3]Politecnico di Milano; [4]Università di Roma Tor Vergata; [5]Lucchini Sidermeccanica

Molds for large plastic automotive components are machined from pre-hardened steel blooms. Due to the size, the heat treatment of the commonly employed ISO 1.2738 steel yields mixed microstructures (varying from surface to core) and low toughness. An alternative steel is examined, which is microalloyed (noticeably with Nb, Zr and B) with the aim of limiting the austenitic grain growth, increasing the hardenability, and ultimately improving the strength and toughness combination. Although the hardenability calculated without the microalloying elements would be similar to the 1.2738 steel, perlite was avoided at core of a large quenched and tempered bloom (1x1.2 m section), whereas it occurred at core of similar 1.2738 blooms. Previous investigations of the microalloyed bloom pointwise microstructural and mechanical properties are extended.Moreover, microalloying effects are examined by comparing the properties of samples with and without the microalloying elements, that were laboratory heat-treated simulating a bloom core thermal history.

2:40 PM
Titanium Smelting Process Using Selective Chlorination and Preform Reduction Process: *Haiyan Zheng*[1]; Toru Okabe[1]; [1]University of Tokyo

For efficiently producing metallic titanium with high productivity and low cost, a new high-speed, semi-continuous, and environmentally sound titanium production process is investigated. In practice, iron (Fe) removal directly from titanium ore (ilmenite) by selective chlorination using calcium chloride (CaCl2) and water vapor at 1023~1293 K was carried out. The obtained deironized titanium ore was directly reduced to metal by calcium (Ca) vapor at 1273 K using the preform reduction process (PRP), which is based on the metallothermic reduction of titanium oxide in the preform. After the PRP, titanium powder with 98% purity was obtained from the upgraded ilmenite ore. It is demonstrated that Fe in the ilmenite was successfully removed and PRP is a feasible process for producing metallic titanium powder with certain purity (>98%). Recovery of chlorine from iron chloride waste—generated by the chlorination process—is also investigated in order to develop an environmentally sound process.

3:00 PM
Thermal Resistant Polymers Modified with Ionic Groups for Ion Selective Membranes: Ana Lucia Skury[1]; *Márcia Azevedo*[1]; [1]Universidade Estadual do Norte Fluminense

The development of polymer materials capable of standing elevated temperatures and still exhibiting high permeability and selectivity for protons, is of interest in fuel cell technology. Many engineering thermoplastic polymers have been considered as possible substitutes for perfluorinated ionomers, provide that a charge group (sulfonic) is introduced into the structural unit. However, the final properties of such polymers do not allow their application in an industrial scale. In this work, polyetherimide (PEI) and poly(4,4'-diphenylether-1,3,4-oxadiazole) (POD-DPE) were investigated. The polymer characterization was accomplished by TGA, intrinsic viscosity, IEC and ion permeability. The IEC and an increase in the water sorption showed that both polymers were effectively sulfonated. The results indicated that the sulfonation lead to a significant reduction in the degradation temperature of the polymer. In comparison with commercial polymers, the degradation temperature of the PEI(S) is higher, which makes this polymer useful for applications involving ion transport.

3:20 PM Break

3:40 PM
Influence of Centrifugal Casting Processing Parameters on the Distribution of Reinforcing Particles in Aluminum Matrix Composites: *Tunde Adelakin*[1]; Gustavo Gutierrez[1]; Oscar Suarez[1]; [1]University of Puerto Rico

In the whole range of functionally-graded materials (FGM), aluminum metal matrix composites (AMC) have attracted much interest partly due to their high wear resistance and high mechanical strength and toughness. Additional features, such as low density and low production costs, can be obtained when the aforementioned FGM AMCs are reinforced with AlB_2 and AlB_{12} particles. Centrifugal casting process then allows gradually redistributing those denser reinforcements. With increasing rotational speed the reinforcing particle distribution changes along the centrifugal force (longitudinal) direction of the cast, the highest being at the periphery decreasing towards the inner zone close to the pouring end. The effect of the functional compositional gradient on superficial hardness and micro hardness measured along the casting longitudinal direction is discussed. The relationship between casting time and pouring temperature with the attained particle distribution is also investigated.

4:00 PM
High Pressure Assisted Sintering of Nanostructured B-Si-Cu-Diamond Composites: Ana Lucia Skury[1]; *Sergio Monteiro*[1]; Guerold Bobrovnitchii[1]; Márcia Azevedo[1]; [1]Universidade Estadual do Norte Fluminense

The synthesis of advanced materials with superior performance and properties is of growing scientific and technological interest. In particular, significant achievements have been attained in the synthesis of nanocomposites associated with superhard materials. This work investigates nanostructured composites obtained by high pressure and high temperature sintering of synthetic diamonds combined with boron, silicon and copper. Diamond powder was mixed with B, Si and Cu, also in the form of powder. The mixture was then submitted to high energy wet milling until a nanopowder was formed. Sintering of this resulting nanopowder was carried out at 5.6GPa of pressure and 1300°C. X-ray diffraction and scanning electron microscopy analysis revealed the formation of new phases in a welll consolidated nanostructure with relatively high density.

4:20 PM
The Interface Behavior of Pyrite Power in Non-Aqueous Environment: Dan Li[1]; *Qiyuan Chen*[1]; Zhoulan Yin[1]; [1]Central South University

Ultrafine grinding pyrite was used ball grinder with particle size analysis. Results show that grinding pyrite powder in anhydrous ethanol is better than grinding in dry-mill powder particle size changes, anhydrous ethanol grinding was more efficient; Add several common aids, Sodium hexameta phosphate was found in pyrite ultrafine grinding process played better grinding effects; Powder saturated with vertical velocity measurement pyrite powder at different liquid medium of wettability, It was found pyrite powder in the body of anhydrous ethanol wettability was the best, followed by anhydrous acid, other organic solvents pyrite wettability was bad; turbidimetric method using the principle, spectrophotometer using different solvents pyrite dispersion effect, drawn in the aids solution, Pyrite Powder in the sodium hexameta phosphate solution to the dispersion stability was the best, but in non-aqueous environment, pyrite powder in anhydrous ethanol in the dispersion stability was the best, pyrite powder in water dispersion stability was bad.

Complex Oxide Materials - Synthesis, Properties and Applications: Ferroelectric/Dielectric Oxides

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division
Program Organizers: Ho Nyung Lee, Oak Ridge National Laboratory; Zhiming Wang, University of Arkansas

Wednesday PM Room: 277
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Gervasi Herranz, Unité Mixte de Physique CNRS/Thales; Kathrin Dörr, IFW Dresden

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2:00 PM
Effect of Delithiation Rate on the Microstructure in Li_xCoO_2 - A Transmission Electron Microscopy Study: *Heike Gabrisch*[1]; Tanghong Yi[1]; Qingfeng Xing[1]; [1]University of New Orleans

$LiCoO_2$ in the rhombohedral R-3m form is used as intercalation compound in rechargeable Li-ion batteries. During electrochemical charge discharge cycling lithium is extracted and re-inserted into the crystal lattice accompanied by reversible changes in the stacking order of the lattice. With lithium extraction the electrostatic repulsion between neighboring CoO_2 slabs increases, leading

to an expansion of the crystal lattice along the c-axis accompanied by a smaller contraction of the a-lattice parameter. Only little is known on the homogeneity of the lithium distribution within a population of powder particles of average composition Li_xCoO_2 or on concentration gradients within one particle, ($0 \le x \le 1$). We compare the microstructure of Li_xCoO_2 using transmission electron microscopy. The powders are produced by chemical delithiation using a solution of NO_2BF_4 in acetonitrile (CH_3CN) at different ratios of $LiCoO_2$: NO_2BF_4. Our results show at high removal rates an inhomogeneous and highly strained microstructure is formed.

2:20 PM

Application of High-ε Ceramics for Enhanced Sensitivity of Electron Paramagnetic Resonance Spectrometer: *Ilia Geifman*[1]; Iryna Golovina[1]; Anatoliy Belous[2]; [1]Institute of Semiconductor Physics of NASU; [2]Institute of General and Inorganic Chemistry of NASU

Earlier the application of single-crystal ferroelectric resonators improved the sensitivity of electron paramagnetic resonance (EPR) method up to 44 times.[1] In the present work new EPR resonators were developed and tested by using ceramic materials having high dielectric constant, ε=80-160. Designed resonators have high quality, $Q=10^3$-10^4, and enhance the sensitivity of EPR spectrometer up to 60 times. The advantages of new ceramic resonators are: 1) cheaper synthesis and simplified fabricating technology; 2) wider temperature interval of usage; 3) higher precision in registering EPR spectra. Due to high ε and low tanδ of applied ceramic material, the resonator increases microwave magnetic field on a sample, thereby improving EPR signal strength. The ceramics is made on the basis of titanates of complex oxides of rare-earth and alkali metals, and have perovskite or potassium-tungsten bronze type structure.
[1]I.N.Geifman, I.S.Golovina, J. Magn. Reson. 174, no.2, 292-300 (2005).

2:40 PM

Characterization of Anatase Synthesized by Sonochemical Means and Effects of Heat Treatments: *Leonardo Gonzalez-Reyes*[1]; Isaias Hernández-Pérez[2]; Francisco C. Robles Hernández[3]; Jose de Jesus Cruz Rivera[4]; Elsa Miriam Arce-Estrada[1]; [1]Instituto Politecnico Nacional; [2]Universidad Autónoma Metropolitana-A, Departamento de Ciencias Básicas; [3]Transportation Technology Center Incorporated; [4]University of San Luis Potosí, Facultad de Ingenieria-Instituto de Metalurgia

In the present research were characterized anatase particles produced by sonochemical means. The TEM, XRD and SEM confirmed that sonochemical synthesis is an effective method to produced nanometric anatase. Relatively low temperature, 500°C, heat treatments show that anatase can be coarsened from 6.4 nm to 28.3 nm after 72 hours of heat treatment and in the absence of phase transformations. Anatase coarseness following a behavior similar to the one described by the LSW theory. The heat treated anatase particles show significant morphological changes as a function of heat treatment time and this directly affects the surface area of the anatase particles. In addition, the results of the effects of heat treatment on Photocatalytic activity are presented. The characterization of the anatase particles were carried out by means of BET, TGA, XRD, Raman, FTIR, UV-Vis and TEM.

3:00 PM

Current Density Influences on Morphology and Adhesion of $RuO_2{\cdot}nH_2O$ Films Electrodeposited on Tantalum Substrate: *Hui Qin*[1]; [1]Central South University

Effects of the different current density on morphology and adhesion of $RuO_2{\cdot}nH_2O$ films were investigated by galvanostatic deposition. The mechanism of electrodeposition of hydrous ruthenium oxide was discussed. We can observe some morphological changes as the samples under SEM(Scanning Electron Microscope). When different current density are applied but the capacitance appears to be sensitive to this variable. Elements of films were analyzed by Energy Dispersive Spectroscope (EDS); Phases of films were analyzed by X-ray Diffraction meter(XRD);Zeta potential of colloid solution was analyzed by DELSA 440SX Analyzer. It can be concluded that the thickness of $RuO_2{\cdot}nH_2O$ films on tantalum substrate would increased with the cathodic current density, but the trend of the crack of $RuO_2{\cdot}nH_2O$ films proportional to the cathodic current density after they were dried in atmosphere. when cathodic current density up to 10mA/cm^2, $RuO_2{\cdot}nH_2O$ film flaked away from the tantalum substrate, adhesion of $RuO_2{\cdot}nH_2O$ film was very poor and hydrous ruthenium oxide easily drop from tantalum substrate.

3:20 PM

The Rule of NanoBoehmite on Spinel Formation in Magnesia Spinel Micro Composites: *Hamid Zargar*[1]; Farhad Golestanifard[1]; Hamidrea Rezaie[1]; [1]Iran University of Science and Technology

The amounts of formed spinel were investigated systematically at temperatures of 700-1500°C for four different groups of micro composites starting material containing 0,3,5 and 7 percents nano boehmite as an additive. In this regard, nanoboehmite added dead burnt magnesia and reactive alumina were mixed, pressed and fired orderly. FTIR was utilized in order to prove spinel formation as well as XRD patterns for both low and elevated temperatures. Results are shown that nano spinel was homogeneously formed on MgO surface at temperature as low as 700°C which these may act as seeds for spinel formation. It was cleared that the more nano boehmite results in lower permanent linear change (PLC). It was also found that spinel formation prohibited at temperatures above 1100°C with nanoboehmite addition. This project is aimed to applying nanotechnology in Iranian refractory industry where is the largest producer in the Middle East.

3:40 PM Break

3:55 PM

Study of the Superficial Behavior of Inorganic Pigments (Iron Oxides): *Oscar Restrepo*[1]; Federico Vásquez[1]; Oswaldo Bustamante[1]; [1]Universite Nacional De Colombia

Present work present the variation of the surface conditions of a pigment (iron oxide Fe_2O_3). We determine how the surface effects of particles, governed to a great extent by pH of the suspension, affect the behaviour of the fluid like colloid as far as viscosity and fluidity, and as well, those changes generated in the surface of particles modify the behaviour like pigment. The best conditions to work a pigment suspension and the best conditions settle down to which the pigment offers excellent benefits, directed to reductions of costs in manufacture and application of the pigment because of smaller costs of operation, low power consumptions in agitation and handling of higher concentrations in suspensions (obtaining of the low viscosities), and as pigment their characteristics of dispersability and colorimetric improved.

4:15 PM

Study on Structure and Properties of Glass Ceramics from Converter Slag: Jiang Maofa[1]; Zhang Dayong[1]; Shi Peiyang[1]; *Haiyan Zheng*[1]; [1]Northeastern University

Glass ceramics with converter slag as a raw material were prepared with the melting method. The effects of the mass fraction of converter slag on crystallization behavior and properties were studied by SEM, XRD and EDS. The results showed: With the mass fraction of converter slag increasing, the transition temperature and melting temperature of glass gradually dereased; the intensity of diffraction peak gradually increased, and the crystal phase of glass ceramics were Ca1.018(Mg0.733Fe0.293)((Si1.67Fe0.304)O6) with Ca(Fe, Mg)Si2O6, and the results of EDS showed there were Fe, Ca, Si, Mg and Al in glass, which the same as the results of XRD; the crystals rate increased, and the dimension of the crystals firstly increased and then decreased; the michardness and the bulk density firstly decreased and then increased, which were the maximum when the mass fraction of converter slag was equal to 70%.

4:35 PM

Synthesis of Superconductor Oxide Ceramics by Combustion of Carbon Nanoparticles: *Karen Martirosyan*[1]; Eduard Galstyan[2]; Y. Xue[2]; Dan Luss[3]; [1]University of Houston, Department of Chemical and Biomolecular Engineering; [2]University of Houston, Texas Center for Superconductivity; [3]Department of Chemical and Biomolecular Engineering, University of Houston

We report a novel cost-effective and simple method to produce a superconductor YBa2Cu3O7-x powder (1-2μm) by self-sustaining one step process, named Carbon Combustion Synthesis of Oxides (CCSO). It produces YBCO much faster (order of seconds) than the common calcination process (order of hours) without any external power consumption and using rather inexpensive raw materials. In CCSO the exothermic oxidation of carbon (-393.5 kJ/mol) nanoparticles generates a thermal reaction wave with temperature up to 850°C that propagates at a velocity of 1 mm/s through the solid reactant Y2O3, BaO2,

CuO mixture converting it to the YBCO. The carbon is not incorporated in the product and is emitted as CO2 from the sample generating high porous (~70%) and friable product. The variations of the magnetization of as-synthesized samples have been determined by SQUID magnetometer and show the onset of superconducting transition at ~91K with shielding fraction of 44% of the -1/(4p) value.

4:55 PM
Oxidation Resistance and Characterization of Nb-Cr-W Superalloys at Elevated Temperatures: *Daniel Castro*[1]; [1]University of Texas - El Paso

The importance of turbine engines in aerospace and energy sectors of industry has lead to the research of high temperature, low oxidation, super alloys for turbine engine component fabrication. An interest in niobium based superalloys has developed due to it's high service temperature capability and oxidation resistance when alloyed with chromium and tungsten. Samples from two different Nb-Cr-W alloy compositions were oxidized at temperatures 700-1400°C and analyzed under a SEM. Previous research on similar alloys with significantly less Chromium illustrated Nb2O4 as the primary oxide constituent. However, alloys studied in this research have affirmed new oxides and oxidation mechanisms in the Nb-Cr-W alloy system. A change in oxidation mechanism is apparent in the samples exposed to temperatures in excess of 900°C. In the resolve of this research, identifying a new oxide formation and mechanism has affirmed the need for more oxidation research in Nb-Cr-W alloys.

Computational Thermodynamics and Kinetics: Integrated Computational Materials Engineering

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, ASM Materials Science Critical Technology Sector, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Phase Transformations Committee
Program Organizers: Yunzhi Wang, Ohio State University; Long-Qing Chen, Pennsylvania State University; Jeffrey Hoyt, McMaster University; Yu Wang, Virginia Tech

Wednesday PM — Room: 288
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Jeff Simmons, Air Force Research Laboratory; Zi-Kui Liu, Pennsylvania State University

2:00 PM Invited
MatCASE and CCMD: Integrating Materials Simulation and Design: *Zi-Kui Liu*[1]; [1]Pennsylvania State University

Individual phases are the building blocks of microstructures which dictate the performance of materials. Developing efficient approaches in accurately obtaining properties of individual phases is critically important in creating knowledge base for materials design and simulation, and promoting the paradigm shift towards integrated computational-prediction and experimental-validation approaches. In this presentation, our approach integrating first-principles calculations and CALPHAD modeling is presented through calculations of thermodynamic properties, thermal expansion coefficient, lattice parameters, elastic coefficients, and diffusion coefficients. Those properties are further used as guidance in processing design and input data in phase-field simulations of microstructure evolutions in the framework of our Materials Computation and Simulation Environment (MatCASE) [J. Comput-Aided Mater. Des., Vol.11, 2004, 183–199] and the NSF Center for Computational Materials Design [Proceedings Education and Professional Development, Materials Science & Technology 2006, Cincinnati, Ohio, 2006, pp. 111-118].

2:25 PM Invited
Discovery of Novel Hydrogen Storage Materials: An Atomic Scale Computational Approach: *Christopher Wolverton*[1]; [1]Northwestern University

Practical hydrogen storage for mobile applications requires materials that contain large amounts of hydrogen, have low decomposition temperatures, and fast kinetics for absorption and desorption. Unfortunately, no reversible materials are currently known that possess all of these attributes. We present here results from an effort we have been developing over the past few years directed a predicting novel hydrogen storage materials from a computational first-principles approach. Such an approach requires several key capabilities: (i) Accurate prediction of decomposition thermodynamics, (ii) Prediction of crystal structures for unknown hydrides, and (iii) Prediction of preferred decomposition pathways. We show examples of progress we've made in these three areas: (i) prediction of hydriding enthalpies and free energies across a wide range of hydride materials, (ii) prediction of crystal structures for multivalent alanates and borohydrides, [such as Ca(AlH4)2 and Mg(BH4)2], and (iii) predicted decomposition pathways for Li4BN3H10 and destabilized systems based on combinations of LiBH4, Ca(BH4)2 and metal hydrides. These capabilites have led to the prediction of several novel high-density hydrogen storage materials and reactions.

2:50 PM
Multiscale Simulation of Processing of Ni-Base Superalloys: Nils Warnken[1]; *Henrik Larsson*[2]; Roger Reed[1]; [1]University of Birmingham; [2]KTH

The solidification and subsequent homogenisation treatment of a Ni-base superalloy have been simulated using phase-field (MICRESS), a front-tracking method and a method based on homogenisation (part of the DICTRA software). Obtained results are compared with experimental data. Directional solidification and further cooling are modelled using macro-scopic heat transfer Finite Element Analysis (ProCAST), yielding temperature history data for use in the micro models. We discuss the relative advantages of the different methods and demonstrate how these tools can be used to optimise the homogenization heat treatment of complex Ni-base superalloys.

3:05 PM
Modelling of the Discontinuous Precipitation Reaction with the Phase-Field Method: Lynda Amirouche[1]; *Mathis Plapp*[2]; [1]Université des Sciences et de la Technologie Houari Boumediene ; [2]Ecole Polytechnique

The discontinuous precipitation reaction appears in many industrial alloys and often results in a degradation of the alloy microstructure, accompanied by a drastic deterioration of its mechanical properties. It transforms a supersaturated mother phase into a two-phase structure consisting of lamellar precipitates and the depleted mother phase. The rate-limiting process is the surface diffusion of solute along grain boundaries and interfaces. We formulate a phase-field model for the description of the discontinuous precipitation reaction and study the structure and growth speed of regular (spatially periodic) precipitation fronts. We find that the shape and the steady-state growth speed of the precipitates are strongly influenced by the relative magnitude of surface diffusivity, volume diffusivity, and grain boundary mobility. We also find that steady states do not exist for certain ranges of these parameters. These findings will be discussed and compared to several theories for discontinuous precipitation.

3:20 PM
Diffusion Databases for Mg-ICME: *Nagraj Kulkarni*[1]; Peter Todd[1]; Yong-ho Sohn[2]; [1]Oak Ridge National Laboratory; [2]University of Central Florida

The development of high fidelity diffusion databases is an integral component of a global Integrated Computational Materials Engineering (ICME) initiative in Mg-based alloys. In the current approach, an experimental approach for the development of robust tracer diffusion databases in light metal alloys systems is proposed that does not invoke any of the previous assumptions in the phenomenological theory of diffusion. Such an approach is based on modern SIMS measurements of diffusion depth profiles of stable isotopes (tracers) on samples prepared using conventional or combinatorial techniques. In comparison with previous diffusion measurements made using radioactive isotopes, it is expected that the time required for measurements and the subsequent analysis to develop a tracer diffusion database using stable isotopes will be significantly reduced. The current effort will include 4 ternaries from the Mg-Al-Mn-Zn system. Preliminary results will be presented.

3:35 PM Break

4:05 PM Invited
Developments in Phase Field Modeling for Ni-Base Alloys in Industrial Applications: *Jeff Simmons*[1]; Y.H. Wen[2]; B. Wang[3]; Yunzhi Wang[3]; James Lill[4]; S.L. Chen[5]; Fan Zhang[5]; [1]Air Force Research Laboratory; [2]UES, Inc; [3]Ohio State University; [4]HPTi; [5]CompuTherm, LLC

Phase Field modeling has become popular because of its ability to simulate realistic microstructures that compare directly with microscopy and give information about local heterogeneities. Industrial applications generally require non-isothermal processing of complex alloy systems of 10 or more elements, so modifications of the method were required to simulate concurrent nucleation and growth of reduced constitutional complexity systems. The concurrent nucleation and growth model allows for simulations of non-isothermal transformations and for validation of proposed transformation mechanisms. A pseudobinary model was developed that reduces the constitutional complexity, but involves approximating a complex system with a fictitious binary system, so an exact fit is not possible. We are, thus, developing an algorithmic calibration method that minimizes deviations from target values. Finally, a ternary model is being developed that accounts for transient diffusional effects. This model directly integrates commercial CALPHAD-type databases into the simulation. Selected results will be given.

4:30 PM Invited
Phase-Field Model for Grain Growth in Nanocrystalline Metals: *Ingo Steinbach*[1]; Xiaoyan Song[2]; [1]RWTH-Aachen; [2]Beijing University of Technology

We investigate the correlation between size dependent thermodynamics and grain growth kinetics of nano crystalline metals both theoretically and experimentally. The theoretical approach is based on the multi-phase-field method considering volume expansion of the grain boundaries with respect to the bulk crystal. Also temperature dependence of the elastic constants is taken into account to investigate the destabilizing effect of increased temperatures on nanograin structures. Experimental results in pure nano crystalline Co showing a discontinuous nanograin growth at a certain temperature are used to verify the model.

4:55 PM
Model for Ion Beam Synthesis of Nanocrystals: *C. Yuan*[1]; I. Sharp[1]; S. Shin[1]; C. Liao[1]; J. Guzman[1]; J. Ager III[2]; E. Haller[1]; D. Chrzan[1]; [1]Lawrence Berkeley National Laboratory; Department of Materials Science, University of California at Berkeley; [2]Lawrence Berkeley National Laboratory

A model for the ion beam synthesis of nanocrystals within an amorphous matrix is developed. The model includes implantation of high-energy ions, motion of these ions in the matrix, and ion beam damage to the growing clusters. The model is studied using both a rate-equation based theory and kinetic Monte Carlo simulations, and the predictions of both methods are in substantial agreement. The model demonstrates that during implantation the island size distribution evolves through a nucleation phase to a growth phase and, ultimately, to a distribution that represents the balance between growth and ion beam damage. The model is compared with experimental results for Ge implanted into silica and results in a scaled nanocrystal size distribution that is in excellent agreement with experimental observations. This work was supported by the U. S. Department of Energy under contract No. DE-AC02-05CH11231.

5:10 PM
Examination and Simulation of the Transformation Path in Ti-Al-Nb Alloys: *Orlando Rios*[1]; Damian Cupid[1]; Hans Seifert[2]; Fereshteh Ebrahimi[1]; [1]University of Florida; [2]Technical University Bergakademie Freiberg

Ti-Al-Nb alloys with at least 40% Al have shown promising properties for high temperature applications. The phase diagram for this system has been well researched however several controversies including the extension of the beta phase field into the aluminum rich corner exist. We have combined computational and experimental efforts to evaluate the transformation paths in Al rich corner of this system. We have limited our studies to the two-phase region where gamma, TiAl(+Nb) and sigma, Nb2Al(+Ti) phase are stable in the 1200 to 800C temperature range. DTA and high temperature XRD techniques with capabilities up to 1600°C were employed in order to determine the transformation temperatures and stable majority constituent phases, respectively. Experimental observations revealed an extended beta phase field beyond the previously reported phase boundary. The CALPHAD methodology was used to calculate the liquidus surface and isothermal phase diagrams which were subsequently optimized implementing the experimental results. NSF-DMR0605702.

5:25 PM
A Ternary Phase-Field Model and Its Application to Precipitation Behavior in Ni-Al-Cr Alloys: *You-Hai Wen*[1]; James Lill[2]; Shuang-Lin Chen[3]; Jeff Simmons[4]; [1]UES Inc.; [2]High Performance Technologies, Inc.; [3]CompuTherm LLC; [4]Air Force Research Laboratory/MLLM

We developed a ternary phase-field model linking to Pandat software directly [CompuTherm, LLC] for thermodynamic evaluations. Using this model, we examined the effect of diffusivity and initial compositional/ordering profiles on the growth process of precipitate. We focused on the composition evolution with time in both the precipitates (gamma prime) and the matrix (gamma) and compared them with experimental observations by Sudbrack et al. [Acta mater., 54:3199,2006]. Our results indicate that random distribution of Cr in the nuclei is important to achieve comparable results with experimental observations. Some analysis of these results will be presented.

5:40 PM
Phase Field Simulations of Substrate-Coating Interactions for Nb-Si Based Alloys: *Sujoy Kar*[1]; Bernard Bewlay[1]; Ying Yang[2]; Bernd Boettger[3]; [1]GE Global Research; [2]CompuTherm LLC; [3]ACCESS Materials and Processes

Nb-Si based composite alloys are promising materials for application as high-temperature structural materials. Typically Nb based alloys are used in conjunction with a silicide based coating. The phase stability issues at the substrate-coating interface are critical during exposure at elevated temperatures. Phase field modeling techniques can be used effectively to simulate the behavior of the matrix-coating interface during exposure. In this work, a binary Nb-Si based matrix with a Si based coating has been simulated using the Phase-field methods to study the phase stability and motion of the interface at high temperature. The nucleation and growth mechanisms of the different phases that form at the interface have been studied.

Electrode Technology Symposium (formerly Carbon Technology): Cathodes Manufacturing and Developments

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Carlos Zangiacomi, Phelps Dodge International Corporation; John Johnson, RUSAL Engineering and Technological Center LLC

Wednesday PM Room: 299
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Morten Sorlie, Elkem Aluminium ANS; Ketil Rye, Elkem Aluminum

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2:00 PM
Constitutive Laws of Carboneous Materials of Aluminium Electrolysis Cell: Current Knowledge and Future Development: *Donald Picard*[1]; Guillaume D'Amours[2]; Mario Fafard[1]; [1]Laval University, Aluminium Research Centre - REGAL; [2]NRC Aluminium Technology Centre

The Hall-Héroult cell behaviour at different stages of the electrolysis process is an important point to take into consideration in the design and the optimization the cell. Nowadays, numerical simulation has become a powerful and essential tool since in situ measurements are difficult to perform and cost expensive. For those numerical simulations, constitutive laws and their parameters' identification in laboratory are required for all the relevant physics of the cell materials. For the mechanical behaviour of the cell, many efforts have been done to characterize, to understand and to develop constitutive laws for the carboneous materials. Plasticity, viscosity, visco-elasticity, baking, etc., are examples of phenomena which have been addressed up to date, on both transient and steady state situations. This paper presents an overview on various level of modeling of the mechanical behaviour of the carbon cell lining material, from elastic to more complex like a thermo-(chemo)-visco-elasto-plactic one.

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Cathode Hot Patching to Prolong Cathode Life at DUBAL: *Maryam Al-Jallaf*[1]; Ali Mohamed[1]; Joseph Antony[1]; [1]Dubai Aluminium Company Limited

The deterioration of cathode blocks may occur by general erosion, characterised by 'W' wear in more graphitised materials and by localised erosion, also known as potholing. Pothole failure is predominant in some cell technologies due to high amperage or certain cathode grades. The three main factors to contribute in the theory of pothole formation are cathode weakness area, high current density and an increase in metal pad velocity. Until 2003, Dubal focussed on using fused alumina, corundum, to hot patch the potholes in order to cease iron attack, thereby prolong cathode life. The cost-benefit analysis was derived in terms of key performance indicators, labour and corundum cost to determine the break even point of extended cathode life in a patched cell. This paper summarises Dubal's experience with fused alumina hot patching in D18 cell technology with respect to stall location and perform overall cost-benefit analysis.

2:40 PM

High-Performance Preparation Plant for Cathode Paste: *Berthold Hohl*[1]; Valery Burjak[2]; [1]Maschinenfabrik Gustav Eirich GmbH and Company KG; [2]OAO Ukrgrafit

In the course of a modernization program, the Ukrainian company Ukrgrafit has converted part of their paste preparation line for cathodes and carbon blocks from conventional mixing technology to Eirich intensive mixers. The new plant was put into operation in the middle of 2006. Apart from the mixer, it comprises coke heating, intermediate storage of the finished mix, transport to the presses as well as the entire automation. The substantially improved homogeneity of the mix as well as the complete documentation of the preparation process clearly refined the quality of the final products. The paper describes the criteria leading to the decision on the system as well as the realization of the task definition. Moreover, first operating results are presented.

3:00 PM

Twelve Years of Experience with a Fully Automated Gas Preheating System for Søderberg and Prebake Cells: *Odd-Arne Lorentsen*[1]; Ketil Rye[2]; [1]Hydro Aluminium; [2]Elkem Aluminum

A preheating system with propane burners suitable for both Prebake and Søderberg cells was successfully developed more than 10 years ago at Elkem Aluminium. Two burners are controlled by a fully automated computer program with a predetermined heating rate, and they are pulsing according to the temperature on top of the cathode blocks. The new preheating concept makes it possible to increase the temperature in the cathode lining in a smooth and uniform manner up to 970°C. By improving the start-up procedure, we now start the cells without an initial anode effect. The paper presents our experience with the preheating system and how to obtain a smooth start-up and early operation.

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Cell Preheat/Start-up and Early Operation: *Ketil Rye*[1]; [1]Elkem Aluminum

The most common methods for preheating industrial aluminum pots are reviewed and advantages and disadvantages of each method are presented. Also, the present industrial trend of reducing pot turnaround times is commented and measurements of deep-lining temperatures at start-up are related to the pot behavior in early operation. In general it is seen that shorter preheating times is unfavorable for pot stability in early operation and this effect may limit the potential for further pot turnaround time reductions or lead to operational problems and/or reduced potlife.

3:40 PM Break

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Properties of Pitch and Furan-Based TiB_2-C Cathodes: *Mohamed Mahmoud*[1]; Trygve Foosnæs[2]; Harald Øye[2]; [1]Aluminium Company of Egypt (Egyptalum); [2]Norwegian University of Science and Technology

Some characteristic properties of pitch and furan-based TiB_2-C bulk samples were studied. The open porosity of pitch and furan-based TiB_2-C bulk materials is 13.3% and 34.6%, respectively. The compressive strength of pitch-based samples is twice the value of furan samples (39 and 16.5 MPa, respectively). The X-ray diffraction patterns of baked pitch and furan-based TiB_2-C samples showed the presence of TiC and $TiBO_3$ due to the oxidation of TiB_2 and/or the reaction between TiB_2 and carbon in the presence of diffusing oxygen. The TGA study in nitrogen atmosphere showed weight losses of 6.2% and 2% for green and baked pitch-based TiB_2-C samples, and 11.2% and 7% for green and baked furan-based TiB_2-C samples, respectively. There was a significantly different behaviour between pitch and furan-based TiB_2-C samples in thermal dilatometer experiments. Pitch-based TiB_2-C samples showed higher shrinkage than furan-based TiB_2-C samples after soaking.

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Electrodeposition of TiB_2 from Cryolite-Alumina Melts: *Dmitry Simakov*[1]; Sergei Vassiliev[2]; Parviz Tursunov[2]; N. Khasanova[2]; Viktor Ivanov[1]; Artem Abakumov[2]; N. Alekseeva[2]; Evgenii Antipov[2]; Galina Tsirlina[2]; [1]Russian Engineering Company Ltd.; [2]Moscow State University

Formation of TiB_2 and various by-products under cathodic polarization is studied in relation to in situ deposition of coatings on graphite directly from cryolite-alumina bath. This procedure can provide a cheap cathode coating having very good wettability. In order to evaluate the nature of products, voltammetry and multistep potentiostatic technique are applied in combination with XRD, SEM and local analysis. For CR between 2 and 3 and alumina content of 2-8 wt.%, current efficiency of TiB_2 formation is found to pass a maximum at current densities of ca. 0.4-0.5 A/cm^2. Parallel deposition of Ti_2O_3 and silicides is confirmed in certain potential regions. Wetting test demonstrated the formation of homogeneous Al film on as-deposited coatings even under open circuit conditions. Wettability was much better as compared to samples with brushed TiB_2-based coating. A disadvantage of the deposited coatings is its dendrite microstructure which may be improved upon in future.

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Creep Deformation in TiB_2/C Composite Cathode Materials for Aluminum Electrolysis: *Jilai Xue*[1]; Qing-Sheng Liu[1]; [1]University of Science and Technology

Diffutional creep deformation in carbon/TiB_2 cathode materials mainly due to sodium penetration during aluminum electrolysis was investigated in a modified Rapoport apparatus. Experimental data for the cathode materials with varying amount of TiB_2 addition were obtained against varying temperature and compressive pressure. Sodium penetration and expansion were measured at the same time along with the diffusional creep process under compressive pressure. In general the creep deformation process shows a trend of increasing with prolonged time of aluminum electrolysis and increased temperatures of cell operation. The addition of TiB_2 into the carbons can, however, reduce the diffusional creep deformation. Numerical simulations were also applied to compare and analyze the effects of various material properties and processing parameters. The technical information obtained is useful for cell design and relining by using the carbon/TiB_2 cathode materials.

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Penetration of Sodium and Electrolyte to Vibratory Compaction TiB_2 Cathode: Zhaowen Wang[1]; *Yungang Ban*[1]; Zhongning Shi[1]; Shaohua Yang[1]; Hongmin Kan[1]; Xiaozhou Cao[1]; [1]Northeasten University, College of Materials and Metallurgy

Vibratory compaction TiB_2 and 30 mass% graphite contained block were used as cathodes in aluminum electrolysis. In the same condition, after 5 h electrolysis, the depth of electrolyte penetrated into the 30 mass% graphite cathode was about 13 mm. It was deeper than that into TiB_2 cathode which was less than 1 mm. SEM and EDS analysis showed that Na, F, Al penetrated slightly into the vibratory compaction TiB_2 cathode much less than that into the 30 mass% graphite cathode. Vibratory compaction TiB_2 cathode can decrease sodium penetration effectively, but it can not hinder sodium penetration thoroughly. The penetration resistance mechanism of vibratory compaction TiB_2 cathode to sodium and electrolyte was also discussed in this paper.

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Effect of Grain Gradation on Tapped Packing Efficiency in Preparing TiB_2-C Composite Cathode for Aluminum Electrolysis: Xiaojun Lü[1]; *Jie Li*[1]; Yan-qing Lai[1]; Qing-yu Li[1]; Zhang-Liang Tian[1]; Zhao Fang[1]; [1]Central South University

The effect of the size distribution of coarse-fine binary particles and the volume fraction of fine particles on the vibratory packing efficiency and porosity of TiB_2 powder were investigated. The results showed that the vibratory packing efficiency increased and the porosity decreased when the size ratio (R) of coarse

particles to fine particles increased. In addition, the vibratory packing efficiency was also governed by the volume fraction of coarse or fine particles, and reached its maximum when the volume fraction of fine particles is 30%~40%. Comparing the experimental results to the values calculated by the Furnas model, it is found that the Furnas model suits TiB_2 ceramic powder system, and the value of the parameter C2 in Furnas model is determined according to experimental results.

Emerging Interconnect and Packaging Technologies: Pb-Free Solders and Other Interconnects: Microstructure, Modeling, and Test Methods

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

Program Organizers: Carol Handwerker, Purdue University; Srinivas Chada, Medtronic; Fay Hua, Intel Corporation; Kejun Zeng, Texas Instruments, Inc.

Wednesday PM Room: 275
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: C. Robert Kao, National Taiwan University; Carol Handwerker, Purdue University

2:00 PM

In-Situ Observations of Unidirectional Solidification in Sn-0.7Cu and Sn-0.7Cu-0.06Ni by X-Ray Imaging: Yosuke Yamamoto[1]; Kazuhiro Nogita[2]; *Christopher Gourlay*[2]; Arne Dahle[2]; Kentaro Uesugi[3]; Hideyuki Yasuda[1]; [1]Osaka University; [2]University of Queensland; [3]Japan Synchrotron Radiation Research Institute, SPring-8

It has previously been shown that Ni additions in the range of 400-1000ppm alter the microstructure and improve the wave soldering performance of Sn-0.7Cu-xNi alloys. However, the influence of Ni additions on the microstructure formation during solidification is not fully understood. In recent years, in-situ observations of solidification of metallic alloys have been developed using time-resolved X-ray radiation in the synchrotron. This paper reports on the results from in-situ observations of unidirectional solidification of Sn-Cu and Sn-Cu-Ni solders at the SPring-8 synchrotron radiation facility. The solidification characteristics of Sn-0.7Cu and Sn-0.7Cu-0.06Ni at growth rates in the range of 1-10μm.s-1 are compared and discussed.

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As Solidified and Aged Microstructure Control Through X Modification in SAC+X Alloys: *Jason Walleser*[1]; Iver Anderson[2]; Fran Laabs[2]; Joel Harringa[2]; [1]Iowa State University; [2]Ames Laboratory

The ideal solder microstructure contains a fine phase distribution while minimizing the size of any brittle phase. A coupled eutectic fits this description. Unfortunately, due to the difficulty in nucleating tin, a 100% eutectic microstructure for Sn rich solder alloys has remained elusive and pro-eutectic Ag_3Sn plates often plague Ag bearing alloys. However, by careful study of Sn-Ag-Cu-X alloys using simulated DSC solder joints, pro-eutectic Ag_3Sn plates can be mitigated and eutectic volume maximized by controlling X and its concentration. Additionally, previous research shows that certain X additions also slow diffusion and resulting IMC growth. Aged samples, identical in composition to the DSC samples, will also be prepared, examined, and mechanically tested. The primary difference is that at this time, the lower concentrations of X will be used. Zn and Mn appear most promising.

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An Investigation of Microstructure and Mechanical Properties of Pb-Free Solders as a Function of Alloy Composition and Cooling Rate: *Sun Kyoung Seo*[1]; Sung Kang[2]; Hyuck Mo Lee[1]; Da-Yuan Shih[2]; [1]Korea Advanced Institute of Science and Technology; [2]IBM Corporation

The microstructure and microhardness are investigated for various Pb-free solders; Sn-Cu, Sn-Ag, and Sn-Ag-Cu. The Cu content examined varies from 0.5 to 2 wt % in Sn-Cu, while the Ag content varies from 0.5 to 3.5 wt % in Sn-Ag or Sn-Ag-Cu. The effects of minor alloying elements such as Cu, Ni and Zn are also examined with various Sn-rich solders. The cooling rates employed during solidification range from 0.02°C/s, to 1-5°C/s and to 100°C/s. Sn grain size and orientation are observed by cross-polarization light microscopy and electron backscatter diffraction(EBSD). The microhardness is measured to find the variation of mechanical properties in terms of alloying contents and cooling rates. It is found that the alloy composition, minor alloying elements, and cooling rate significantly affect the Sn grain size and hardness in various solder alloys. Sn grains become smaller with increasing minor alloying contents, and also with the fast cooling rate.

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Effect of Reflow Profile on Sn Undercooling and Microstructure Evolution of Sn-3.5Ag and Sn-3.9Ag-0.6Cu Lead Free Alloys: *Yan Xing*[1]; James Woods[1]; Eric Cotts[1]; [1]Binghamton University, Department of Physics and Materials Science

This study focuses on the effects of high temperature annealing on the Sn solidification temperature in Sn-Ag-Cu solder, and on the final microstructure of Sn-Ag-Cu samples. Reflow temperature, reflow time and number of reflows were varied in eutectic Sn-3.5Ag and Sn-3.9Ag-0.6Cu alloys. It was found that the solidification temperature of the Sn phase decreases with higher reflow temperature, and with longer annealing times at elevated temperatures. Cross sectioned samples were examined by means of optical microscopy and scanning electron microscopy. The size and number of Ag_3Sn and Cu_6Sn_5 precipitates was found to vary dramatically with reflow conditions. For instance, in samples which had been reflowed at high temperatures (~400C-500C), no primary Ag_3Sn precipitates (plates) were observed in the cross sections of both Sn-3.5Ag and Sn-3.9Ag-0.6Cu solder balls. The rate of nucleation and growth of Ag_3Sn in undercooled melts was examined and found to vary with reflow conditions.

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Role of Au in the Massive Spalling at the SnAgCu/Ni Interface: *Su-Chun Yang*[1]; C. Rober Kao[1]; [1]National Taiwan University

Massive spalling of intermetallic compound in the SnAgCu/Ni system has been reported in the literature. In this study, we pointed out that the presence of a Au layer between solder and Ni has the effect of triggering the massive spalling at even earlier stage. Experimentally, Sn-Ag-Cu lead-free solders of various compositions were reflowed on Au/Ni metallization. The thickness of Au layer was varied from 0 to 3 μm. The thicker the Au layer is, the easier massive spalling can occur. The reason for the Au effect will be presented in this talk.

3:15 PM Break

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Effects of Phosphorus on Fluidity and Microstructure of Sn-xCu-yNi Lead-Free Solder: *Kazuhiro Nogita*[1]; Jonathan Read[1]; Christopher Gourlay[1]; Tetsuro Nishimura[2]; Shoichi Suenaga[2]; Arne Dahle[1]; [1]University of Queensland; [2]Nihon Superior Company, Ltd

Phosphorus is sometimes added to lead-free solders as an anti-oxidation agent during wave soldering. This paper reports on the effect of phosphorus on the maximum fluidity length, and microstructure of Sn-xCu-yNi alloys. Fluidity tests were conducted by the vacuum fluidity test (the Ragone method) using Pyrex elbow tubes (800mm x 150mm and ID:3mm) at a constant superheat of TLiq+40K. The results clearly demonstrate that phosphorus additions decrease the maximum fluidity length. The most significant decrease in fluidity length occurs in the range 20 to 90 ppm P in Sn-0.7Cu-0.05Ni-zP alloys. The mechanisms responsible for these effects are discussed in terms of the nucleation and growth dynamics and microstructure in this system.

3:45 PM

Cross-Interaction between in Ni/Sn5Ag/Cu and Ni/Sn/Cu Solder Joints: *Hua-wei Tseng*[1]; Liu Chengyi[1]; [1]National Central Unversity

Recently, peoples start realizing the importance of the mutual interaction between two different interfacial reactions in solder joints. It has been reported by many researchers that the dissolved elements from metal pads, such as Cu and Au, would diffuse across the molten solder and influence the opposite solder/metal pad interfacial reactions. Cross-interaction between two interfacial reactions in Ni/Sn5Ag/Cu and Ni/Sn/Cu solder joints were investigated. In this talk, we will report: (1) In Ni/Sn/Cu sandwich structure, the asymmetrical microstructure across Ni/Sn and Sn/Cu interfaces was found, which greatly

affects the interfacial strength in Ni/Sn/Cu solder joint. (2) In Ni/Sn5Ag/Cu sandwich structure, we found that Ag tend to migrate and form Ag_3Sn compound on the Ni side with longer reflow time. The Ag migration mechanism will be discussed based on the isothermal Sn-Ag-Cu diagram.

4:00 PM
First Principles Modeling of Copper Interconnect Resistance: *Donald Nicholson*[1]; X.-G. Zhang[1]; Bala Radhakrishnan[1]; Nagraj Kulkarni[1]; Sirish Namilae[1]; Li An-Ping[1]; [1]Oak Ridge National Laboratory

First principles quantum mechanical methods are needed to accurately model electron transport in interconnects once they are so narrow that the width and grain size approach the electron mean free path. We have used a local density based approach to calculate the resistance of individual grain boundaries. The Layer KKR multiple scattering code is used to calculate the transmission of Bloch waves through tilt and twist boundaries. Various levels of atomic and electronic relaxation of the grain boundary are investigated. A procedure for incorporating local electronic transport results into a lattice Boltzmann model corresponding to particular interconnect grain structures will be described. Research sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory (ORNL), managed by UT-Battelle, LLC for the U. S. Department of Energy under Contract No. DE-AC05-00OR22725.

4:15 PM
Characterization of Tungsten Carbide Electrical Contacts for High-Temperature Electronics: *Claudiu Muntele*[1]; Kennesha Nettles[2]; Daniel Walker[2]; Satilmis Budak[1]; Abdalla Elsamadicy[2]; Daryush Ila[1]; [1]Alabama A&M University; [2]University of Alabama in Huntsville

Silicon carbide based electronics components are sought after for applications in harsh environments (high temperature, corrosive atmosphere etc.). For such applications, electric connects made of tungsten carbide seems to be the best choice, for chemical and mechanical stability. In this work we are investigating the stoichiometry, microstructure, and electrical behavior of tungsten carbide thin films on silicon carbide substrates. We used Rutherford Backscattering spectrometry and X-ray photoelectron spectroscopy for measuring the stoichiometry and depth profile, scanning electron microscopy to monitor the surface morphology change, and Van der Pauw electrical measurements for determining the sheet resistivity as a function of operating temperature, ranging from room temperature to 800°C.

WEDNESDAY PM

Emerging Methods to Understand Mechanical Behavior: Diffraction Methods: Synchrotron X-Ray

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Brad Boyce, Sandia National Laboratories; Mark Bourke, Los Alamos National Laboratory; Xiaodong Li, University of South Carolina; Erica Lilleodden, Forschungszentrum

Wednesday PM — Room: 285
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Donald Brown, Los Alamos National Laboratory; Stefan Zaefferer, Max Planck Institute for Iron Research

2:00 PM Invited
Using Quantitative Texture Methods and Finite Element Analysis to Understand Internal Stresses in Deforming Polycrystals: *Matthew Miller*[1]; Jun Park[1]; Tong Han[2]; Paul Dawson[1]; [1]Cornell University; [2]Yonsei University

Stress state is a key factor for many important phenomena related to processing and performance of engineering alloys. Microplasticity, recrystallization, phase transformation and crack initiation are driven by stress state on the size scale of the individual crystal. In this talk, a synchrotron x-ray based experimental method – motivated by quantitative texture analysis – designed to measure the orientation-dependent distributions of crystal stresses over a deforming aggregate is described. The experimental data from tests conducted on sintered copper are compared to finite element results from a crystal-based simulation methodology. Stress distributions are compared component by component at each load level. To better understand trends over orientation space, we also expand both sets of data in a series of spherical harmonic functions and compare the modes and coefficients between experiment and simulation.

2:30 PM
A Method to Determine the Full Second Order Strain Tensor from a Single 2D Diffraction Pattern: *Apurva Mehta*[1]; David Bronfenbrenner[2]; James Belasco[1]; Matthew Bibee[3]; Monica Barney[4]; M.R. Mitchell[4]; Alan Pelton[4]; [1]Stanford Synchrotron Radiation Laboratory; [2]University of California, Berkeley; [3]Stanford University; [4]Nitinol Devices Corporation

When a structural element is subjected to external load, the resultant mechanical deformation is a complex response function best captured by a second order strain tensor. This tensor can be thought of as a description of a 3D ellipsoidal surface, where the deviation (eccentricity) from a spherical surface is the measure of local strain along that direction. Conventional x-ray diffraction strain measurement analyzes one point on this ellipsoid. The sample is rotated along two non-colinear axes to sample many different locations on the ellipsoid and thus determine the full strain tensor. This forms the basis of the sin-squared-psi (ssp) technique. We will present an adaptation of the ssp technique using a large area detector, which will yield the full second order strain tensor from a single diffraction pattern. We will show a comparison of the strain tensors obtained from "stationery", single-shot method to the ones obtained from standard ssp method.

2:50 PM
Determination of Local Strain for a More Fundamental Understanding of Mechanical Response in Materials: *Monica Barney*[1]; David Bronfenbrenner[2]; Sam Daly[3]; Apurva Mehta[4]; M.R. Mitchell[1]; Alan Pelton[1]; [1]Nitinol Devices Coporation; [2]University of California, Berkeley; [3]California Institute of Technology; [4]Stanford Synchrotron Radiation Laboratory

In order to move toward a more fundamental understanding of the strain response to load on a local level in materials, development of a non-destructive, spatially resolved strain measurement technique using highly coherent, monochromatic synchrotron X-rays is underway. By studying conventional elastic-plastic strain behavior of the common metallic crystal structures (FCC, BCC, HCP), the validity of this newly developed approach can be verified and these X-ray measurements compared to global strain quantities to shed light on a more detailed understanding of mechanical behavior. In addition, the precise onset of plastic deformation should be readily determined from the changes in the X-ray diffraction peaks and compared to the transition between deformation modes observed in global strain measurements.

3:10 PM
Micromechanical Study of Superelastic and Shape Memory NiTi by Novel Synchrotron X-Ray Diffraction Techniques: *David Bronfenbrenner*[1]; Monica Barney[2]; M. Mitchell[2]; Apurva Mehta[3]; Alan Pelton[2]; Ronlad Gronsky[1]; [1]University of California-Berkeley; [2]Nitinol Devices Corporation; [3]Stanford Synchrotron Radiation Laboratory

Conventional strain measurements treat the specimen as a homogeneous continuum and provide a macroscopic assessment of the elastic and plastic strain in response to applied force. In the current study, strain is assessed at the microscopic level for superelastic and shape memory NiTi. In response to an applied global displacement, NiTi experiences four modes of micromechanical deformation. Reversible deformation consists of both bond stretching (Hookian elastic) as well as thermodynamically driven phase transformations. Irreversible deformation consists of dislocation driven deformation (conventional plasticity) as well as low-symmetry crystallite reorientation. The ellipsoidal X-ray diffraction technique used in this study is ideally suited to distinguish between these four deformation modes at the microscopic level. Our group has used in-situ synchrotron x-ray diffraction to distinguish and quantify the micromechanical response of deforming superelastic and shape memory NiTi and has compared these with conventional global strain measurements.

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Diffraction Stress/Strain Analysis in Integrated Circuits: Conal Murray[1]; *Ismail Noyan*[2]; [1]IBM T.J. Watson Research Center; [2]Columbia University

Modern microelectronic circuits are heterogeneous devices which contain very significant locked-in manufacturing strains. These strains form the basis of "strained-silicon" devices with enhanced performance characteristics. Because of the current device dimensions, independent measurement of these strains is a non-trivial exercise. In this paper we will discuss the technology, physics and mechanics relevant to strained-Si devices and summarize our attempts at measuring these strain fields using microbeam x-ray diffraction.

4:15 PM Invited
Single Grain Characterization Techniques at the APS 1-ID Beamline: *Ulrich Lienert*[1]; Jonathan Almer[1]; Bo Jakobsen[2]; Wolfgang Pantleon[2]; Henning Poulsen[2]; Christopher Hefferan[3]; Robert Suter[3]; [1]Argonne National Laboratory; [2]Risø National Laboratory; [3]Carnegie Mellon University

The use of high-energy synchrotron x-rays provides a unique combination of bulk penetration power (mm to cm) with high spatial (about 1 micrometer) and temporal resolution. The APS 1-ID beamline is dedicated to high-energy diffraction and the status of the single grain diffraction program will be presented. Recently, a dedicated setup for diffraction imaging has been installed. A forward modeling algorithm has been developed to reconstruct the grain boundary outlines. Grain maps of polycrystalline aluminum have been reconstructed. Strain sensitivity is achieved in the far-field regime. The technique has been extended to high reciprocal space resolution by novel x-ray optics providing a narrow bandwidth beam and by positioning an area detector at very long distance behind the sample. The use of area detectors provides fast data acquisition, and therefore three-dimensional reciprocal space maps can be recorded. The reciprocal space resolution of about 0.001 1/Å is almost isotropic.

4:45 PM
Using X-Ray Microbeams to Assess Long Range Internal Stresses in Materials: *Michael Kassner*[1]; Lyle Levine[2]; Bennett Larson[3]; Jon Tischler[3]; Peter Geantil[1]; [1]University of Southern California; [2]National Institute of Standards and Technology; [3]Oak Ridge National Laboratory

The presence of counterbalanced stresses within microscopic volumes (or cells) in deformed materials was predicted more than two decades ago and has been inferred from numerous indirect experiments. Yet, direct proof of their existence had been elusive, as spatially-resolved measurements of the stress magnitudes and distributions critical for testing theories and computer modeling were not possible until recently. Researchers using the intense, submicron, x-ray beams at the Advanced Photon Source made the first quantitative, spatially resolved measurements of elastic strains within dislocation cells. The measurements indicated that the structural materials were under significant, variable internal stresses of opposite direction on submicron length scales corresponding to the dislocation substructure. The results provide critical data for validating and guiding the development of detailed dislocation-based simulations and models for every major facet of dislocation structure evolution and dislocation transport.

5:05 PM
In-Situ Investigation of Twinning in Individual Mg Grains with Synchrotron X-Rays: *Cahit Aydiner*[1]; Bjorn Clausen[1]; Joel Bernier[2]; Ulrich Lienert[3]; Don Brown[1]; Carlos Tome[1]; [1]Los Alamos National Laboratory; [2]Lawrance Livermore National Laboratory; [3]Argonne National Laboratory

Formation of tensile twins and their stress evolution in individual Mg grains is investigated with a state-of-the-art, high-energy synchrotron X-ray diffraction method. In-situ measurements are performed under compressive load where sample rotations in each step allow recording of diffraction spots from most atomistic planes of targeted grains. Analysis of these spots allows identification of individual grains through their orientation and yields their strain tensor. It is crucial to note that these grains are investigated in the bulk, in their native environment.Here, the known crystallographic relation between the parent grain and twin variant means that the location of the diffraction spots of the prospective twin can be predicted. We have observed the realization of these spots (formation of the twin) and the increase in their intensity with further loading (growth of the twin). Ultimately, we detail the stress interaction between the twin and the parent at a single grain level.

5:25 PM
Microstructure as Seen by X-Ray Line Broadening, Its Relevance to Mechanical Behavior: *Tamas Ungar*[1]; [1]Eotvos University

Mechanical behavior, in a broader sense, is determined by the microstructure. The major constituents of microstructure are composition, second phases, dislocations, grain boundaries and planer defects, especially stacking faults and twin boundaries. X-ray line profile analysis proves to be a powerful method for charcterizing these microstructure elements both, qualiatively and quantitatively. The dislocation model of strain anisotropy enables the precise determination of dislocation densities and charcters together with subgrain size and size-distribution. The Taylor and Hall-Petch models of strength can be evaluated by using these data. Twinning can be determined quatitatively by X-ray diffraction and, together with scanning electron microscopy, provides a comprehensive microstructure characterization, especially in hexagonal metals.

Energy Conservation in Metals Extraction and Materials Processing: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Aqueous Processing Committee, TMS: Pyrometallurgy Committee, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Edgar Vidal, Brush Wellman, Inc.; Cynthia Belt, Aleris International Inc

Wednesday PM — Room: 287
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Edgar Vidal, Brush Wellman, Inc.

2:00 PM
Develop a Synergic Thermal-Mechanical Process for Al-Mg-Si Alloys: *Mingdong Cai*[1]; [1]University of Houston

AlMgSi extrusion alloys are usually processed sequentially as casting, homogenization, extrusion and artificial ageing treatment (T5/T6). Due to low ageing temperature, it usually takes over 1000 minutes for peak-ageing treatment. This largely limits the manufacturing efficiency and requires large energy consumption. We propose to overcome this issue through combining conventional processing techniques (such as rolling, extrusion and forging) and ageing, a synergic thermal-mechanical process named dynamic ageing. Preliminarily, dynamic ageing of two AlMgSi alloys at 170°C was conducted by equal channel angular extrusion (ECAE). It is shown that the ageing time-scale is reduced to around 10 minutes, and the dynamically aged samples have higher ultimate tensile strength than statically peak-aged samples with comparable ductility. It is therefore concluded that ECAE-aided dynamic ageing is efficient in executing ageing treatment that results in superior mechanical properties. We expect that this new technique have great potential in automobile and aerospace industries.

2:20 PM
Direct Electrochemical Production of Titanium Alloys from Oxide Precursors: *Kevin Dring*[1]; [1]Norsk Titanium

Current production of titanium alloys is accomplished via the Kroll process; a discontinuous, greenhouse gas emitting process that is labor intensive. A more efficient process based on electrolysis was forecasted by Kroll himself over fifty years ago, however, this prophecy has remained unfulfilled. The present work demontrates the production of low-oxygen beta titanium alloys - of both conventional and novel compositions - via the direct electrochemical reduction of blended oxide precursors in molten salt electrolytes at elevated temperatures. Electroanalytical and materials characterisation techniques were applied to partially and fully deoxidized material in order to ascertain the reduction mechanisms and pathways. A beneficial effect on the reduction pathway of titanium dioxide was obtained from the presence of secondary oxides during the reduction process. The earlier onset of beta titanium formation greatly facilitated the extraction of the final remnants of oxygen from the metallic titanium.

2:40 PM

Electrochemical Synthesis of Non-Oxide Ceramic Powders in a Eutectic $CaCl_2$-NaCl Melt: *Xiao Yan*[1]; Mark Pownceby[1]; Mark Cooksey[1]; Marshall Lanyon[1]; [1]CSIRO

A fused salt electrochemical process was investigated for synthesis of non-oxide ceramic powders directly from metal oxide, boric oxide, and carbon powders by electro-deoxidation. Sintered pellets of the mixed powders were designed to act as a composite cathode in the electrolytic cell. Electrolysis was carried out in molten $CaCl_2$-NaCl eutectic at 3.1 V and at 600-900ºC under argon. The favoured cathodic reaction was the removal of the oxygen from the oxides to produce metal atoms that reacted with either the reduced boron atoms, forming a metal boride, or the carbon initially present in the sintered pellets to produce a metal carbide. The technical viability of the process was demonstrated by the electro-deoxidation of the sintered pellets of TiO_2/B_2O_3 and TiO_2/C to produce TiB_2 and TiC, respectively. Compared to the existing carbothermic reduction process, this new electrochemical process is much less energy intensive and may lead to lower production costs.

3:00 PM

On Promoting Energy Conservation in Hydrometallurgical Processing of Complex Materials: *Katragadda Sarveswara Rao*[1]; [1]Institute of Minerals and Materials Technology (Formerly Regional Research Laboratory (CSIR), Bhubaneswar)

The present paper firstly describes some recent trends from literature on energy management via process intensification to arrive at sustainable technologies for aqueous processing of minerals, metals and materials. Secondly, the energy saving measures practiced in leaching, solid-solution separation, solution purification, metal and material winning and environmental aspects have been discussed. The emerging importance of interdisciplinary nature of hydrometallurgy for practical applications is also highlighted. With the above background, the present paper describes an overview of energy conservation and autoclave processes with a special reference to hydrometallurgical research activities of our Laboratory.

3:20 PM

Control and Optimization of Teniente and PS Converters Operations for Energy Efficiency and Process Intensification: *Florian Kongoli*[1]; F. Condore[2]; R. Riveros[2]; I. McBow[1]; E. Z. O'Brien[1]; J. Bobadilla[2]; G. Achurra[2]; [1]Flogen Technologies, Inc.; [2]Codelco-Chile

Energy efficiency is one of the most important issues for various existing and new extraction processes. An efficient process uses the minimum possible energy in various forms and ways in order to extract or process several materials. This is closely related to several factors from decreasing the number of operational problems and accidental shutdowns to the control, optimization and automation of these processes. In this paper, the authors discuss their recent work in effectively controlling and optimizing the day-to-day operational parameters in Teniente and PS Converters as well as the intensification, optimization and automation of these processes through a unique non-equilibrium physical process modeling that correctly simulate the industrial furnace processes and avoid guessing and uncertainties. The advantages of this new approach have been discussed.

3:40 PM Break

4:00 PM

Sulfide Ore Looping Oxidation Process: Maximizing Energy Recovery and Minimizing Environmental Impact: Larry McHugh[1]; *Jean Mozolic*[2]; [1]Orchard Material Technology LLC; [2]The Mozolic Consulting Group LLC

This paper is the subject of a recent patent application demonstrating major improvements in sulfide ore processing. This two-step "looping oxidation" process effectively removes sulfur while producing materials of adequate purity in an energy proficient and environmentally friendly manner. The first step results in a metal sub-oxide and a highly concentrated sulfur oxide off-gas stream. This gas stream can be processed for recovery in a more effective manner regarding both energy and capital equipment costs. In the second step, this metal sub-oxide is further oxidized to efficiently generate energy that can be recovered and utilized without the typical complications of an "acid gas" stream process. Additionally, a portion of the final fully oxidized product can be looped back to the first step as the oxidizing agent. The process will be described in detail and compared to existing technologies in the following areas: production efficiency, energy utilization, sulfur recovery and cost.

4:20 PM

Simulation of the Thermopower of Zn_4Sb_3: *Zhongliang Xiao*[1]; Dan Liu[1]; Qiyuan Chen[1]; [1]Changsha University of Science and Technology

A new method was proposed to calculate the absolute thermopower of semiconductors from their density of states. In metals the electrochemical potential of the electrons is a unique function of the temperature. In semiconductors, the potential is also a unique function of temperature T, if valence electron concentration n_e in turn is a well-defined function of T. The temperature dependence of the potential can then be represented by two parts, one at constant electron concentration plus one due to the known change of the electron concentration. The second term is small in semiconductors. The thermopower of a semiconductors is related to the ratio either of two transport coefficients or of the gradients of the electrochemical potential of the electrons and the temperature. A function obtainable from the density of states of the metal as illustrated on a thermopower isotherm of Zn_4Sb_3.

4:40 PM

Preparation of Metal Lithium by Molten Salt Electrolysis with Li_2CO_3 as Raw Material: *Jidong Li*[1]; Mingjie Zhang[1]; Tingan Zhang[1]; Dan Li[1]; Zhuo Zhang[1]; [1]Northeastern University

Metal lithium was prepared by using graphite anode, wire cathode and a mixture of LiF-LiCl as the molten salt electrolysis with Li_2CO_3 as raw material in a laboratory cell. The back electromotive force was measured by continuous pulse-computer. The result is as follows: with the current density and temperature increasing, the back electromotive force increases in terms of logarithm and decreases respectively. When 2g Li_2CO_3 is added into the fused mass of LiF-LiCl, the back electromotive force decreases significantly by 0.8V. Then the back electromotive force comes back gradually with Li_2CO_3 consumed by electrolyzing continuously, so it is determined that the period of feeding Li_2CO_3 is 20mins. At 550°C, the 3.8g metal lithium was obtained finally by electrolyzing in 5A for 4h, which the current efficiency can reach 72%.

5:00 PM

The Influence of Different Calcination Atmosphere on the Photocatalytic Reactivity of Water Splitting to O_2 over Rutile TiO_2: Daoxin Wu[1]; *Qiyuan Chen*[1]; Zhoulan Yin[1]; Jie Li[1]; [1]Central South University

The Rutile TiO_2 were prepared under air, Ar and H_2 calcination atmosphere by low temperature hydrolysis using Tetrabutyl titanate. Powers were characterized by by power X-ray diffraction, UV-vis diffuse reflectance and X-ray photoelectron spectroscopy. The influence of the calcination atmosphere on the photocatalytic reactivity of Rutile TiO_2 for oxygen production was investigated. The photocatalytic reactivity of Rutile TiO_2 prepared under air, Ar and H_2 atmosphere was compared under ultraviolet and visible light radiation with Fe^{3+} as electron acceptor. The results showed that Rutile TiO_2 prepared under Ar and H_2 atmosphere had higher photocatalytic activity for oxygen production than that prepared under air atmosphere. The oxygen production rates under ultraviolet irradiation were 427.5,378.2 and 357 μmol. $L^{-1}h^{-1}$ respectively when Rutile TiO_2 was prepared under Ar,H_2 and air atmosphere. The oxygen production rates under visible light irradiation were 78.2,52.2 and 13.5 μmol.

5:20 PM

Electrochemical Behavior of Pb-Ag-Bi Alloys as Anodes in Zinc Electrowinning: Zhong Shui-ping[1]; Lai Yan-qing[1]; Jiang Liang-xing[1]; *Li Jie*[1]; Liu Yexiang[1]; [1]Central South University

A new type Pb-Ag(0.8wt%)-Bi(0~5wt%) alloy for used as anode in electrowinning of zinc from sulphuric acid electrolytes was prepared. The electrochemical and corrosion properties of this ternary lead alloys have been tested and compared with those of the Pb-Ag(1wt%) anodes used in industry. Results showed that the corrosion resistance and anodic overpotential of the new anodes was not affected when the content of Bi was less than 3 wt%, but when the content of Bi was 3 wt%, the anodic overpotential was about 40–80 mV lower than that of Pb-Ag(1wt%) alloy. The formation of oxide layers on the surface of these alloys was traced by cyclic voltammetric methods and the composition of the anodic oxide layers was characterized by X-ray and SEM analyses. The surface layer on the two type anodes examined was composed mainly of $PbSO_4$, α–PbO_2 and ß–PbO_2. But different structure of the surface layer was observed.

General Abstracts: Electronic, Magnetic, and Photonic Materials Division: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: Biomaterials Committee, TMS: Chemistry and Physics of Materials Committee, TMS: Electronic Materials Committee, TMS: Electronic Packaging and Interconnection Materials Committee, TMS: Nanomaterials Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Sung Kang, IBM Corporation

Wednesday PM Room: 276
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Long Qing Chen, Pennsylvania State University; Sung Kang, IBM Corporation

2:00 PM
Lightning Causes and Effects – A Systematic Protection Approach for the Aluminum Industry: *Franco D'Alessandro*[1]; [1]ERICO Products Australia Pty Ltd

The continuous production of primary aluminum relies on a reliable electricity supply and the use of data acquisition, automation and control technologies to maximise profits. The losses incurred as a result of a lightning incident in the aluminum industry cannot be underestimated, e.g., Alcoa Tennessee shutdown in April 2007 and the subsequent downtime of about 7 weeks. A key first step in today's highly technological environment is the implementation of a risk management plan, so the paper discusses how to carry out a lightning risk assessment. Secondly, the paper presents the theme that, whilst no single technology can guarantee 100% prevention of damage, a "six point protection approach" provides a comprehensive yet practical checklist covering the major damage mechanisms related to direct-strike, surge and transient effects. Finally, the paper discusses the importance of a low impedance ground plane and good connections throughout the site.

2:20 PM
Magnetic Domain Evolution and Strain Mechanism in Terfenol-D Crystals: *Yongxin Huang*[1]; Yongmei Jin[1]; [1]Texas A&M University

Phase field model is used to simulate the magnetic domain evolution and the corresponding deformation process in magnetostrictive Terfenol-D crystals. The model considers the two competing magnetization mechanisms, i.e., magnetization rotation and domain wall movement, addresses the magnetic field-induced deformation by magnetoelastic coupling through magnetostriction phenomenon, and takes into account the domain microstructure-dependent long-range magnetostatic and elastostatic interactions. The simulations quantitatively describe the magnetization and deformation processes. The results reveal the interplays of the two magnetization mechanisms, the interactions of magnetic domains with underlying crystallographic microstructures (growth twins, polycrystals), and the effects of compressive stresses on magnetostrictive behaviors.

2:40 PM
Magneto-Optical Study of Cobalt Ferrite Nanoparticles: Byron Scott[1]; *Kevin Stokes*[1]; [1]University of New Orleans

We present a magneto-optical study of $Co_xFe_{1-x}Fe_2O_4$ nanoparticles, where 0=x=1. The ferrite nanoparticles were produced using a generic wet-chemical synthesis procedure. Stoichiometric amounts of Fe^{2+}, Fe^{3+} and Co^{2+} salts are dissolved in a non-aqueous polar medium (diethylene glycol). A coprecipitation reaction with sodium hydroxide produces ferrite nanoparticles with average diameter of 6 nm. The nanoparticles can be stabilized in water, or, alternatively, the nanoparticles can be treated with a hydrophobic capping ligand with a carboxylic acid or amine head group and suspended in a non-polar organic solvent. Faraday rotation was measured on nanoparticle samples dried on an amorphous silica substrate. The magneto-optical spectra show the characteristic features of both Fe_3O_4 and $CoFe_2O_4$ as x increases as well as evidence of electronic transitions involving both Co^{2+} and Fe^{2+}. The results are discussed in terms of the distribution of cations in the crystal lattice.

3:00 PM
Microwave Response of Patterned Nanosize Stripes of Exchange Biased Bilayers: *Leszek Malkinski*[1]; Minghui Yu[1]; Donald Scherer[1]; [1]University of New Orleans

The exchange bias is the interface phenomenon originating from the spin coupling of two different magnetic materials and it manifests as asymmetry or a shift of the hysteresis loop. The understanding of this phenomenon is still incomplete and there exist a few competitive theories of the effect. Majority of research on exchange bias systems was carried out on thin film systems. We present experimental study of static and dynamic magnetic properties of exchange biased nanostructures of Co and IrMn. The samples have a form of arrays of nanosized stripes. They were fabricated by electron beam nanolithography, magnetron sputtering and lift off process. The results of the hysteresis loop measurements using SQUID magnetometer and microwave response by the means of ferromagnetic resonance technique of the arrays will be compared to those of the continuous reference bilayers. The results will be discussed in the light of existing models of exchange bias.

3:20 PM Break

3:50 PM
Monte Carlo Simulation Model for Charge Transport in Polymers: *Nenian Charles*[1]; Brandon Howard[1]; Pedro Derosa[2]; [1]Grambling State University; [2]Louisiana Tech University/Grambling State University

Since the discovery that conjugated polymers can conduct electricity, provided adequate doping is added to them, there has been vast research to better understand the physical, electrical and chemical properties of these molecules. Furthermore, tools to simulate the conductive behavior are of great interest nowadays. A number of models have been proposed so far. A Monte Carlo-based method early proposed by Bässler and coworkers have proved successful in reproducing a number of experimental findings. We propose a model based on the Bässler model to study conduction in polymers which adds a realistic description of the polymer network. Configurational disorder is reproduced by disordered arrangements of hopping sites rather than by a parameter. In addition, the model proposed here allows for the association between hopping sites and polymers, thus intramolecular conduction can be distinguish from intermolecular conduction. The initial testing of this model vs. the Bässler model has produced encouraging results.

4:10 PM
Transparent Al-Doped ZnO Thin Films Synthesized by RF Magnetron Sputtering: *Soo Young Seo*[1]; Changha Kwak[1]; Yongbyung Lee[1]; Sunhong Park[2]; Seonhyo Kim[1]; Sangwook Han[3]; [1]POSTECH; [2]Research Institute of Industrial Science andTechnology; [3]Chonbuk National University

Transparent and conductive Al-doped zinc oxide thin films were fabricated on Al_2O_3 substrates by a RF-magnetron sputtering procedure from a ZnO target mixed with a various wt% Al_2O_3. The quantity of aluminum at the target was varied from 2 to 5 weight %. The structural and optical characteristics of Al-doped ZnO thin-films were studied using field emission scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), UV-VIS-IR and photoluminescence (PL) measurements. The XRD measurements revealed that all of Al-doped ZnO thin-films had a wurtzite crystal structure and had the (002) preferred orientation. We did not observed any extra peak from the XRD patterns. The best visual light transparency of the films was 90%. The band gap of the films was about 3.35 eV and the visual light absorption edge was about 350 nm. We will discuss the chemical and optical properties, comparing with the structural properties and Al-doping ratio.

General Abstracts: Structural Materials Division: Microstructure/Property Relations of Steels II

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: Product Metallurgy and Applications Committee, TMS: Refractory Metals Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Titanium Committee
Program Organizer: Ellen Cerreta, Los Alamos National Laboratory

Wednesday PM Room: 387
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Hany Ahmed, Netherlands Institute for Metals Research

2:00 PM
Substructural Analysis of Adiabatic Shear Bands in Standard and Enhanced Rebar Steel Using Transmission Electron Microscopy: *Lisa Dougherty*[1]; Ellen Cerreta[1]; George Gray[1]; Carl Trujillo[1]; Mike Lopez[1]; [1]Los Alamos National Laboratory

Rebar steel that has been enhanced in composition and microstructure for optimal corrosion and strength characteristics exhibits greater microstructural instability at high strain rates than standard rebar. Adiabatic shear bands have been observed to form in enhanced rebar steel at strain rates below that required for shear band formation in standard rebar, indicating an increased propensity for shear localization in the enhanced rebar steel. Optical microscopy and scanning electron microscopy, as well as electron backscattered diffraction, indicate complete annihilation of the original microstructure within the shear bands of enhanced rebar, whereas residual, highly deformed grains can be found within the shear bands of standard rebar. This current study utilizes transmission electron microscopy to examine the microstructure inside the shear bands and at the boundaries of the sheared regions in both the standard and the enhanced rebar steels to understand the differing mechanisms operating during shear localization in these materials.

2:20 PM
Microstructure Evolution in Nano/Submicron AISI 301 Austenitic Stainless Steel: Shreyas Rajasekhara[1]; *Paulo Ferreira*[1]; Pentti Karjalainen[2]; Antero Kyrolainen[3]; [1]University of Texas at Austin; [2]University of Oulu; [3]Outokumpu Stainless Oy

Nano/submicron grained AISI 301 austenitic stainless steel has been achieved by heavy cold rolling to 52% reduction, to induce the formation of martensite, and subsequent annealing at 600°C, 800°C and 1000°C from 1 to 100 seconds. The microstructural evolution was analyzed using transmission electron microscopy (TEM) and X-ray diffraction (XRD), which reveal that the as-cold rolled samples are textured and primarily consist of heavily deformed lath-type martensite. Subsequent annealing at 600°C results in negligible martensite to austenite reversion, while the texture is retained from the cold-rolled samples. However, upon annealing at a higher temperature of 800°C and 1000°C, significant austenite reversion and M23C6 carbide precipitation is observed. As carbide precipitation typically occurs in stainless steels that are annealed for long durations (>0.1 hours/360 seconds), this work shows for the first time carbide precipitation in short annealed (1, 10 and 100 seconds) commercial austenitic stainless steels.

2:40 PM
Oxide-Dispersion Strengthened Ferritic Alloys by Internal Oxidation: *Joachim Schneibel*[1]; Sang Shim[1]; [1]Oak Ridge National Laboratory

Oxide-dispersion strengthened (ODS) ferritic alloys such as MA956, MA957, and PM2000 contain high number densities of fine oxide particles with diameters as small as a few nm. Their processing involves mechanical alloying of metallic powders with Y2O3, which is an expensive and energy-intensive process. An alternative process, which is used commercially in ODS copper alloys, is internal oxidation. This process was applied to the intermetallic compounds Fe17Y2 and Fe11TiY at temperatures ranging from 600 to 1000°C. Oxide particles or lamellae as small as ~10 nm were obtained, and the room temperature nano-hardness increased significantly with decreasing particle size. Preliminary results on the internal oxidation of Fe-Ti-Y solid solution alloys will also be presented.

3:00 PM
Structure and Strength of Quenched Martensite in Fe-C Steels: *Oleg Sherby*[1]; Jeffrey Wadsworth[2]; Donald Lesuer[3]; Chol Syn[3]; [1]Stanford University; [2]Battelle Memorial Institute; [3]Lawrence Livermore National Laboratory

The lattice parameters of quenched martensite change discontinuously as a function of carbon content. The discontinuity occurs at 0.6 wt%C, designated as the H point. Martensite in quenched Fe-C steels is described based on the formation of two different, but sequential, martensitic transformations. The first transformation, designated as primary martensite, is the only transformation process for steels from nearly zero to the H point. Primary martensite (known as lath martensite) is interpreted as evidence for the formation of martensite with a BCC structure containing meta-stable carbide particles. Above the H point, the structural changes during quenching occur by the formation of primary martensite followed by secondary martensite where the BCT structure is obtained. Secondary martensite is the acicular structure of martensite. The hardness of quenched martensite is reviewed and explained on the basis of the two different martensites. Nano-carbides and fine cell/subgrain structures are the principal contributors to strength.

3:20 PM
Textural Characteristics and Magnetic Properties of High Strength Electrical Steels: *Sam Chang*[1]; Hee Park[1]; [1]Pohang University of Science and Technology

For the recent developoment of new high strength electrical steel to meet the requirement for high fatigue resistance of core materials using for large electric motors, it is of important to investigate the textural characteristics and its relation with magnetic properties and fatigue strength. The steels are desighed to obtain yield strength of 450 N/mm2 and core loss less than 3 Watt/Kg and some special elements like Nb, Sn, and Ni are added to 3%Si steel to increase strength as well as to improve magnetic properties. Textural features depending on alloying elements are quantitatively analyzed and relationship of texture with tensile strength and fatigue strength is clarified.

3:40 PM Break

4:10 PM
The Effect of Boronizing and Austempering on the Wear Behavior of a Ductile Iron: Seckin Akray[1]; *Murat Baydogan*[1]; Huseyin Cimenoglu[1]; Eyup Kayali[1]; [1]Istanbul Technical University

Boronizing is a surface hardening method to improve hardness and wear resistance creating a hard layer at the surface. Austempering is an isothermal annealing to improve strength and toughness giving an acicular ferrite morphology in the microstructure. In this study, combining effect of these two processes on surface hardness and wear resistance were investigated. A ferritic ductile iron was boronized at 900C for 90 min. and then transferred to an isothermal salt bath at 300C and held at that temperature for several durations, and then air cooled. Borided layer was characterized by XRD and SEM. Microhardness measurements were performed from the surface to the center of the specimen, indicating that a high hardness of 1200 HV was obtained at the surface. Reciprocating wear tests performed revealed that wear resistance increased 6.5 times after boronizing and austempering when compared to the as cast state.

4:30 PM
Effect of Strain Rate on High Temperature Mechanical Properties of PM2000 Alloy for Advanced Nuclear Reactor Applications: Md Rahman[1]; K. Raja[1]; *Carl Nesbitt*[1]; Manoranjan Misra[1]; [1]University of Nevada Reno

Next generation nuclear reactors will operate at relatively higher temperatures and severe environmental conditions. It is critical to understand the behavior of structural materials under these severe conditions. Ferritic ODS alloy (PM 2000) which has excellent creep and oxidation resistance at temperature up to 1200°C is a candidate for these purposes. In this study high temperature mechanical properties of PM2000 has been evaluated as a function of strain rate. Failed specimens were characterized by using an ex-situ Scanning Kelvin Probe Force Microscope which measures the variation of work function of specimen surface

due to microstructural variations and can be used for estimating the remnant lifetime of the component. Preliminary Results showed a decrease in stress and strain as the strain rate decreased from 1.0×10^{-3}/s to 1.0×10^{-5}/s. Results from mechanical tests as well as characterization of failed specimen using SKPFM, SEM, EDX and optical microscopy will be presented in the paper.

4:50 PM
Passive Film Characteristics and Corrosion Performance of 9Cr/0.1C Microcomposite Steel in Simulated Concrete Pore Solutions: *Greg Kusinski*[1]; Mauricio Mancio[2]; Tom Devine[2]; [1]Clemson University, School of Materials Science and Engineering; [2]University of California, Berkeley

The passive film formed on the Microcomposite steels, (wt%: C 0.1, Cr 9.0, Mn 0.6, bal. Fe; microstructure consisting of multilayers of lath martensite with thin films of retained austenite at lath boundaries), in highly alkaline environments was investigated. A series of in-situ Surface-Enhanced Raman Spectroscopy (SERS) experiments were conducted in conjunction with cyclic potentiodynamic polarization curves over a wide range of potentials, in order to characterize the formation and breakdown mechanisms of the passive films. A solution of 0.55M KOH + 0.16M NaOH, with and without chlorides (0% and 3.5% NaCl), was used for the SERS experiments. In addition, the corrosion performance of Microcomposite steels as a function of pH (9–14) and ionic strength of the solution (from 10-5 to 1M) was evaluated via polarization resistance (Rp) experiments, and compared to the performance of regular carbon steel rebar.

5:10 PM
Structural Strength Variation of Freight Car Carbody during Static Loadings: *Jeongguk Kim*[1]; Jung-Won Seo[1]; Sung-Cheol Yoon[1]; Sung-Tae Kwon[1]; [1]Korea Railroad Research Institute

The structural strength assessment for carbody of freight car in railway applications was performed using engineering analysis techniques to verify the structural strength of newly manufactured carbody. The freight car was designed with A441 steel and stainless steel for the railway transportation. Prior to the assessment of structural strength, finite element method (FEM) characterization was used for the stress and structural analyses on stress distribution in a carbody of freight car. The strain gages were attached on the carbody based on the FEM results. The actual vertical loading test and horizontal compression loading test were conducted, and the results were compared with the previous FEM results. In this investigation, the evaluation method for the structural strength in a structural component has been introduced using several engineering techniques, and experimental and theoretical results were compared.

Hael Mughrabi Honorary Symposium: Plasticity, Failure and Fatigue in Structural Materials - from Macro to Nano: Mechanical Properties of Ultrafine-Grained (UFG) Metals II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: K. Jimmy Hsia, University of Illinois; Mathias Göken, Universitaet Erlangen-Nuernberg; Tresa Pollock, University of Michigan - Ann Arbor; Pedro Dolabella Portella, Federal Institute for Materials Research and Testing; Neville Moody, Sandia National Laboratories

Wednesday PM — Room: 386
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Marc Meyers, University of California, San Diego; Oliver Kraft, Forschungszentrum Karlsruhe

2:00 PM Keynote
Mechanical Properties of Nanocrystalline Ni as Studied by Nanoindentations and Creep Tests: *Mathias Göken*[1]; Johannes Mueller[1]; [1]University Erlangen-Nürnberg

Nanocrystalline Ni with average grain sizes between 25 nm and some hundred nm was produced by pulsed electrodeposition (PED) to study the mechanical properties in the temperature range from 298 K to 473 K by nanoindentation tests and compression creep tests. Nanoindentation is a suitable method to characterise the mechanical properties of these materials including strain rate sensitivity. The maximum flow stress, the strain rate sensitivity of flow stress and the rate of work hardening increase with decreasing grain size. The results are compared with data available for coarse grained and ultrafine-grained materials and an interpretation of the data is given in terms of the responsible deformation mechanism. It is shown by Auger analysis of in-situ fractured specimens that impurities which are deposited during the electrodeposition process at the grain boundaries play an important role on the deformation behavior.

2:30 PM Invited
Stress Generation Mechanisms in Thin Films Impacted by Energetic Ions: Sulin Zhang[1]; Sachin Terdalkar[2]; Joseph Rencis[2]; *K. Jimmy Hsia*[1]; [1]National Science Foundation; [2]University of Arkansas

Molecular dynamics simulations are performed to study the stress generation mechanisms in carbon thin films in the forms of graphene sheet (2D) and diamond (3D) impacted by energetic carbon neutrals. The simplicity of the 2D cantilever graphene sheet model enables direct observation of free-end deflection during the impact, while the 3D model captures more realistic physics. Both simulations demonstrate that the stress due to the impact of energetic C neutrals is strongly dependent on the incident energy. At low-energy impact, the film undergoes tensile stress; a transition from tensile to compressive stresses occurs at an intermediate impacting energy; the compressive stress peaks at around 50eV; further increasing the incident energy lowers the compressive stress. Examinations of the atomic stress and film microstructures reveal that the formation of the film stress is attributed to competing mechanisms between production and annihilation of point defects. A simple analytical model satisfactorily explains the results.

2:50 PM Invited
Deformation of Nanocrystalline Metals and Alloys Investigated by Microcompression and Nanoindentation: Ravi Kottada[1]; Ruth Schwaiger[1]; Jörg Weissmüller[1]; Jürgen Markmann[1]; Yulia Ivanisenko[1]; *Oliver Kraft*[1]; [1]Forschungszentrum Karlsruhe

Pure nanocrystalline metals have been studied with an overwhelming interest in the last two decades due to their attractive and unusual mechanical properties. However, only little information is available on the mechanical behavior of nanocrystalline metallic alloys. Here, we show comparative micro-compression and nanoindentation test results for nanocrystalline metals and alloys, namely electrodeposited nanocrystalline Ni and Ni-20%Fe, and Pd and Pd-Au prepared by inert gas condensation and subsequent densification via high pressure torsion. Micro-columns with diameters ranging from 1 – 3µm were prepared by Focused Ion Beam (FIB) machining. Subsequently, the micro columns were uniaxially compressed to strains of 10-50% using a nanoindenter equipped with a flat diamond punch. Nanoindentation experiments at various indentation strain rates complement the mechanical testing. The results, which were analyzed with respect to strain hardening and strain rate sensitivity indicate that the alloyed metals show a weaker strain rate sensitivity compared to their pure counterparts.

3:10 PM
Influence of Grain Boundary Character on Nanocrystalline Plasticity: *Mark Tschopp*[1]; Shreevant Tiwari[2]; Sudipto Ghosh[2]; David McDowell[1]; [1]Georgia Institute of Technology; [2]Indian Institute of Technology Kharagpur

The objective of this research is to use atomistic simulations to investigate the influence of the grain boundary character distribution on the plasticity of nanocrystalline materials. In this work, we generate a 3D periodic columnar grain structure about the <110> tilt axis using an algorithm that enables the preferential assignment of low order CSL boundaries. Three nanocrystalline configurations are generated: one with increased Σ3 content, a random distribution, and one with increased Σ9 content (lower nucleation stresses). Deformation simulations under uniaxial tension show that the strength of nanocrystalline materials is enhanced with higher populations of Σ3 boundaries. However, the grain boundary plane inclination also plays a role in plastic deformation. Last, the influence of the grain size in plastic response is investigated to determine if increasing the low order CSL boundary content has a diminished influence in regimes where grain rotation and grain boundary sliding are the dominant deformation mechanisms.

3:25 PM

Mechanical Properties of Nanostructured Al-Cu Alloy Produced by High Energy Ball Milling and Hot Isostatic Pressing: T. Shanmugasundaram[1]; V. Subramanya Sarma[1]; B. Murty[1]; Holger Saage[2]; *Martin Heilmaier*[2]; [1]Indian Institute of Technology Madras; [2]Otto von Guericke University

Nanostructured materials exhibit usually improved strength but at the expense of ductility. The development of bi-modal grain size distribution with micron-sized grains embedded in a matrix of nanostructured grains has been shown to restore ductility without significant loss in strength. In the present work, we present the mechanical properties of nanostructured precipitation hardening Al-Cu alloy prepared by high energy ball milling of the inert gas atomized Al-Cu alloy powder. After milling, X-ray diffraction (XRD) analysis was used to evaluate the grain size. After 15 h of milling, 30 nm crystallite size was obtained. Subsequently, these nano powders were blended with 15 and 30 wt.pct. coarse alloy powder (1 to 10 μm). Blended powders were degassed and consolidated by hot isostatic pressing. The microstructures and mechanical properties of the samples will be characterized with the aim of establishing optimized microstructures and processing conditions for simultaneously achieving high strength and ductility.

3:40 PM Break

3:50 PM Invited

Micro and Nano Defects Formation During Grain Refinement by ECAP: *Rimma Lapovok*[1]; Peter McKenzie[1]; Dacian Tomus[1]; Yuri Estrin[1]; Terry Lowe[2]; [1]Monash University; [2]Los Alamos National Laboratory

Severe plastic deformation via ECAP is a method to refine the grain size of metals. Generally, UFG metals have a superior strength and high cycle fatigue life compared to their course grained counterparts. However, the fatigue performance of various UFG metals is not always consistent with trends in tensile properties due to defects introduced during ECAP. A study of micro defects, such as decohesion of particles within the matrix, brittle fracture of particles, formation of voids at the grain boundaries have been performed using SEM and TEM. To investigate the formation of nano-defects, namely the voids of 1 to 200 nm size, Small-Angle Neutron Scattering technique has been employed. The SANS measurements on CP-Ti processed by ECAP revealed that defects in the range of 5-20 nm were not influenced by the level of back-pressure, whereas defects of the 20-200 nm size could be manipulated by application of hydrostatic pressure.

4:10 PM Invited

Analytical and Molecular Dynamics Modeling of Void Growth: Sirirat Traiviratana[1]; Eduardo Bringa[1]; David Benson[1]; *Marc Meyers*[1]; [1]University of California

Void growth is the principal failure mechanism in ductile metals. Yet, it is only recently that a dislocation mechanism was postulated by Lubarda et al (Acta Mat,1397, 2004), who showed that both prismatic and shear loops nucleating at the void surface can provide the material transport required for growth. Atomistic simulations on monocrystalline and polycrystalline copper (using the Mishin-Farkas potential)reveal the detailed nature of the dislocation activity surrounding a growing void. The emission of dislocations is the first stage, followed by their reactions and interactions. Calculations are carried out for voids having different sizes and predict different critical stresses for plasticity, in disagreement with the Gurson model, which is size independent. The calculations are applied to grain-boundary voids, important in polycrystals.

4:30 PM

Single Crystals Deformed by HPT: *Martin Hafok*[1]; Reinhard Pippan[1]; [1]Erich Schmid Institute of Materials Science

The fragmentation process of high pressure torsion deformed samples is commonly studied by the use of polycrystalline specimens. Usually it is widely accepted that the interaction between adjacent crystallites facilitates the fragmentation process, but how is the fragmentation process influenced in a single crystalline material? On account of this, single crystalline nickel samples were manufactured in the form of small discs with <111> and <001> orientation parallel to the cylinder axis to characterize the evolution of microstructure and microtexture. Due to the shear deformation a characteristic microtexture has developed that exhibits preferred orientations, depending on the alignment of the crystal system with respect to the deformation state. In addition to the characterisation of the microstructure and microtexture by BSE and EBSD measurements, the torque during the deformation can be readily measured and analysed with respect to the two different crystallographic orientations.

4:45 PM

Mechanical Anisotropy in Ultrafine Grained Copper and IF Steel Processed via Equal Channel Angular Extrusion (ECAE): Effects of Grain Morphology and Texture: *Mohammed Haouaoui*[1]; Majid Al-Maharbi[1]; Ibrahim Karaman[1]; Hans Maier[2]; [1]Texas A&M University; [2]University of Paderborn

The effect of strain path and level on the flow stress anisotropy and Bauschinger effect (BE) in UFG OFHC copper and IF steel was investigated considering grain morphology, dislocation mean free path and texture evolution. The materials were deformed via multipass ECAE using a 90° square die. The room temperature experiments were conducted under tension and compression along three perpendicular directions in each billet. Bauschinger experiments were conducted in both forward tension/reverse compression and forward compression/reverse tension modes. The strong anisotropy in flow stress, softening and BE were correlated to grain morphology and texture. The observed stronger tension strength and tension/compression asymmetry was attributed, in addition to the effect of hydrostatic pressure on dislocations, to the dislocation mean free path via grain morphology. The comparison between copper (fcc) and IF steel (bcc) elucidated further the contribution of texture on the flow anisotropy in UFG materials.

5:00 PM

High-Strain-Rate Shearing of Nanocrystalline Aluminum Film: *Buyang Cao*[1]; Zhizhou Zhang[1]; Kaliat Ramesh[1]; [1]Johns Hopkins University

Nanocrystalline aluminum film is made through vapor deposition with a mean grain size of 30nm. It is then subjected to high-rate shearing deformations with strain rates of 10^3–10^6 s^{-1}. The experimental configuration is that of a compression-torsion kolsky bar, where the specimen is a thin film mounted on a stainless steel ring. Superimposed pressures of 400 MPa are also developed. The experimental technique provides high-rate stress-strain curves as well as deformed films for microstructure analysis. The results of microscopic characterization using TEM and HRTEM are described.

5:15 PM

Effect of Annealing Twins on Microstructure Evolution in Electrodeposited Nanostructured Ni: *Indranil Roy*[1]; Farghalli Mohamed[1]; [1]University of California, Irvine

This paper presents evidence for the effect of annealing twin boundaries on microstructure evolution in electrodeposited nanostructured Ni based. This evidence is illustrated by means of transmission electron micrographs (TEM) and orientation maps generated by electron backscatter diffraction (EBSD). Statistics of annealing twin boundaries and corresponding lamellar spacing as a function of several variables, including grain size, temperature, and annealing time, to engineer a microstructure having a maximum twin density are also investigated. The results are discussed with reference to the role of annealing twinning not only in improving intergranular corrosion resistance but also in simultaneously increasing strength and ductility in nanocrystalline materials.

5:30 PM

Application of the Normalization JIC Fracture Methodology to Ductile Polymers: *Eric Brown*[1]; [1]Los Alamos National Laboratory

The single specimen normalization technique is extensively investigated for ductile polymers. This employs an analytical solution with power law behavior for tip blunting and crack initiation and transitions smoothly to a linear relationship for steady-state crack growth to accurately estimate the position of the crack tip throughout the test. The evolution and validation of the normalization technique and its application to ductile polymers is discussed. Results are presented for PTFE7C, PCTFE, Kel-F800, THV500, and PEEK450G with a focus on the effects of transitioning through the glass transition temperature and temperature induced phase transitions. Results are compared to the literature and presented in the context of extensive fractography to elucidate fracture mechanisms on length scales from the nano- to macro-scale.

Magnesium Technology 2008: Corrosion, Surface Finishing and Joining

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Wednesday PM Room: 291
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Neale Neelameggham, US Magnesium LLC; Susan Slade, US Magnesium LLC; Norbert Hort, GKSS Research Center

2:00 PM
Electrochemical Fabrication and Biocompatibility of the Hydroxyapatite Coating on Magnesium Alloy for Implanted Applications: *Shaokang Guan*[1]; Li Peng[1]; Qun Luo[1]; Cuilian Wen[1]; [1]Department of Materials Science and Engineering, Zhengzhou University

The hydroxyapatite coating composed by needle-like hydroxyapatite(HAP) fiber was fabricated on magnesium alloy substrate by electro-deposition method to improve the corrosion resistance and biocompatibility. There were vibrations of HPO4 2-, PO43-, CO32- and OH- in the coating, which was the composition of bone. The biocompatibility of the coating was deeply investigated. It reveals that there was obviously specific mass gain in the first week and the HAP coating could protect the Mg substrate from quickly degradation during the first period of immersion. The degradation in psychological environment could not cause the rise of magnesium in the serum. The HAP coating could reduce the corrosion of magnesium alloy at the first period of implanting and the HAP/Mg could stimulate the hurl of the bone tissue. The histopathology result further testified that HAP/Mg could improve the circulation of the blood of bone defects and stimulate the growth of new bone tissue.

2:20 PM
Gas Dynamic Spray (GDS) Coatings for Improving Galvanic Corrosion and Sliding Wear Resistance of Magnesium Alloys: *Jiaren Jiang*[1]; Lijue Xue[1]; [1]National Research Council of Canada (NRC)

Magnesium alloys are very attractive for many industrial applications, particularly, for reducing vehicle weight in the automobile and aerospace industry to reduce greenhouse gas emission. However, the poor wear and galvanic corrosion resistance seriously limits the wider industrial applications of magnesium alloys. In this paper, the improvement of galvanic corrosion and dry sliding wear resistance of magnesium alloys has been investigated by applying aluminum-based coatings on cast AZ91D and AM60B magnesium alloy substrates using gas dynamic spray (GDS) process. It is shown that GDS coatings can be directly applied on dry-machined or cast magnesium alloy substrate with very good bonding without requiring special surface preparations. Salt spray testing showed excellent galvanic corrosion protection results for die-cast AM60B magnesium alloy by applying the developed innovative GDS coatings. The dry sliding wear resistance of the cast magnesium alloys is also significantly improved by the aluminium-based GDS coatings.

2:40 PM
The Microstructure and Performance of AZ31 Joint Welded by Gas Tungsten Arc: *Yefeng Bao*[1]; Yongfeng Jiang[1]; [1]Hohai University

Welds of AZ31 magnesium alloy was carried out using gas tungsten arc (GTA) welding. The microstructures of AZ31 magnesium alloy base metal, heat affected zone(HAZ) and weld metal were investigated by optical and scanning electron microscopy (OM and SEM) and EDS. The fusion line between weld metal and HAZ was distinct. The HAZ was overheated and the grain size was coarser. However, the grain size of weld, which was quenched like casting and fine equiaxed grains were formed, was smaller than that of base metal and HAZ. The discontinuous network phase, Mg17Al12 and Mg17(AlZn)12 and divorced eutectic distributed around gain boundary in weld metal was detected. The hardness of weld metal was higher than base metal, where, was lower than HAZ. The corrosion resistances in weld and base metal were similar, in contrast, that of HAZ was lower.

3:00 PM
Influence of Cutting and Non-Cutting Processes on the Corrosion Behavior and the Mechanical Properties of Magnesium Alloys: *Martin Bosse*[1]; [1]Leibniz Universität Hannover

An important aim for the automotive industry is to reduce vehicle weight in order to decrease fuel consumption and emission of CO_2. The use of light metals as construction materials is generally considered of key importance for the future. Magnesium alloys fulfill the demands of a low specific weight with excellent machining properties and high recycling potential. The low corrosion resistance in comparison with other light materials is one of the reasons for the rare application as construction material. The aim of the presented joint research project within the priority program 1168 of the German Research Foundation is to influence the mechanical properties and corrosion resistance by cutting processes and mechanical surface treating through deep rolling. The corrosion and mechanical properties are significantly affected by the surface and subsurface properties of the workpiece. Therefore the influence of surface treatment and different alloy compositions on the corrosion rate has been studied.

3:20 PM
Micro Galvanic Corrosion Behavior of Mg Alloys as a Function of Aluminum Content: *Mridula Bharadwaj*[1]; Shashank Tiwari[1]; Yar-Ming Wang[1]; Vijayalakshmi Mani[1]; [1]General Motors

The corrosion behavior of the cast Mg-Al alloys was studied as a function of varying Al concentration. The beta phase is cathodic to the more reactive (anodic) primary phase called the alpha phase and hence it is critical to control the amount and distribution of this beta phase around the alpha phase. We present the polarization and direct galvanic current measurement results of the above alloys along with electrochemical impedance spectroscopy response of the surface oxide layer. These results are presented in correlation with the microstructural mapping of component phases to interpret the beta phase functionality. It was observed that a continuous network of the beta phase around the alpha phase imparts anodic-barrier role to the beta phase. The objective of these studies is to better design the Mg alloy microstructure so as to minimize the micro galvanic corrosion between component microstructural phases.

3:40 PM Invited
Thread Forming In Magnesium Alloys: *Klaus Pantke*[1]; [1]University of Dortmund

With cold forming tapping tools, it is possible to produce internal threads by cold forming. Due to this, thread strength could be significantly enhanced. The core requirement for cold forming is the adequate plasticity of the workpiece material. Insufficient plasticity of magnesium alloys limits cold tap forming. A new process of pre-forming tap offers a good opportunity to handle the described problem. To implement this, threads are produced in a two-stage operation. In the first step, an incomplete thread will be generated (machined) by a modified thread tool. The diameter of this tap is slightly less than the normal tap. In the second step, a cold forming tap is finishes the thread to final diameter. Through this procedure it is possible to increase thread strength up to 40 percent. This article will address the microstructure influence of thread forming in magnesium alloys and the resulting thread strength.

Magnesium Technology 2008: Creep-Resistant Magnesium Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Magnesium Committee
Program Organizers: Mihriban Pekguleryuz, McGill University; Neale Neelameggham, US Magnesium LLC; Randy Beals, Chrysler LLC; Eric Nyberg, Pacific Northwest National Laboratory

Wednesday PM Room: 292
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Bob Powell, General Motors Corporation; Randy Beals, Chrysler LLC

2:00 PM
Creep and Hot Working Behaviour of a New Magnesium Alloy Mg-3Sn-2Ca: Norbert Hort[1]; *Kamineni Rao*[2]; Tarek Abu Leil[1]; Hajo Dieringa[1]; V.Y.R.K. Prasad[2]; Karl Kainer[1]; [1]GKSS Research Center; [2]City University Hong Kong

With regard to the limited number of magnesium alloys in use, an approach has been undertaken to develop a new class of magnesium alloys based on the system Mg-Sn-Ca. From recent investigations the alloy Mg3Sn2Ca has been identified as one of the most promising magnesium alloys. It will be shown that this alloy offers superior creep properties even compared to the creep resistant alloy AE42. Mechanical properties will be presented as well as results from compressive creep experiments at 80 MPa and temperatures of 135-175°C. Additionally, the Mg3Sn2Ca alloy also offers good hot deformation behaviour in the temperature range of 300-550°C and deformation rates of 10-3.5-10-1 s-1, as revealed by the processing maps generated for the alloy. Compared with the most widely used alloy AZ31 the Mg3Sn2Ca can be easily hot worked over the entire temperature range investigated at fairly high strain rates, and the alloy recrystallized completely.

2:20 PM
Neutron Diffraction Measurements of Residual Stresses in Creep-Resistant Magnesium Alloys: *Dimitry Sediako*[1]; Michael Gharghouri[1]; [1]National Research Council of Canada

Recent efforts to develop high-temperature creep-resistant alloys for automotive applications have resulted in a number of experimental alloys. Chemical composition and the casting/rolling production routes frequently determine the cost and properties of the wrought material. The scope of this study was limited to evaluation of creep properties and texture evolution for the following alloying systems: Mg-Al-Ca, Mg-Al-Sr, Mg-Al-RE, and Mg-Zn-RE. Crystallographic texture strongly influences the deformation behaviour of magnesium alloys during hot forming operation. Neutron diffraction was used to characterize the texture of several creep resistant alloys produced using exactly the same sequence of permanent-mold casting and one-stage extrusion processes. Alloys with large variations in chemical composition were used in order to clearly show the effects of the major alloying elements on the resulting microstructure, crystallographic texture, and high-temperature creep properties.

2:40 PM
High Performance HPDC Alloys as Replacements for A380 Aluminum Alloy: Boris Bronfin[1]; *Nir Moscovitch*[1]; [1]Dead Sea Magnesium

One of the most direct methods of improving fuel economy and reducing CO_2 emissions is automotive light weighting. Currently the automotive industry is searching for lightweight materials for powertrain parts, which are subjected to both high temperature and dynamic loads. However, until recently, the utilization of magnesium alloys in powertrain parts was very limited due to some reasons mainly associated with increased magnresium cost compared to aluminum and, to great extent, with lack of suitable HPDC magnesium alloys with properties that match closely those of HPDC aluminum alloys like A380. The present paper addresses high-pressure die cast magnesium alloys MRI153M and MRI230D that combine numerious advantages of standard magnesium alloys over aluminum alloys with enhanced general corrosion resistance and creep performance at competitive cost. Specific benefits and weight reduction associated with the use of MRI alloys in different applications are discussed and disclosed.

3:00 PM
Microstructrual Evolution and Creep Resistance in Mg-Sn-Ca Alloy: *Do Hyung Kim*[1]; Hyun Kyu Lim[1]; Ju Youn Lee[1]; Won Tae Kim[1]; Do Hyang Kim[1]; [1]Center for Noncrystalline Materials

Among the trace elements to improve creep resistance in magnesium alloys, Sn and Ca have attracted many researcher's attention due to the formation of thermally stable phases such as Mg_2Sn and Mg_2Ca. In the present study, creep properties of Mg-Sn-Ca alloys depending on the microstructrual evolution have been investigated. As-cast microstructures of the Mg-5Sn-0~4Ca (wt%) alloys exhibit typical dendritic structure consisted of primary α-Mg and secondary solidification phases. The secondary solidification phases have been identified as Mg_2Sn, CaMgSn or Mg_2Ca depending on the Ca/Sn ratio. Although the CaMgSn phase shows high thermal stability, its coarse morphology deteriorates the creep resistance, acting as crack initiation sites. On the other hand, the alloy containing the Mg_2Ca phase (higher Ca/Sn ratio) shows remarkably enhanced creep resistance. The detailed analysis indicates that improved creep resistance is originated from the precipitation of the Mg_2Ca phase preserving coherency with α-Mg matrix.

3:20 PM Break

3:40 PM
Influence of Pressure Levels on Tensile Properties of Squeeze Cast Mg-Al-Sr Alloy: *Shuping Wang*[1]; Henry Hu[1]; [1]University of Windsor

The focus of this study is on development of alternative manufacturing processes for potential high temperature magnesium-aluminium-strontium alloys. The effect of external pressure on tensile properties of squeeze cast Mg-Al-Sr alloy was investigated. Four different applied levels, 0, 30, 60 and 90 MPa, were employed to exert on a Mg-6 wt.% Al – 0.5 wt.% Sr alloy during squeeze casting. The results of tensile testing indicate that the ultimate tensile strength (UTS), yield strength (YS) and elongation (Ef) of the squeeze cast Mg-Al-Sr alloy increase with increasing applied pressure level. The microstructural analysis and porosity measurements suggest that the tensile property enhancement resulting from applied pressure should be attributed to microstructure refinement and porosity reduction of the squeeze cast alloy.

4:00 PM
The Mg-Al-Zn-Mn-Ca-Sr Alloy System: Backbone of Understanding Phase Formation in AXJ Alloys and Modifications of AZ and AM Alloys with Ca or Sr: Andreas Janz[1]; Joachim Groebner[1]; *Rainer Schmid-Fetzer*[1]; [1]Clausthal University of Technology

Progress is reported on experimental study and thermodynamic modeling of phase formation in key subsystems pertinent to the Mg-Al-Zn-Mn-Ca-Sr alloy system, or abbreviated as (Mg)-AZMXJ. This ongoing work enables thermochemical calculations, which are an important tool for the focused alloy development and process optimization. As an example, additions of Ca and Sr to alloys of the AZ series may provide improved mechanical and corrosion properties. Understanding the contribution of phase formation to these effects and, based on that, tackling a purposeful optimization is hardly possible without that information. This applies also in the case of Sr-addition to AM, essentially forming AJ alloys or, including the Ca-addition, to the potential offered by the AXJ system. This work is supported by the German Research Foundation (DFG) in the Priority Programme "DFG-SPP 1168: InnoMagTec."

4:20 PM
Development of Creep-Resistant Magnesium Die Casting Alloy RSM-9#: Weijian Tao[1]; Shu Wang[1]; *Fang Yu*[1]; [1]Nanjing Welbow Metals Company, Ltd.

This paper presents a creep-resistant magnesium alloy (RSM-9#) with high performance and low cost. The effects of alloying elements Al, Ca, Sr and Ce on the mechanical properties at both room and high temperature have been studied by using die cast specimens. It was found that this alloy (RSM-9#) has a steady state creep rate of 3.43×10-10/s at 175°C and 70MPa. Microstructure Examination, SEM and EDAX show that the integration of reduced ß-Mg17Al12 phase and appearance of high melting point inter-metallic phases with Ca and Ce improved the high temperature mechanical properties of RSM-9# alloy.

4:40 PM
A Comparative Study of the Microstructures and Creep Behavior of AE42 and AE44 Die-Casting Alloys: Suming Zhu[1]; Mark Gibson[2]; *Jian-Feng Nie*[1]; Mark Easton[1]; Trevor Abbott[3]; Per Bakke[4]; [1]CAST, Monash University; [2]CAST, CSIRO Manufacturing and Materials Technology; [3]Advanced Magnesium Technologies; [4]Hydro Magnesium Competence Centre

It has been reported that the creep performance of AE42 tends to deteriorate at temperatures above 150°C while AE44 exhibits much better creep performance at elevated temperatures. However, the relationship between microstructures and creep resistance in these Mg-Al-RE die-casting alloys has not been well understood. In the present work, the microstructures and creep behaviour of AE42 and AE44 die-casting alloys were investigated and compared. The creep tests were conducted at 175°C in a stress range of 75 - 90 MPa. The microstructures in the as-cast and the aged conditions were characterised in detail by transmission electron microscopy. The correlation between creep resistance and microstructure suggests that it is the level of Al solute retained in the α-Mg matrix after die-casting that is the key factor in influencing creep resistance, with high levels leading to the precipitation of $Mg_{17}Al_{12}$ during creep and thus reducing the creep resistance.

Materials in Clean Power Systems III: Fuel Cells, Hydrogen-, and Clean Coal-Based Technologies: PEM Fuel Cells and Solar Technologies

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee
Program Organizers: Zhenguo "Gary" Yang, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; K. Scott Weil, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Ayyakkannu Manivannan, National Energy Technology Laboratory

Wednesday PM Room: 392
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Jin Yong Kim, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University

2:00 PM Invited
High Temperature Polymer Electrolyte Membrane Fuel Cells: *Dominic Gervasio*[1]; [1]Arizona State University

A High Temperature Proton Electrolyte Membrane Fuel Cell (HT PEM FC) has an ion conducting membrane that operates with little or no need for hydration at temperatures above 100°C. Plastic PEMs are shock and vibration tolerant and operating above the water boiling temperature gives simplified one-phase fluidics, heat rejection and tolerance to impurities for a more robust, compact and efficient system. Unfortunately, the state-of-the-art HT PEM FC, which uses a phosphoric acid loaded polybenzimidazole membrane, operates at 0.6 volt and 200 milliamp per square centimeter at ambinent pressure and 170°C, so the cell is only 50% efficent and has low areal power density. New membranes based on protic salt electrolytes and thin lightweight corrosion-resistant metal-foil cell housings are under study at Arizona State University and recent results in these two efforts for making improved fuel cells will be discussed.

2:30 PM
Alloy Effects and Processing of Nitrided Metallic Bipolar Plates for Proton Exchange Membrane Fuel Cells: Michael Brady[1]; *Peter Tortorelli*[1]; Harry Meyer[1]; Heli Wang[2]; John Vitek[1]; John Turner[2]; Dan Connors[3]; Mahlon Wilson[4]; Fernando Garzon[4]; Josh Pihl[1]; Don Gervasio[5]; James Rakowski[6]; [1]Oak Ridge National Laboratory; [2]National Renewable Energy Laboratory; [3]GenCell Corporation; [4]Los Alamos National Laboratory; [5]Arizona State; [6]Allegheny Ludlum

Formation of protective Cr-nitride surface layers on stainless steel proton exchange membrane fuel cell (PEMFC) bipolar plates by gas nitridation necessitates decreasing the high nitrogen permeability in these materials. This is accomplished by preoxidation of the material to form a Cr-rich oxide surface followed by thermal conversion of this surface oxide to Cr-nitride by nitridation. Small additions of vanadium to the alloy improve the behavior by modifying the Cr-oxide to make it more amenable to nitridation. A multi-organization effort to scale up and evaluate this approach with thin stamped and nitrided V-modified Fe-Cr base stainless steel bipolar plates was recently initiated. Initial results related to alloy design and processing efforts to lower the levels of Cr and V needed to yield the protective Cr-nitride surface, in order to reduce alloy cost and improve amenability to stamping, will be presented.

2:55 PM
Metallurgical and Corrosion Evaluation of 316L Stainless Steel for PEM Fuel Cell Bipolar Plate Application: *Daniel Wilkosz*[1]; Mark Ricketts[1]; Tsung-Yu Pan[1]; Michael Santella[2]; Alan Frederick[2]; [1]Ford Motor Company; [2]Oak Ridge National Laboratory

The austenitic stainless steel 316L is a candidate material for PEM fuel cell bipolar plates. The process to make a bipolar plate involves stamping, trimming, and laser welding. The effect of laser welding on the microstructure and corrosion of 316L was investigated in this study. Full-penetration linear weld beads were produced in 0.1-mm-thick samples using a 400W pulsed YAG laser. Weld translation speeds were 8.5-12.7 mm/s. Corrosion and metallurgical evaluations were conducted between weld-bead and original samples. The welded materials were corrosion tested using electrochemical polarization measured between -0.5Vnhe and 1.5Vnhe at a scan rate of 5mV/s. Static polarization at 1Vnhe for 6hour duration were also undertaken. The current response was measured and the test solution was analyzed to determine the quantity of metal ion leached. All electrochemical tests were carried out in pH 3 sulfuric acid solution. Metallurgical specimens were prepared by cross-sectioning and examined by light microscope.

3:20 PM Break

3:30 PM Invited
Tantalum Oxide for PEM Electrode Applications: *Jin Kim*[1]; Jeff Bonnett[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

Over the past decades, a significant amount of research effort has been made to reduce the cost of PEM fuel cell stacks by minimizing or eliminating the use of platinum in the electrodes. Even though several alternative materials have been attempted to replace expensive Pt-based catalysts, none of them show satisfactory properties in term catalytic activity and stability. Our preliminary research exhibits that tantalum oxide has high oxygen reduction potential comparable to Pt even though the reduction current is by and large limited by its poor electrical conductivity. One of the approaches to improve the reduction current of tantalum oxide is to increase its triple-phase boundaries where the oxygen reduction occurs. In this study, the feasibility of sputtered tantalum oxide for PEM catalysts has been investigated. The detailed results to date will be discussed.

4:00 PM
Characterization and Stability of Carbon Supported Pt and PtCo Alloy Fuel Cell Catalysts: *Obiefune Ezekoye*[1]; Karen Adams[2]; Chi Paik[2]; George Graham[1]; Xiaoqing Pan[1]; [1]University of Michigan; [2]Ford Motor Company

Carbon supported PtCo is a promising catalyst material that has the potential to mitigate some of the performance and durability issues associated with pure Pt proton-exchange membrane (PEM) fuel cells catalysts. In this work, the electrocatalytic activity and durability of fuel cell electrodes containing carbon supported Pt and PtCo catalysts were investigated using voltage cycling methods for 450 minutes in 1M H_2SO_4. After voltage cycling, powders removed from these electrodes were investigated with high resolution transmission electron microscopy (HRTEM) and compared with untreated commercial catalyst powder. HRTEM results support conclusions, reported elsewhere, that cobalt alloying significantly reduces platinum particle size growth after voltage cycling. In this study, however, HRTEM was used to quantify the effects of alloying on both the mean particle size and the particle size distribution in carbon supported Pt and PtCo fuel cell catalyst.

4:25 PM
Photoelectrochemical Investigation of InN Thin Film Electrodes: Sushil Sitaula[1]; *Aurangzeb Khan*[1]; Mohammad Alam[1]; [1]University of South Alabama

The photoelectrochemical (PEC) method of renewable hydrogen production is a promising alternative to generate environment friendly energy to alleviate the current energy crisis. It produces clean energy with only sunlight as an energy source and only water as a by-product. Finding a suitable material for the harvesting of sunlight to break water has been a major challenge. This work reports photoresponse of indium nitride thin film electrodes prepared by pulsed laser deposition. X-ray diffraction and scanning electron microscopy were used for the structural characterization of the films. Indium nitride electrodes showed n-type behavior. Maximum photocurrent of several μA was observed. The effect of growth parameters on the photoresponse of the films was studied with the intent of optimization. No clear trend between the photoresponse of the films and the growth parameters was observed. At 175 mJ of pulse energy and 5000 laser pulses, optimum photocurrent was observed.

4:50 PM
Growth of InN on Silicon and Sapphire Substrates Using Direct Pulsed Laser Deposition: Sushil Sitaula[1]; *Aurangzeb Khan*[1]; Mohammad Alam[1]; Albert Gapud[2]; [1]University of South Alabama, Department of Electrical and Computer Engineering; [2]University of South Alabama, Department of Physics

InN is a novel material, and many of its physical and optical properties are yet to be established. Recently, it has received a significant attention in opto-electronic applications as well as in photoelectrochemical hydrogen production. Due to lower dissociation temperature of around 550°C, high quality growth of InN is very difficult. This paper reports successful deposition of InN films on silicon and sapphire substrates using direct pulsed laser deposition. Laser pulse energy and the growth temperature were found to be important growth parameters. Higher pulse energy led to splashing of the molten target, forming a rough surface morphology. As observed from scanning electron microscope, optimum surface morphology was obtained with 175 mJ of pulse energy. At 525°C, the best surface morphology was obtained. The X-ray diffraction pattern of the films reveals the growth of polycrystalline InN, with (002) and (111) as major peaks.

5:15 PM
Study on the Preparation of Dendritic Zinc Powder for Zinc-Air Battery by Electrodeposition: *Qing-hua Tian*[1]; Xueyi Guo[1]; [1]Central South University

Dendritic zinc powder is applied in preparation of cathode of zinc–air battery. Based on a series of experiments, the electrodeposition was determined for the preparation of dentritic zinc powder in the solution system of Zn2+-NH4+-SO42—H2O. The thermodynamic equilibrium analysis was conducted for this system, and the thermodynamics curves were drawn. Then the effects of current density temperature pH of solution concentration of reactants were explored by experiments, the pure and well crystalized zinc was obtained. Based on the test results the optimal conditions were as follows:[Zn2+] is 15g/L, [(NH4)2SO4] is 30~40g/L, pH of electrolyte is 4.5~5.45, and temperature of it is 25°C. SEM Photos of the obtained powder shows that the particles are dentritic in narrow size distribution.

Materials Informatics: Enabling Integration of Modeling and Experiments in Materials Science: Informatics and Cyberinfrastructure

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizer: Krishna Rajan, Iowa State University

Wednesday PM Room: 271
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: To Be Announced

2:00 PM
CyberDesign: *Mark Horstemeyer*[1]; Tomasz Haupt[1]; [1]Mississippi State University

This presentation discusses the concept of the CyberDesign, that is, the application of emerging IT technologies to exploit the recent transformative research in material science involving multiscale physics based predictive modeling, multiscale experiments and bio-inspired design, in conjunction with robust design and manufacturing process optimizations with uncertainty. The cyberinfrastructure will enable the integration of otherwise independent research areas to accelerate agglomeration of structure-properties relations into material models. The results of such agglomeration will lead to higher fidelity, validated designs of structural components and predictive material models that can capture structure-properties relations and include material history, thus closing the significant knowledge gaps in a unifying physics based modeling theme that captures a broad range of materials, admits structure-property relations, can be used in simulation-based design, can be implementable into finite elements codes, can be experimentally validated, can capture history effects, and can morph into a metamodel for rapid use prognostics.

2:20 PM
Cyberinfrastructure for Design Optimizations: *Tomasz Haupt*[1]; Mark Horstemeyer[1]; [1]Mississippi State University

Simulation-based design optimization is playing an increasingly important role in engineering product development, reducing or eliminating the need for physical prototyping, and increasing the quality of manufactured products. The complexity of the new generation of simulation codes demands the employment of high-performance computing platforms. Currently, it is a very tedious and error-prone manual effort by the designer to submit, monitor and coordinate hundreds of jobs in heterogeneous distributed environments which requires the designer to learn the arcana of ever-changing IT technologies such as operating systems, batch systems, storage systems, networking, and security. This presentation demonstrates the use of the modern information infrastructure based on Service Oriented Architecture (SOA), Web Services and Grid computing streamlining of the process of gathering experimental results, and deriving the material properties for a particular material model and employing the material model in finite element analysis in the process of building validated metamodels and design optimizations.

2:40 PM
Data/Modeling-Driven toward Innovation of Materials Design: *Ying Chen*[1]; Hao Wang[2]; Yasunori Kaneta[1]; Shuichi Iwata[2]; [1]University of Tokyo, School of Engineering; [2]University of Tokyo, Graduate School of Frontier Sciences

One of the most challenging tasks in material science is designing materials with certain atomic constitutions, which achieves the pre-defined properties. As a typical inverse problem, it is very hard to find the best solution from a large amount of possibilities. There are two typical approaches in materials design. One is the inductive approach based on the empirical regularities from analyzing a materials data collected, another is the deductive approach based on theoretical calculations on specific systems following general physical laws. These two approaches so far have been developed almost independently. In recent years, we proposed an integrating approach by combining two types of approach, the data mining and the first principles calculations, as an efficient way to accelerate

the speed of finding target atomic configuration from huge number of candidates and predicting properties of new materials. Several examples will be presented.

3:00 PM

Incorporating Collaborative Tools in MatDL Pathway: *Laura Bartolo*[1]; Cathy Lowe[1]; [1]Kent State University

The NSF supported National Science Digital Library Materials Digital Library Pathway (MatDL) is implementing an information infrastructure to disseminate government funded materials research results and to provide content as well as services to support the integration of research and education in materials. This paper describes how we are incorporating open-source collaborative tools, such as wikis and collaborative source code development systems, into the MatDL Pathway to support users in materials research and education as well as interactions between the two. These tools also have the potential to facilitate interactions between theorists and experimentalists.

Mechanical Behavior, Microstructure, and Modeling of Ti and Its Alloys: Physical/Mechanical Property Prediction

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Titanium Committee
Program Organizers: Ellen Cerreta, Los Alamos National Laboratory; Vasisht Venkatesh, TIMET; Daniel Evans, US Air Force

Wednesday PM Room: 384
March 12, 2008 Location: Ernest Morial Convention Center

Session Chair: Kuang-Tsan Chiang, Southwest Research Institute

2:00 PM Invited

Modeling of Phase Transformation in Titanium Alloys: *Amit Chatterjee*[1]; David Furrer[1]; Michael Glavicic[2]; Jonathan Miller[2]; S. Semiatin[2]; [1]Rolls-Royce Corporation; [2]Air Force Research Laboratory

The evolution of microstructure in commercial titanium alloys is critical to the optimal development of final component mechanical properties. The growth of primary alpha upon cooling from solution heat treatment and subsequent decomposition of remaining beta grains in alpha-beta alloys, such as Ti64 and Ti6242 has been studied and applied to production process development and optimization. Similarly, the prediction of the decomposition microstructure within beta processed alloys, such as Ti6246, is critical to understanding the resultant mechanical properties. The decomposition of beta phase follows the Burger's relationship between the BCC and HCP phases making 12 total possible alpha orientation variants possible. Current studies have shown the potential of preferentially selecting specific variant orientations resulting in the potential of a precipitation texture. Results from initial efforts to identify the mechanisms and a modeling approach to predict anisotropic variant selection will be presented. Deformation texture is also prevalent within the processing of alpha-beta titanium alloys. Beta phase deformation texture can occur during high strain processing and alpha phase texture can also rapidly occur during deformation processing as a result of rigid-body rotation and crystallographic slip. A modeling tool to predict the texture evolution during deformation processing has developed and demonstrated. An overall assessment of how these types of microstructure modeling tools can be linked with and can support component and process design processes will also be discussed.

2:30 PM

The Application of Bayesian Neural Network Modeling for the Prediction of Fracture Toughness in α/β Ti Alloys: *Santhosh Koduri*[1]; Vikas Dixit[1]; Peter Collins[1]; Hamish Fraser[1]; [1]Ohio State University

The development of computational tools that permit microstructurally-based predictions of tensile and fracture toughness properties of Ti-based alloys is an essential step towards the accelerated maturation of materials. This paper will discuss the development of Bayesian Neural Network Models to predict the fracture toughness of Ti-6Al-4V at room temperature. The development of these rules-based models has required the population of extensive databases, including both compositional and microstructural information. Once trained and tested, these models have been successfully used to identify the influence of individual microstructural features on the mechanical properties through the use of virtual experiments. For example, they have been used to identify the importance of the equiaxed α size/fraction and the thickness of grain boundary α on fracture toughness for α+β and β-processed Ti-6-4, respectively. Such critical features have been further investigated using state-of-the-art characterization techniques (including 3D techniques) to determine their role in fracture toughness.

2:50 PM

Microstructural Evolution and Mechanical Property Prediction in Beta Ti Alloys: Soumya Nag[1]; Arda Genc[1]; Peter Collins[1]; Gopal Viswanathan[1]; Srini Rajagopalan[1]; Rajarshi Banerjee[2]; *Hamish Fraser*[1]; [1]Ohio State University; [2]University of North Texas

This paper describes research aimed at understanding the evolution of microstructure in beta-Ti alloys as a function of heat-treatment. Of particular interest are the nucleation mechanisms that influence the distribution of alpha Ti from the beta phase. Here, attention is paid to the influence of beta phase separation and the omega phase on process of nucleation. The differences between the precipitation processes in alpha/beta Ti alloys and those observed in beta Ti alloys will be discussed. The understanding gained is used to predict ways to optimize microstructure for given combinations of properties. The prediction of mechanical properties, through microstructure/property relationships, is effected through data mining techniques. Specifically, neural networks have been developed to relate microstructure to mechanical properties, and these will be demonstrated and discussed. Additionally, these models are used to develop an understanding of functional dependencies of properties on specific microstructural features. These will be presented and discussed.

3:10 PM

Anisotropic Elastic/Plastic Model for Description of High Purity Alpha Titanium: *M.E. Nixon*[1]; Oana Cazacu[2]; R.A. Lebensohn[3]; [1]Air Force Research Laboratory and University of Florida; [2]University of Florida/REEF; [3]Los Alamos National Laboratory

Accurate modeling of anisotropic hexagonal closed packed (hcp) polycrystals such alpha-titanium requires the description of the interplay between slip and twinning and its effects on texture evolution and hardening response. In this paper, an anisotropic model that captures the influence of evolving texture on the plastic response of hcp metals is proposed. Yielding is described using a new criterion which captures simultaneously anisotropy and compression-tension asymmetry associated with deformation twinning. The anisotropy coefficients as well as the size of the elastic domain are evolving with the plastic strain. Application of the model to the simulation of the three-dimensional deformation of high-purity alpha-titanium beams subjected to four-point bend tests along different directions is presented. Comparison between predicted and measured macroscopic strain fields and beam sections shows that the proposed model describes very well twinning and its role on evolving the material anisotropy.

3:30 PM Break

3:50 PM

A Constitutive Model of Cavitation-Induced Damage in Ti Alloys: *Shyam Keralavarma*[1]; Ahmed Benzerga[1]; [1]Texas A&M University

One major challenge in the hot working of Ti-based alloys is minimizing cavitation induced damage such as flow localization in shear bands that limit the formability of such metals. There is a need for accurate modeling of microstructure evolution under various loading conditions to ensure that such defects are minimized in the final product. We present a rigorous micromechanics based constitutive model of porous solids incorporating the effects of void shape and plastic anisotropy of the matrix. An important feature of the model is that it predicts the influence of plastic anisotropy and cavity shape on the evolution of porosity, which can potentially yield more accurate modeling of cavitation induced damage during material processing and cavity sealing upon strain-path changes. The model predictions for the yield surface and microstructure evolution for representative materials are compared with numerical limit analysis and finite element simulation results on porous unit cells for validation.

4:10 PM
Dislocation Characterization and Modeling of Creep Mechanisms in Zirconium Alloys: *Benjamin Morrow*[1]; Robert Kozar[2]; Ken Anderson[2]; Michael Mills[1]; [1]Ohio State University; [2]Bechtel Bettis, Inc.

Zirconium alloys are important as a material in nuclear reactors. Accurately predicting creep deformation of zirconium alloys throughout the lifecycle of a reactor depends on reliable deformation models. The Modified Jogged-Screw Model asserts that the motion of tall jogs in screw dislocations act as the rate controlling mechanism during creep in certain regimes. Previous studies at The Ohio State University have shown that the Modified Jogged-Screw Model accurately describes creep behavior in titanium alloys, which have a similar (HCP) crystal structure to zirconium alloys. Scanning transmission electron microscopy (STEM) was used to directly observe and characterize the dislocation structure of creep tested Zircaloy-4 over a broad range of test conditions. The applicability of the Modified Jogged-Screw Model was demonstrated and physically-based values for model parameters were determined. Thorough characterization will provide a better understanding of thermal creep in zirconium alloys, which will ultimately result in more robust creep deformation predictions.

4:30 PM
Micromechanical Modeling of Deformation Behavior of Duplex Type TNB Alloy with a Multi-Scale Modeling Approach: *Mohammad Rizviul Kabir*[1]; Liudmila Chernova[1]; Marion Bartsch[1]; [1]German Aerospace Center

Room temperature deformation behavior of niobium-containing two-phase TiAl alloy has been investigated numerically using a two-scale micromechanical FE model. A polycrystal with duplex microstructure consisting of globular and lamellar grains is modeled with 3D unit cells that take into account specific volume fractions and orientations of the grains. The constitutive behavior of the α_2 and γ-phases is described by continuum mechanics based crystal plasticity model. Quantitative representations of microstructural details are obtained from the SEM analysis of a particular TNB alloy. The material parameters of the α_2 and γ-phases are initially taken from literatures and modified to fit the tensile behavior of the TNB alloy. The model provides an insight into the local variation of stresses and strains of the duplex microstructure consisting of globular and lamellar colonies. The role of dislocations and slip systems on the evolution of local plastic deformation is discussed.

4:50 PM
High Strain-Rate Modeling of Titanium-Aluminum Laminates: *Charles Randow*[1]; George Gazonas[2]; [1]Bucknell University; [2]U.S. Army Research Laboratory

Laminate composites created by alternating layers of titanium and aluminum have shown an improved resistance to fracture with a reduction in density. In these studies, hot pressing has been used to bond the layers together, resulting in the creation of the intermetallic $TiAl_3$. In the present work, commercially pure titanium (CP-Ti) and 1100-Al are bonded using an ultrasonic consolidation process before hot pressing to form a CP-Ti/$TiAl_3$/Al laminate, with the goal of developing a system capable of withstanding the high strain-rate loads encountered in ballistic impact and blast applications. One of the difficulties in modeling such systems is the lack of information available on the high strain-rate properties of $TiAl_3$. Through the use of dynamic finite element modeling and plate impact experiments, the effect of $TiAl_3$ on the laminate strength is investigated. These results are used in predictive models of the laminate subjected to impact loading.

5:10 PM
Modeling High Temperature Strength and Flow Stress Curves of Titanium Alloys: Zhanli Guo[1]; Nigel Saunders[1]; Peter Miodownik[1]; *Jean-Philippe Schille*[1]; [1]Sente Software, Ltd.

Process design and simulation for any metals requires an understanding of their high temperature mechanical behavior, particularly with respect to flow stress as a function of temperature and strain rate. This paper describes the recent developments of JMatPro, a computer software for material property simulation, on calculating the high temperature mechanical properties of titanium alloys. Extensive validation has been carried out and shown in the present work. Good agreement between calculated and experimental results has been achieved for a variety of titanium alloys, including alpha, near-alpha, alpha+beta, and beta types, over a wide range of temperature and strain rates. The material properties calculated from JMatPro can now be passed directly into computer-aided engineering packages for process simulation.

Neutron and X-Ray Studies for Probing Materials Behavior: Scattering and Understanding of Materials Properties

Sponsored by: National Science Foundation, The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter K. Liaw, University of Tennessee

Wednesday PM Room: 391
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Emil Zolotoyabko, Technion-Israel Institute of Technology; Gene Ice, Oak Ridge National Laboratory

2:00 PM Invited
Small-Angle Scattering of Neutrons and X-Rays in Materials Science - A Comparison: *Gernot Kostorz*[1]; [1]ETH Zurich

After a short comparison of the basic features of small-angle scattering of X-rays and neutrons, examples of recent applications will be given. Experimental rsults will be discussed, in particular those concerning phase separation of alloys, emphasizing the possibilities of spatial and temporal resolution of the decomposition process, high temperature-in-situ studies, and combinations with diffuse scattering and transmission electron microscopy. In some Al-, Cu- and Ni-base alloys, shape, volume fraction, spatial arrangement and other details of precipitates can be analyzed with great precision.

2:25 PM
Understanding Particle Morphology during High Energy Milling of Complex Metal Hydrides: *Tabbetha Dobbins*[1]; Ejiroghene Oteri[1]; Jan Ilavsky[2]; [1]Louisiana Tech University; [2]Advanced Photon Source, Argonne National Laboratory

A program comprised of ultrasmall-angle x-ray scattering (USAXS) experiments was performed at sector 33-ID at the UNI-CAT for the study of particle morphology changes in the alanates (e.g. $NaAlH_4$—both before and after transition metal salt catalytic dopant additions by high energy ball milling). The variation in surface fractal dimension and particle size with milling time and dopant content was tracked. These studies have shown that dopant content level (e.g. 2 mol % and 4 mol %) and dopant type (i.e. $TiCl_2$, $TiCl_3$, VCl_3, and $ZrCl_4$) markedly affects $NaAlH_4$ powder particle surface area (determined using surface fractal dimension). This study was able to link powder particle surface area to catalytic doping. We will report links among codopants (i.e. two dopants) and powder particle surface area studied using USAXS.

2:45 PM Invited
Diffuse Scattering and Monte Carlo Studies of Relaxor Ferroelectrics: *Richard Welberry*[1]; [1]Australian National University

A renewed interest in the field of ferroelectricity has taken place in recent years since the finding of exceptional piezoelectric properties in the lead-oxide class of relaxor ferroelectric (RF) materials typified by the disordered perovskite $PbZn_{1/3}Nb_{2/3}O_3$ (PZN).Although PZN and numerous related materials have been extensively studied over a long period a detailed understanding of the exact nature of their polar nanostructure has still not emerged. In this paper we describe experiments in which full three-dimensional diffuse neutron scattering data have been recorded from a single crystal of PZN using the time-of-flight (tof) Laue technique. Monte Carlo simulation has been used to demonstrate that the observed diffuse patterns are due to planar nano-domains oriented normal to the six <110> directions. A simple model has been developed which explains the observed scattering.

3:10 PM Invited
Concomitant Determination of Planar Faults, Dislocations and Subgrain Size by X-Ray Line Profile Analysis: *Tamas Ungar*[1]; Levente Balogh[1]; [1]Eotvos University

X-ray line profile analysis is based on (i) the different profile shapes and (ii) the different hkl dependence of line broadening or shifts corresponding to the different types of lattice defects: (a) grain or subgrain boundaries, (b) dislocations and (c) planar defects, i.e. stacking faults and twin boundaries. It is shown that physically well established, theoretically calculated profile functions can be attributed to the three fundamental defect types, (a) to (c). These *defect-related profile-functions* have the same mathematical form for each Bragg reflection throughout the entire diffraction pattern. However, their breadths and positions are scaled by defect specific, hkl dependent scaling rules. Since the scaling rules are only weakly correlated the diffraction effects of the three different defect types can be separated and the defect properties well characterized.

3:35 PM Break

3:45 PM Invited
Probing Phonons and Phase Transitions in Solids with X-Ray Thermal Diffuse Scattering: *Tai Chiang*[1]; [1]University of Illinois

X-ray scattering by thermally populated phonons in crystals give rise to diffuse scattering. Measurements of such thermal diffuse scattering (TDS) intensities as functions of momentum transfer and sample temperature yield information about the lattice dynamics including the phonon dispersion relations. This method has a long history, and after being largely ignored for decades, it has revived and become a powerful tool with the advent of intense synchrotron x-ray sources. In this talk, we review the basics of TDS together with recent experiments of phonon studies in various systems. Issues of high order scattering and background contributions will be discussed in connection with details of data analysis. Examples will be presented to illustrate the power of TDS as a probe of phase transitions that involve phonons. The method is well suited for detailed studies of soft mode behavior and critical exponents.

4:10 PM Invited
Local Structure of Decagonal Quasicrystals by DAFS: *Hiroshi Abe*[1]; Hiroyuki Saitoh[2]; Hironori Nakao[3]; [1]National Defense Academy; [2]Japan Atomic Energy Research Institute; [3]Tohoku University

Al-Ni-Co (ANC) and Al-Ni-Fe (ANF) system is well known to be decagonal quasicrystals, which have two-dimensional quasiperiodic planes. The structures depend both on concentrations and on temperature extensively. Recently, short-range order (SRO) in quasicrystals was detected on a quasiperiodic plane by anomalous-X-ray scattering method.[1] In this study, local structure of quasicrystals was examined along the periodic direction compared with a quasiperiodic plane by X-ray diffraction anomalous fine structure (DAFS) technique. In principle, DAFS possesses site and spatial selectivity. DAFS experiment was performed on the beamline BL-4C of the Photon Factory at the High Energy Accelerator Research Organization in Japan. Apparent differences of DAFS between periodic and quasiperiodic directions were shown in ANC and ANF. [1]H. Abe, H. Saitoh, T. Ueno, H. Nakao, Y. Matsuo, K. Ohshima and H. Matsumoto, J. Phys.: Condens. Matter 15 (2003) 1665.

4:35 PM Invited
Diffuse Scattering in Quasicrystals of Icosahedral Symmetry: *Sonia Francoual*[1]; Marc de Boissieu[2]; Roland Currat[3]; Shiro Kashimoto[4]; Tsutomu Ishimasa[4]; [1]National High Magnetic Field Laboratory; [2]Sciences et Ingenierie des Materiaux et Procedes; [3]Institut Laue-Langevin; [4]Hokkaido University

According to the hydrodynamic theory of quasicrystalline materials, the long-range quasiperiodic order yields in quasicrystals new long-wavelength modes, the phason modes, in addition to the acoustic phonons. Phason excitations are diffusive and associated with internal atomic rearrangements the dynamics of which is expected to lead to a distinctive diffuse scattering signal around Bragg peaks in reciprocal space. Up to now, experimental demonstration for the existence of phason diffuse scattering was given in the enlightening single case of the Al-Pd-Mn Mackay-type icosahedral (i-) phase from which the phason elastic constants were extracted and the thermal activation revealed. In the present communication, we report on X-ray Synchrotron investigations of the diffuse scattering in the i-Zn-X-Sc (X = Mg, Ag, Co) phases and the Zn-Sc 1/1 periodic approximant bringing evidence for the presence of long-wavelength phasons also in this new class of icosahedral quasicrystals and ruling it out in the 1/1 approximant structure.

5:00 PM Invited
Towards Systematic Procedures for Interpreting Diffuse Scattering from Disordered Molecular Materials: Hans-Beat Buergi[1]; *Christina Hoffmann*[2]; [1]University of Zuerich; [2]Oak Ridge National Laboratory

Real crystals show many kinds of disorder giving rise to diffuse scattering which may cover the whole reciprocal space and is generally much weaker than Bragg scattering. However, with the advent of new, more intense radiation sources like synchrotrons and neutron Spallation sources it has become possible to measure diffuse features accurately. New X-ray and neutron single crystal diffractometers have been designed for fast data collection and repeatability of experiments at controlled conditions. X-ray and neutron experiments will be measured on the same sample by both methods, allowing to analyze the collected data simultaneously. Although state of the art computing resources allow to process the large amounts of data produced in such experiments, the methods to produce reliable models of disorder are less developed in comparison to single crystal structure analysis. Here we will sketch steps towards systematic procedures for interpreting diffuse scattering from molecular materials.

5:25 PM Invited
X-Ray Imaging for Materials and Biomedical Sciences: *Jung Ho Je*[1]; Y. Hwu[2]; G. Margaritondo[3]; [1]Pohang University of Science and Technology; [2]Academia Sinica, Institute of Physics; [3]Ecole Polytechnique Fédérale de Lausanne

Radiology is the oldest and by far the largest field of application of x-rays. In recent years, this domain has been literally revolutionized by the exploitation of the unique characteristics of synchrotron sources. The results are particularly spectacular when the high spatial coherence of the radiation is used for novel and powerful approaches to radiology. The results are very high quality microradiology and microtomograhy images and movies - taken with a limited x-ray dose – that find a variety of applications in materials science, biology and medical research. In this talk we review basic theory and selected applications of phase contrast x-ray microscopy to materials and biomedical sciences. Furthermore we introduce a new strategy of combining phase contrast radiology and diffraction x-ray microscopy to visualize atomic level defects such as misfit dislocations and micropipes in semiconductor single crystals. Finally phase contrast x-ray imaging in nanometer-resolution (<30 nm) will be demonstrated.

5:50 PM
Panel Discussion

Donald Nicholson, Oak Ridge National Laboratory

Wolfgang Pantleon, Risø National Laboratory

Gernot Kostorz, ETH Zurich

Emil Zolotoyabko, Technion-Israel Institute of Technology

Vaclav Holy, Charles University

Richard Welberry, Australian National University

Henning Poulsen, Risø National Laboratory

Philip Withers, Manchester University

Donald Brown, Los Alamos National Laboratory

Sunil Sinha, University of California, San Diego

Ulrich Lienert, Argonne National Laboratory

Hiroshi Abe, National Defense Academy

Branton Campbell, Brigham Young University

Lyle Levine, National Institute of Standards and Technology

Tamas Ungar, Eotvos University

6:20 PM Concluding Comments

Particle Beam-Induced Radiation Effects in Materials: Nanostructures

Sponsored by: The Minerals, Metals and Materials Society, American Nuclear Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Gary Was, University of Michigan; Stuart Maloy, Los Alamos National Laboratory; Christina Trautmann, Gesellschaft fur Schwerionenforschung; Maximo Victoria, Paul Scherrer Institute and Lawrence Livermore National Laboratory

Wednesday PM — Room: 389
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Daryush Ila, Alabama A&M University; Tongde Shen, Los Alamos National Laboratory

2:00 PM Invited
Ion Beam Processing of Nanostructures: From keV to GeV in Ion Energy: *Lumin Wang*[1]; Q. Wei[2]; A. Perez-Bergquist[3]; X. Xiang[3]; W.X. Li[3]; Y.W. Zhang[4]; V. Skuratov[5]; [1]University of Michigan, Departments of Nuclear Engineering and Radiological Sciences and Materials Science and Engineering; [2]University of Michigan, Department of Materials Science and Engineering; [3]University of Michigan, Department of Nuclear Engineering and Radiological Sciences; [4]Pacific Northwest National Laboratory; [5]Joint Institute Nuclear Research, Center of Applied Physics, Flerov Laboratory of Nuclear Reactor

The unique nano-scale interaction volume between an energetic particle and the solid allowed one to use the particle beams to process some unique nanostructures. With low energy ions (<10 keV), while sputtering and redeposition are significant, patterned semiconductor nano-dot array have been obtained. With immediate energy (30 keV to 1 MeV) heavy ions, point defect production and migration have lead to nano-porous structures consists nano-pores and nano-fibers of uniform diameters (~10nm) in several semiconducting materials. Micro-sized patterns containing the nano-fibers have been fabricated on the surface of Ge and GaAs using focused ion beam. Embedded GaSb nanofibers has formed after 1 MeV Au+ irradiation, and the thickness of the surface crust covering the nano-fibers has been found to be ion fluence dependent. With 100 MeV to GeV heavy ions, while ionization is dominant along the ion trajectory, aligned nano-tubes have been fabricated in volatile element containing insulators, e.g., fluorapatite.

2:40 PM
Studies of the Ion Irradiation Effects in Bulk Nanocrystalline TiNi: *Askar Kilmametov*[1]; Adam Balogh[2]; Horst Hahn[3]; Ruslan Valiev[1]; [1]Ufa State Aviation Technological University; [2]Darmstadt University of Technology; [3]Forschungszentrum Karlsruhe GmbH

Nanocrystalline intermetallic alloys, which possess a long-range chemical ordering, considered being a good object to examine irradiation effects on the stability or degradation of crystal superlattice in the ordered state. TiNi alloy as one of the most important engineering materials due to its superior shape memory and mechanical properties was studied at present work. Bulk ordered nanocrystalline samples of altered grain size values (25 and 40 nm) were processed using the method of severe plastic deformation, namely high pressure torsion technique. Ordered nanocrystalline and coarse-grained samples were subjected to 1.5 MeV Ar ion irradiation at room temperature with damage doze up to 5.6 displacements per atom. Comparative analysis of long-range disordering and amorphisation kinetics revealed the enhanced irradiation resistance of nanocrystalline TiNi alloy. It was shown that at the equal damage doze nanostructured state is able to retain a long-range ordering meanwhile the coarse-grained counterpart was just essentially amorphised.

3:00 PM
Nanoscale Nano-Systems by MeV Ion Beam: *Daryush Ila*[1]; Robert Zimmerman[1]; [1]Alabama A&M University

MeV ion beam used to form Nanolayers of Nanocrystals of various materials within selected host materials by sequentially co-depositing host, selected species and depositing the host. The Nanocrystals are formed along the direction of MeV Ion beam passage due to the electronic ionization of the substrate. Example: a system consists of Nanolayers of Nanocrystals generating highly efficient thermoelectric generators (TEG). TEG produced by enhancing the electrical conductivity, enhancing the thermal insulation and increasing the Seebeck Coefficient. Some material systems, we had to dope the nanolayers by keV implantation of selected species before MeV bombardment. We measured thermal conductivity, electrical conductivity, and the Seebeck coefficient as a function of ion bombardment fluence. The induced nanoscale effects enhanced the thermoelectric properties of selected materials, which is increased figure of merits. We will discuses the mechanism behind such ion beam bombardment effects and the reason why thermoelectric properties may be changed.

3:20 PM
Modeling of Point Defect Cluster Evolution in the Framework of Mean Field Approximation: *Stanislav Golubov*[1]; Alexey Ovcharenko[2]; Roger Stoller[1]; Steven Zinkle[1]; [1]Oak Ridge National Laboratory; [2]All Russia Institute for Agricultural Microbiology

Nucleation, growth and coarsening of point defect clusters or secondary phase precipitates are generic processes in the study of defect accumulation in crystalline solids. These processes are responsible for numerous changes one observes in the physical and mechanical properties of materials during thermal treatment or under irradiation. If random spatial distribution of defect clusters is assumed, the evolution is described in terms of their size distribution, which obeys the kinetic equation describing growth and dissolution of the clusters due to different reactions with mobile defects. The kinetic equation in the form of a Master Equation or Fokker-Plank Equation has been treated by means of both analytical and numerical methods. In this work the numerical methods, which permit approximate solutions to be obtained for practically interesting cases are critically reviewed and analyzed. Application of different methods to the evolution of different types of clusters is presented.

3:40 PM Break

4:00 PM
Nanostructuring by Energetic Ions: *Devesh Avasthi*[1]; [1]Inter University Accelerator Centre

The ions in diffferent energy regims can (i) produce nano particles (ii) change the shape of particle or nano structure and (iii) alter the size and the size distribution of nano particles. In low energy regime, 1 keV Ar atom source producing a wide beam is used to synthesize metal particles in insulating matrix with definite advantages over other conventional methods. C nanowires are created in fullerene matrix by irradiation of fullerene film by 100 MeV Au ions. The nucleation and growth of Ag nano particles in insulating matrix by ion irradiation is investigated. The growth of Au nanoparticles in silica matrix is studied in the in-situ XRD experiment during ion irradiation. There have been major activities in nanostructuring by ions at IUAC Delhi, in recent past, which show the unique features of ion beam in different energy regime in creating or modifying the nanostructures in a controlled way.

4:20 PM
Nanostructure under Irradiation: Is Smaller Better?: *Tongde Shen*[1]; Shihai Feng[1]; Ming Tang[1]; James Valdez[1]; Yongqiang Wang[1]; Kurt Sickafus[1]; [1]Los Alamos National Laboratory

Previous experimental observations indicate that nanosized zirconia or silicon, when embedded in an amorphous silica matrix (thus forming two-phase nanocomposites), are more susceptible to radiation-induced amorphization when compared to their corresponding large-grained counterparts. We compare and contrast ion-induced radiation damage in a single-phase oxide $MgGa_2O_4$, with microstructures consisting of either large (10 μm) or nanosized (10 nm) grains. We demonstrate a substantial enhancement in amorphization resistance for nanocrystalline versus large-grained $MgGa_2O_4$. In particular, we observed an amorphization transformation in large-grained $MgGa_2O_4$ at a relatively low ion dose of about 10 displacements per atom (dpa), while we found nanocrystalline $MgGa_2O_4$ to be unaffected by ion irradiation to a dose nearly an order of magnitude higher (100 dpa). Our experimental results and thermodynamic analysis suggest that smaller can be better under certain conditions.

4:40 PM
Nanoscale Surface Modifications of Conductors and Insulators Induced by Slow, Highly Charged Ion Impact: *Rene Heller*[1]; Stefan Facsko[1]; [1]Forschungszentrum Dresden

In the interaction of highly charged ions (HCI) with solids a large amount of potential energy is released. Each ion carries thereby up to several tens of keV potential energy which is mainly deposited into a strongly localized area (~nm2) on the surface within a very short time (~fs). Due to the highly excited electronic system and the subsequent relaxation various structural as well as morphological changes are induced. We present scanning probe investigations (AFM/STM) of surface modification in conductors and insulators induced by slow, highly charged ions with different potential energies. In addition, a theoretical model for the interaction process of HCIs with alkali halides is proposed.

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VII: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee
Program Organizers: Sinn-wen Chen, National Tsing Hua University; Srinivas Chada, Medtronic; Chih Ming Chen, National Chung-Hsing University; Hans Flandorfer, University of Vienna; A. Lindsay Greer, University of Cambridge; Jae-Ho Lee, Hong Ik University; Kejun Zeng, Texas Instruments Inc; Katsuaki Suganuma, Osaka University

Wednesday PM — Room: 278
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Jae-Ho Lee, Hong Ik University; Hans Flandorfer, University of Vienna

2:00 PM Invited
The Intermetallic System Cu-Ni-Sn: *Hans Flandorfer*[1]; Clemens Schmetterer[1]; Usman Saeed[1]; Herbert Ipser[2]; [1]University of Vienna; [2]University of Wien

The intermetallic system Cu-Ni-Sn is highly important for lead-free soldering as it concerns the majority of solder/substrate interactions of hitherto used solders. Although first phase diagram data for this system have been published approx. 30 years ago literature information turned out to be still confusing. Contradictions concern mainly the ternary solubilities and the possible existence of ternary compounds. Experimental work is hampered by several phase transformations in the Cu-Sn and Ni-Sn binaries and difficulties to quench the corresponding high-temperature phases. We prepared approx. 100 samples and annealed them at 400, 500 and 600°C; a temperature of 200°C was only applied on samples with high Sn-contents. We investigated the samples performing XRD, metallography including EPMA and thermal analysis. Based on our recent results for the Ni-Sn binary system, we could provide more detailed information on the phase relations of Cu-Ni-Sn and clarify some of the contradictions.

2:20 PM
Thermodynamic Modeling of the Sn-Sb-Ag and Sn-Sb-Cu Systems: *Wojciech Gierlotka*[1]; Sinn-wen Chen[1]; An-ren Zi[1]; Po-yin Chen[1]; [1]National Tsing Hua University

Sn-Sb-Cu and Sn-Sb-Ag ternary systems are of interests for lead-free solder applications. Thermodynamic models of these two ternary systems are developed based on the CALPHAD method using the experimental information found in the literature as well as our own experimental data. There are 2 binary phases, 2 continuous solid solutions and three terminal phases in the Sn-Sb-Ag system, and there are 15 binary phases 1 ternary phase and three terminal phases in the Sn-Sb-Cu system. Sublattice models were adopted as a new description of intermetallic compounds and compositional homogeneities of promising solder materials were reproduced. Results of the calculation, such as thermodynamic properties of liquid and solid phases, isothermal and isopleths sections, and liquidus projections are compared with the experimental data. The calculated results are in good agreement with the experimental determinations.

2:35 PM
Liquidus Projection of the Sn-Zn-Cu Ternary System: *Yu-Chih Huang*[1]; Sinn-wen Chen[1]; Chin-yi Chou[1]; Wojciech Gierlotka[1]; [1]National Tsing Hua University

Liquidus projection of the Sn-Zn-Cu ternary system has been experimentally determined and thermodynamically calculated. Sn-Zn-Cu alloys of various compositions were prepared. These alloys were melted at high temperatures, removed from the furnace and solidified in air. The solidified samples were metallographically examined to determine the primary solidification phases. There is no ternary compound and all the primary solidification phases are the terminal solid solutions and binary intermetallics. They are Sn, η-Cu6Sn5,ε -Cu3Sn, γ-(Cu-Sn), β-Cu17Sn3, α-Cu, β-CuZn, γ -Cu5Zn8, δ-CuZn3, ε-CuZn5 and Zn. Thermodynamic modeling was developed based on the thermodynamic models of the constituent binary systems and the experimental results from this study and those in the literatures. The calculated liquidus projection is in agreement with the experimental determinations. Solidification curves of the Sn-Zn-Cu alloys were determined using DTA, and the solidification paths of these Sn-Zn-Cu alloys were illustrated based on the solidification curves and the determined liquidus projection.

2:50 PM
Characterization of Ni-Si Phase Transformation: *Shiang Yu Tan*[1]; [1]Chinese Culture University

The interest in the low resistivity NiSi increased significantly because of the foreseeable use as contact to the source, drain, and gate of sub-65 nm and 45 nm CMOS devices. However, the thermal stability of NiSi is worse as the high resistivity phase NiSi2 nucleates at about 750°C and film agglomeration occurs even at a temperature as low as 600°C. The phase transformation from NiSi to $NiSi_2$ is of crucial importance for understanding the properties of nickel silicide and improving the process of IC industry. In order to obtaining a thermally stable Ni-FUSI gate electrode, we introduced properly tuned thickness of the initial Ni film and two-step RTP process during the poly-gate formation to push the transformation of NiSi2 to higher temperatures and retard agglomeration. Several measurement techniques such as XRD, TEM, Resitivity, AFM and XPS were carried out to characterize its physical and electrical properties.

3:05 PM
Phase Equilibria and Diffusion Paths to Fabricate Single-Phase Half-Heusler MNiSn (M=Ti, Zr, Hf) Thermoelectric Alloys: *Yoshisato Kimura*[1]; Chihiro Asami[1]; Hazuki Ueno[1]; [1]Tokyo Institute of Technology

Half-Heusler compounds MNiSn (M=Ti, Zr, Hf) are well-known to show excellent n-type thermoelectric properties at elevated temperatures around 1000 K. They are attractive because not only of high thermoelectric performance, but also of eco-friendly compositions containing no toxic element. However, it is very hard to prepare monolithic MNiSn alloys. Heterogeneous multi-phase microstructures are inevitable to form via non-equilibrium solidification in cast metallurgy, and even in powder metallurgy, since the melting point of Sn (505 K) is too much lower than other constituent elements. In the present work, phase equilibria in the M-Ni-Sn ternary systems were examined to establish the phase diagram information by means of electron probe microanalysis. Moreover, diffusion paths to form MNiSn phase were investigated using various types of diffusion couples. Based on these results, we have succeeded to fabricate single-phase MNiSn alloys, for instance, by directional solidification using the floating zone method.

3:20 PM Break

3:35 PM Invited
Effect of Water-Base Flux Containing Oxalic Acid on Interfacial Reaction between Cu Substrate and Pb-Free Solders: *Xing Jun Liu*[1]; Shun Mao Lin[1]; Cui Ping Wang[1]; [1]Xiamen University

In recent years, great attention has been given to the development of Pb-free solders for electronic interconnection materials because of the health and environmental safety problems caused by conventional Sn-Pb solders. However, for the Pb-free solders, it is required to further improve their wettability, and to restrain the growth of the intermetallic compounds (IMC) between solders and Cu substrate, which is associated with the reliability problem. In this work, a new type water-base flux containing oxalic acid was developed and good wettability between solders and Cu substrate was obtained by using this water-

base flux. The effects of the water-base flux containing oxalic acid on growth of the intermetallic compounds were investigated. Results indicate that the water-base flux with oxalic acid can hinder the growth of IMC, and improve the wettability and reliability in electronic packaging.

3:55 PM Invited
The Effect of Additives on Small Copper via Filling by Electroplating Method: Sukei Lee[1]; *Jae-Ho Lee*[1]; [1]Hong Ik University

Copper via filling by electroplating method became a common technology for interlayer metallization in 3D SiP(System In Package) and then it has been actively studied. The void free copper via can be obtained with varying the additives of the electrolytes and, the current density and types, pulse and pulse reverse. The effects of 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 10~20 micron via hole without void. The 10~20 micron small vias were successfully filled by copper electroplating.

4:15 PM
Study of Cu Surface Oxidation: *Zi-ming Huang*[1]; Chengyi[1]; [1]National Central University

Wettability of Pb-free solders on Cu metal bond pad highly depends on the oxidation condition of Cu bond pad surface. In this study, the oxidation behavior on Cu surface was studied. In this talk, two main parts will be presented: (1) Kinetic and phase formation of oxidation on Cu surface. The kinetic of Cu oxidation will be measured by ESCA and XPS. XRD will be used to analyze the Cu oxidation phase and structure. (2) We found that water contact angle is highly sensitive to the degree and condition of Cu surface oxidation. A correlation between the water contact angle and the Cu surface oxidation was established and will be reported.

4:30 PM
The System Ni-P-Sn and Its Binary Constituents: *Clemens Schmetterer*[1]; Jiri Vizdal[1]; Alexandre Kodentsov[2]; Herbert Ipser[1]; [1]University of Vienna; [2]Eindhoven University of Technology

Due to the use of phosphorus containing nickel substrates in microelectronics, understanding their reaction with Sn-based solders and the knowledge of the corresponding reaction products is highly important. Therefore, the ternary Ni-P-Sn system and relevant subsystems (Ni-P, P-Sn) have been studied experimentally (SEM + EDS, DTA/DSC, XRD) and theoretically (CALPHAD approach). On the basis of these investigations new versions of the binary phase diagrams were established. In the ternary Ni-P-Sn system the phase equilibria at various temperatures between 200 and 1050°C were determined. These results were enhanced with information obtained from a diffusion couple technique. New ternary phases were characterized with respect to their stability range and crystal structure.

4:45 PM
Evaluation of Peel Strength on Flexible Copper Clad Laminate with Ni-Cr Layer: *Bo-In Noh*[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University

In the rapid development of ultra large scale integrated (ULSI), Cu/polyimide system has been one of crucial technical concerns in multi interconnection scheme. In particular, as the number of output channel of IC driver increases and inner lead bonding (ILB) pitch in chip on flex (COF) demands the size below 30μm, the direct Cu deposition in two-layer flexible copper clad laminate (FCCL) fabrication will replace the present tie-coating process. Therefore, Cu metallization on polyimide will have steadily receive a great deal of attention because of continuous growing of IT market. The good adhesion between the metallic layer and the substrate in combination with the small foil thickness make flexible printed circuit boards (FPCBs) mechanically flexible. In addition, if polyimide is used for substrate material, the circuit board can operate at high temperatures. Therefore, we estimated that the reliability of FCCLs was affected by Ni-Cr layer using peel test method.

5:00 PM
The Effects of Pulse Plating Parameters on Ni-W Alloy Electroplating for UBM Application: Yeong Kwon Ko[1]; *Jae-Ho Lee*[1]; [1]Hong Ik University

Nickel has been used as a diffusion barrier material in UBM. Electro and electroless nickel plating have been extensively investigated. Even though nickel did not form IMC with tin at low temperature, nickel formed IMC with tin at high temperature. Nickel consumed the solder materials and then it affects on reliability of UBM. Tungsten is very good diffusion barrier material, however tungsten cannot be electroplated and has poor adhesion with tin. To utilize the advantage of nickel and tungsten, Ni-W alloy electroplating was investigated. Ni-W alloy film with crack free and low residual stress were obtained when pulse electroplating were employed. The interface of Ni-Sn and Ni(W)-Sn were analyzed by AES and EDS.

Recycling: General Sessions

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizers: Christina Meskers, Delft University of Technology; Greg Krumdick, Argonne National Laboratory

Wednesday PM — Room: 280
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Joseph Pomykala, Argonne National Laboratory

2:00 PM Introductory Comments

2:05 PM
On the Separation of Zinc from Dust in Ironmaking and Steelmaking Off-Gas Cleaning Systems: *Naiyang Ma*[1]; [1]Mittal Steel USA

Ironmaking and steelmaking off-gas cleaning system solid wastes often contain too high zinc to recycle for iron recovery through blast furnaces, but contain too low zinc to economically recover zinc from the wastes. The status of zinc in the ironmaking and steelmaking off-gas cleaning systems has been analyzed from three aspects: thermodynamics and kinetics of zinc deposition, correlation of zinc concentration with size and density of particles, and aerodynamics of dust particles in the off-gas cleaning systems. Theory and supporting data show that spontaneous separation of zinc from iron-bearing dust occurs in the ironmaking and steelmaking off-gas cleaning systems. Dry off-gas cleaning systems provide the most favorable conditions and environments for zinc to separate from dust. The coarser and heavier fraction of iron-rich solid wastes would be zinc-free or have low-zinc content if high-efficiency separators were to be used and appropriately arranged in the off-gas cleaning systems.

2:25 PM
Evaporation Behavior of ZnO with Ar-O_2-HCl-H_2O Atmosphere: *Kohei Yajima*[1]; Sang Han Son; Hiroyuki Mastuura[1]; Fumitaka Tsukihashi[1]; [1]University of Tokyo

The dusts and fly ash in the steelmaking industry and municipal solid waste incineration process contain metallic elements such as Fe, Zn, Pb and Cd with considerable amount of halogens. In Japan, zinc in dusts is recovered by Waelz kiln or MF process, etc. However, the presence of halogen in the dust affects the evaporation and condensation behavior of zinc and other metals, and disturbs the direct recycling of dust to steelmaking processes, because alkali or heavy metals reacted with halogen vaporize easily as halides and/or oxyhalide compounds in pyrometallurgical process. Recently, the formation mechanism of zinc oxychloride vapor was investigated and the formation of zinc oxychloride such as ZnOCl was proposed. However, considering the practical operation, the effect of water vapor on the evaporation behavior of oxychloride is not fully clarified yet. This study is focused on the investigation of evaporation behavior of ZnO with Ar-O_2-HCl-H_2O atmosphere.

2:45 PM
The New Resourcing Method of Converter Slag: *Liu Chengjun*[1]; [1]Northeastern University

According to the status of lower comprehensive utilization level for the converter slag in the word, and base on the chemical compositions and phase features of converter slag, the new resourcing method of converter slag were developed that some value-added products could be prepared such as calcium sulfate whisker and nano-Fe_2O_3. After converter slag was leached in H_2SO_4, calcium sulfate whisker and nano-Fe_2O_3 could be prepared from leching liquid

and solid products by the hydrothermal method. Effects of the temperature and solid-to-liquid ratio on the slenderness radio of calcium sulfate whisker were investigated, and effects of the pH and reaction temperature on the style, size and color of nano-Fe_2O_3 crystal were investigated also by XRD, TEM and chemical analysis. The growth mechanism, transformation condition and control method for calcium sulfate whisker and nano-Fe_2O_3 were set up by thermodynamic analysis and kinetic model.

3:05 PM

Study of Phosphorus Recovery from Rare Earth Solution with Phosphorus by Citric Acid Coordination: *Bian Xue*[1]; Wu Wenyuan[1]; [1]Northeastern University

Citric acid coordinated with rare earth elements, and the coordination products dissolved in water. So in this paper, the citric acid was used to separate rare earth with phosphorus. The relationship among the ratio of phosphorus recovery, citric acid addition, pH, temperature, and time was studied by the quadratic regression orthogonal analysis, and then the regression equation was obtained. The optimum process conditions of phosphorus recovery were obtained as follows: citric acid addition: 0.05mol•L-1, pH: 10, temperature: 30°C, and time: 20min, the ratio phosphorus recovery: 99.8%, rare earth loss: 0.98%. In addition, XRD analysis was used to investigate the sediment, and the reaction of alkality process was determined.

3:25 PM Break

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Study on the Adsorption Behavior of Modified Orange Peel Biosorbent on Cu2+: *Ningchuan Feng*[1]; Xueyi Guo[1]; [1]Central South University

A biosorbent, the chemically modified orange peel, was prepared from hydrolysis of the grafted copolymer, which was prepared by reacting methyl acrylate with cross-linking orange peel and the infrared spectroscopy (IR) was used for ensuring the structure of the biosorbent. Adsorption characteristics and effects of various factors of the biosorbent on Cu2+ were discussed including pH, adsorption time and initial concentration of Cu2+. Experiment results show that when pH was 6.0 and the initial concentration of Cu2+ was 50mg/L, the removal rate was 91.73% and adsorption capability was 80.53 mg/g after 6 h adsorption. After five times being reused, 85% removal rate could be obtained by the biosorbent.

3:55 PM

Removal of Pb^{2+} from Aqueous Solutions Using Modified Spent Grains: Qingzhu Li[1]; Liyuan Chai[1]; Qingwei Wang[1]; *Qing-hua Tian*; [1]Central South University

The adsorption process of Pb^{2+} from aqueous solutions by spent grains modified with 1mol/L NaCl solution was investigated. The effects of solution pH, contact time, adsorbent concentration, initial Pb^{2+} concentration, particle size on Pb^{2+} removal were also studied. The initial adsorption process was rapid and 90.35% of Pb^{2+} adsorption occurred within 30 min. The absorption equilibrium was obtained at 4h. When pH was 5.5, Pb^{2+} concentration in aqueous solutions decreased from the initial content of 20 mg/L to 0.01 mg/L after 5 hours by application of 10g/L adsorbent at 25°C, Pb^{2+} removal efficiency reached up to 99.95% and the Pb^{2+} remaining in aqueous solutions was under China Sanitary Standard for Drinking Water (GB5749-85). Adsorption data fitted well with Langmuir and Freundlich models. However, Langmuir isotherm model was better to describe Pb^{2+} adsorption than Freundlich model, indicating that the applicability of monolayer coverage of Pb^{2+} on the surface of adsorbent.

4:15 PM

Recycling of Wastes from Water Treatment Plant into Clayey Ceramic: *Carlos Maurício Vieira*[1]; Jhonatas Vitorino[1]; Sergio Monteiro[1]; [1]State University of the Northern Fluminense

In this work the properties and the microstructure of a clayey ceramic incorporated, up to 10 wt.%, with three type of wastes from a water treatment plant was evaluated. To determine the physical and mechanical properties such as linear shrinkage, water absorption and flexural strength, specimens were prepared by 18 MPa uniaxial pressure and then fired in laboratory furnace at 700°C.The microstructure of the ceramics was analyzed by SEM and XRD. The results indicated that the wastes changed the fired phases and increased the porosity of the ceramic. The results showed that these wastes modified the plasticity/workability of the clayey body as well as the evaluated properties of the fired ceramic. From the results it was found possible to recycle these wastes into clayey ceramic in low amounts to avoid deleterious effect on the quality of the fired product.

4:35 PM

The Shrinkage Behaviour of Recycled Glass Compacts: *Adele Garkida*[1]; Jiann-Yang Hwang[2]; Xiaodi Huang[2]; Charles Okuofu[1]; Bowen Li[2]; Abashiya Ahuwan[1]; [1]Ahmadu Bello University; [2]Michigan Technological University

Compacts of crushed waste window glass, fluorescent tubes, drinking glasses and laboratory glass wares were made at various proportions from 106 microns and minus 75 microns powders in combination with 5% bentonite as binder for each glass type, after several trials to determine compactibility of particle sizes. They were made using the uniaxial press at 10,000 psi and sintered at a temperature range of 700-750°C. The results showed anisotropic shrinkage behavior for all the samples, exhibiting an axial shrinkage lower than the radial shrinkage. Sintered compact from waste drinking glass which comprised of 60% 106 microns and 40% minus 75 microns powders showed the least sintering shrinkage factor of 1.125, fused at 700°C and the highest shrinkage factor of 1.286 at 750°C. This shrinkage behavior connotes that sintering can be employed to utilize these waste glasses up to the maximum of 95% recycled glass content in ceramic tile production.

Refractory Metals 2008: Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Refractory Metals Committee
Program Organizers: Todd Leonhardt, Rhenium Alloys Inc; Jim Ciulik, University of Texas at Austin

Wednesday PM
March 12, 2008

Room: 388
Location: Ernest Morial Convention Center

Session Chairs: James Ciulik, University of Texas at Austin; Omar Dogan, Albany Research Center, National Energy Technology Laboratory

2:00 PM

Analysis of the Purification of Iridium by Electron Beam Melting: *Evan Ohriner*[1]; [1]Oak Ridge National Laboratory

The purification of iridium metal by electron beam melting has been characterized for 48 impurity elements using glow discharge mass spectrographic (GDMS) analysis. No significant removal of six refractory metal elements occurred. The elements B, C, Al, Si, Cr, Fe, Ru, Rh, and Pt were partially removed by vaporization during electron beam melting. The remaining elements were undetectable following melting. Purification was analyzed using Langmuir's equation for vaporization with equilibrium vapor pressures calculated using Henry's law. Activity coefficients were obtained from published data for the elements Fe, Ti, and Pt. Activity coefficients were estimated from enthalpy data for several elements known to bond strongly to iridium. An ideal solution model was used for the remaining elements. The results are consistent with ideal mixing and some localized heating of the melt pool. Sponsor: Office of Radioisotope Power Systems, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

2:25 PM

New Method for Measuring Potassium Impurities in Molybdenum: Dick Beercheck[1]; *Leanna Ergin*[2]; David Kluk[2]; John Shields[3]; [1]Goldstein Group Communications; [2]NSL Analytical Services; [3]Mill Creek Materials Consulting

Potassium impurities in molybdenum can cause defects in sputtered coatings on LCD displays, leading to expensive coated glass having to be scrapped. To reduce defects, screen manufacturers are demanding higher purity molybdenum (in the 3 ppm potassium range), and the testing community is scrambling to develop more accurate techniques to verify these purity levels. Flame Atomic Absorption Spectroscopy (Flame AA) has been the preferred method for measuring potassium content of molybdenum. However, at the low levels being demanded by industry, Flame AA does not provide the required measurement accuracy. A method based on Gas Chromatography Mass Spectroscopy (GC/

MS) shows promise as an alternative to Flame AA in measuring potassium at the 3 ppm level. At its present stage of development, GC/MS can provide repeatable measurements at the 10 ppm level, and 5 ppm is in sight. Future work is aimed at providing accurate, repeatable measurements at the 3 ppm level.

2:50 PM
In-Situ Fracture Studies and Modeling of the Toughening Mechanism Present in Wrought LCAC, TZM, and ODS Molybdenum Flat Products: *Brian Cockeram*[1]; Kwai Chan[2]; [1]Bechtel Bettis Inc.; [2]Southwest Research Institute

Fractographic examinations of tensile and fracture toughness specimens from unalloyed LCAC, TZM, and Oxide Disperison Strengthened molybdenum have indicated that these alloys exhibit a ductile laminate toughening mechanism that is characterized as thin sheet toughening. In-situ examinations of fracture toughness specimens using a DISMAP method provide information on the stress-intensity values needed for crack propagation, path of crack propagation, and localized measurements of strain at the crack tip. C-scan measurements confirm that delaminations occur at the crack tip, and the development of the delamination zone with increasing stress-intensity is determined for each molybdenum alloy. A micromechanical model is developed to relate the toughness values to the features of the microstructure. Molybdenum alloys with a finer grain size, such as ODS molybdenum, are shown to exhibit higher toughness values at lower temperatures. The improvement in DBTT with respect to microstructural features is understood in terms of the thin sheet toughening mechanism.

3:15 PM
Measurement of High Temperature Properties of Refractory Metals: *Samuel Causey*[1]; Randall Jenkins[1]; Jack Spain[1]; Jason Wood[1]; [1]Southern Research Institute

Southern Research Institute has been characterizing materials for use in extreme environments for well over 50 years. The applications have included re-entry bodies, rocket motors, antenna windows, and nuclear reactor materials among many others. The range of temperatures has been from cryogenic up to about 3030°C (5500°F). These materials have included ceramics, composites, graphites, metals, and cermets. The specimens developed have been recently modified for testing rhenium, tungsten, and molybdenum, and their alloys across a wide temperature range. These modifications as well as important aspects of the techniques and hardware designs used in the testing of refractory metals are discussed. Two companion papers will discuss data generated on rhenium and molybdenum-rhenium using these specimen geometries and test techniques.

3:40 PM Break

3:50 PM
Anomalous Strain Rate Dependence of Ductility in a 50Mo-50Re Alloy at Room Temperature: *Jianhui Xu*[1]; Todd Leonhardt[2]; John Farrell[3]; Michael Effgen[3]; Tongguang Zhai[1]; [1]University of Kentucky; [2]Rhenium Alloys Inc.; [3]Semicon Associates

Tensile tests were conducted on a fully recrystallized 50Mo-50Re alloy, in the form of sheet of 0.216 mm thick, at different strain rates ranging from 10^{-6} s^{-1} to 1 s^{-1} at room temperature. It was found that the total elongation of the alloy increased significantly with increasing the strain rate. The fracture surface changed from brittle to ductile with increase in strain rate. Plastic deformation was found to occur by both slip and twinning in this alloy. The electron back scatter diffraction measurement revealed non-uniform plastic strain within individual grains, especially in the vicinity of grain boundary triple junctions. The decrease in ductility at low strain rates in this alloy was possibly related to the interaction between dislocations and trace interstitial elements.

4:15 PM
Tensile Ductility Reduction in Pure Rhenium at High Temperature: *Mark Opeka*[1]; Jie Zhang[1]; Samuel Causey[2]; Randall Jenkins[2]; Jennifer Gaies[1]; Keisha Sylvester[1]; [1]Naval Surface Warfare Center; [2]Southern Research Institute

Mechanical and thermal properties were obtained from pure rhenium samples produced by sintering and hot isostatic pressing, sintering and cold rolling, electroforming, and by chemical vapor deposition. Mechanical and thermal properties for these materials were obtained from room temperature up to 3500°F. The tensile testing results will be summarized. For all manufacturing processes, the highest tensile strains of 10 to 40% were observed at room temperature, with strain varying strongly with sample texture. The tensile strains decreased significantly with increasing temperature and values of 3 to 5% were observed at 3500°F and independent of texture. Reductions-in-area were very low or not measurable and grain boundary cleavage type failure was typically observed.

4:40 PM
Anisotropic Yield Function for Modeling Formability of Conventionally Rolled RRR Niobium: Amir Zamiri[1]; Hairong Jiang[1]; *Thomas Bieler*[1]; Farhang Pourboghrat[1]; Chris Compton[1]; Terry Grimm[1]; [1]Michigan State University

The overall mechanical properties of the high purity niobium (RRR) sheets, which are used in fabrication of the superconducting accelerator cavities, were investigated. High purity niobium has microstructure and texture gradients that lead to a rough surface and anisotropic deformation. High purity niobium is a rate sensitive material and its strength and ductility increases with increasing strain rate. Due to the presence of an unstable texture, the r values in high purity niobium sheets decrease with plastic deformation. From tensile tests in different in-plane directions and bi-axial bulge tests, a new mathematical model was developed to describe the evolving yield function that predicts the variation of the r values with respect to the effective strain and angle to the rolling direction. From this analysis, suggestions for optimal microstructure and texture for fabricating cavities from high purity niobium are proposed.

5:05 PM
First Principles Design of Ductile Refractory Alloys Validated by Experiments: *Michael Gao*[1]; Michael Widom[1]; Omer Dogan[2]; Paul King[2]; [1]Carnegie Mellon University; [2]National Energy Technology Laboratory

The purpose of this work is to demonstrate that the synergy that integrates first-principles calculations, computational thermodynamics and kinetics, and experimental design can be appropriately used to develop new materials for high-temperature applications in energy systems. Specifically, the Rice–Thomson parameter will be used as a computational screening tool for identifying ductilizing additives to the refractory alloys in addition to Poisson ratio. The advantage of using the Rice–Thomson parameter and Poisson ratio derives from the fact that they can be evaluated completely from first principles with virtually no empirical information. The calculations will be applied first to model simple prototype binary alloys based on Cr. Further, experimental measurements concerning selected Cr-binaries such as Cr-W will be conducted and used as benchmarks to examine theoretical calculations. Applications of current first principles calculations for phase diagrams and thermodynamics of refractory alloys will also be presented.

Structural Aluminides for Elevated Temperature Applications: New Class of Gamma Alloys

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Young-Won Kim, UES Inc; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Rui Yang, Chinese Academy of Sciences; Christoph Leyens, Technical University of Brandenburg at Cottbus

Wednesday PM
March 12, 2008
Room: 394
Location: Ernest Morial Convention Center

Session Chairs: Young-Won Kim, UES Inc; Vijay Vasudevan, University of Cincinnati

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2:00 PM Invited
A β-Stabilized γ-TiAl Based Alloy for Improved Processing Performance: *Helmut Clemens*[1]; Harald Chladil[1]; Wilfried Wallgram[1]; Sascha Kremmer[2]; Andreas Otto[3]; Volker Guether[3]; Arno Bartels[4]; [1]University of Leoben; [2]Bohler Schmiedetechnik GmbH&CoKG; [3]GfE Metalle und Materialien GmbH; [4]Hamburg University of Technology

The development of high-temperature materials is the key to technological advancements in aero-engines, where materials have to withstand extremely demanding conditions. TiAl alloys offer many attractive properties, such as low density, good oxidation and burn resistance as well as good creep properties

and high strength at elevated temperatures. Conventional high Nb bearing γ-TiAl based alloys exhibit a relatively strong tendency to segregations because of their peritectic solidification path. This leads to local microstructural inhomogeneities causing severe problems regarding hot-working behaviour and a scatter in mechanical properties. The investigated β-solidifying Ti-43Al-4Nb-1Mo-0.1B alloy (at.%) shows a homogeneous and fine grained microstructure in the cast state. Due to a high volume fraction of β-phase the alloy can be, e.g., forged under near conventional conditions. With subsequent heat-treatments a significant reduction of the β-phase is achieved, as predicted by thermodynamic calculations. Alloy characterization was conducted by SEM, EBSD, TEM, texture measurements, and mechanical testing.

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Microstructure and Properties of Beta-Solidifying Gamma-Titanium Aluminide Alloys: *Michael Oehring*[1]; Fritz Appel[1]; Jonathan Paul[1]; Renat Imayev[2]; Valery Imayev[2]; Viola Küstner[3]; [1]GKSS Research Centre; [2]Institute of Metals Superplasticity Problems; [3]Max-Planck-Institut für Metallforschung

Gamma titanium aluminide alloys solidifying solely via the beta-phase exhibit characteristic solidification microstructures, which often involve equiaxed instead of columnar structures, weak textures, and modest segregation. These features result from single-phase solidification and the subsequent solid-state transformations. In view of the development of improved cast alloys, the potential of the beta/alpha transformation with respect to microstructural refinement and its dependence on the addition of several alloying elements has been investigated. It was found, that particularly fine and very homogeneous microstructures can be obtained for certain alloy compositions. The microstructural refinement can be attributed to the alloying effect on the kinetics of the beta/alpha transformation and even can be achieved after slow cooling from high-temperature heat treatments. The paper discusses alloy concepts aiming at refined cast alloys that also appear to be useful with respect to ingot material for wrought processing routes.

2:50 PM Invited

Microstructure and Corresponding Tensile Properties of as Cast β-Solidifying γ-TiAl Based TNM Alloys: *Volker Guether*[1]; Andreas Otto[1]; Joachim Klose[2]; Christiane Rothe[2]; Helmut Clemens[3]; Werner Kachler[4]; Susanne Winter[4]; Sascha Kremmer[5]; [1]GfE Metalle and Materialien GmbH; [2]FNE Forschungsinstitut fuer Nichteisen-Metalle Freiberg GmbH; [3]University of Leoben; [4]Zentrum fuer Werkstoffanalytik Lauf GmbH; [5]Bohler Schmiedetechnik GmbH&CoKG

VAR ingots of γ-TiAl based TNM alloys which solidify completely via the β-phase exhibit fine grained microstructure consisting of lamellar γ / α2- colonies and globular γ- and β/B2-grains. The volumetric ratio between the three phases depends mainly on the aluminum content and the content of the major β-stabilizing alloying elements molybdenum, niobium and boron. The thermodynamic stability of the microstructure at operation temperature is characterized by the investigation of the equilibrium phase formation at elevated temperatures using high temperature XRD measurements. The excellent tensile properties at room temperature and 700°C of thermally stress relieved as cast VAR materials are related to the microstructure and, thus, to the chemical composition of the TiAl alloy. It is expected that γ-TiAl based alloy compositions can be adjusted to the specific needs of both final application and further processing steps.

3:20 PM

"Near Conventional" Forging of a β-Stabilized γ-TiAl Based Alloy: *Sascha Kremmer*[1]; Harald Chladil[2]; Wilfried Wallgram[2]; Helmut Clemens[2]; Andreas Otto[3]; Volker Güther[3]; Arno Bartels[4]; Wilfried Smarsly[5]; [1]Bohler Schmiedetechnik GmbH&CoKG; [2]Montanuniversität Leoben; [3]GfE Metalle und Materialien GmbH; [4]Hamburg University of Technology; [5]MTU Aero Engines GmbH

Due to the strong demand for higher efficiencies, reduced CO2 emissions and weight reduction in aircraft engines, the substitution of presently used materials by novel light-weight, high-temperature alloys like γ-TiAl based alloys is at the edge of realization. This paper summarizes detailed investigations on a "near conventional" forging route for the fabrication of TiAl components. γ-TiAl based alloys with high Nb contents exhibit a narrow forging window, which show severe shifts if only slight changes in alloy composition occur. However, when using an alloy with an optimized content of β-stabilizing elements the forging window can be widened with respect to billet temperature, die temperature and die speed. Forging experiments conducted on this specially developed Nb and Mo containing γ-TiAl based alloy (TNM), demonstrate the feasibility of a robust and economic manufacturing route. Additionally, the results of texture measurements are shown and the mechanical properties of forged TNM material are presented.

3:40 PM Break

3:50 PM Keynote

Development of Beta Gamma TiAl Alloys: Opening Robust Processing and Greater Application Potential: *Young-Won Kim*[1]; Sang-Lan Kim[1]; Dennis Dimiduk[2]; Christopher Woodward[2]; [1]UES Inc.; [2]Air Force Research Laboratory

The primary material and manufacturing limitations of gamma TiAl alloys include large cast lamellar grains, an inhomogeneous distribution of microstructure and composition, and processing difficulties. Efforts have been made in developing a new class of TiAl-based alloys, called beta gamma, which would overcome or reduce such barriers. The design concept was to identify gamma alloy systems, which are solidified through the beta-solidification process, thereby producing phase fields where the β phase amount is adequate at elevated temperatures but low or negligible at the anticipated use temperatures. Such alloy compositions were determined to exist within Ti-(40-45)Al-(2-8)Nb-(1-9)(Cr, Mn, V, Mo)-(0-0.3)B in and around the ternary phase field (γ + β/B2+ α/α2). Three groups of beta gamma alloys, rich with gamma phase and having distinct transformation pathways, were explored, featuring remarkably refined microstructures and compositional homogeneity. The ongoing development process will be discussed, along with the initial properties and associated engineering implications.

4:30 PM

Effect of Small Boron and Carbon Additions on the Mechanical Properties of a Novel High Niobium-Containing Gamma Titanium Aluminide Alloy: *Janny Lindemann*[1]; Maria Glavatskikh[1]; Stefan Schmidt[1]; Christoph Leyens[1]; Dan Roth-Fagaraseanu[2]; [1]BTU Cottbus; [2]Rolls-Royce Deutschland

Recently, the alloy design of high niobium-containing gamma titanium aluminide alloys is focused on increased beta-stabilized alloys (TNM alloys). Increased beta-stabilizing of TNM alloys compared to conventional high niobium-containing alloys (TNB alloys) leads to an increased beta-volume content in these alloys which can be beneficial with regard to ductility and formability of the alloys. However, the fatigue performance and the creep resistance of the TNM alloys can be reduced due to the beta-volume content. In the present work the mechanical properties of an extruded TNM alloy were compared with those of a conventional TNB alloy. Furthermore, the effect of a small amount of boron and carbon on the mechanical properties was investigated. While the presence of borides could significantly improve microstructure refinement and homogenization during extrusion, a small amount of carbon can lead to higher strength and creep resistance due to precipitation hardening.

4:50 PM

Development of Multiphase Gamma-TiAl Alloys with Enhanced Mechanical Properties: *Renat Imayev*[1]; Valery Imayev[1]; Timur Khismatullin[1]; Tatiana Oleneva[1]; [1]Institute for Metals Superplasticity Problems

The effect of heat and thermomechanical treatment on the microstructure and mechanical properties of two groups of beta-solidifying gamma-TiAl alloys with the metastable or stable beta (B2) phase in the composition range of Ti-(43-46)Al-(4.5-7)(Nb,Mo)-0.2B-(0-0.3)C has been considered. In the first group of the alloys the molybdenum content was within the limits of solubility in the gamma and alpha-2 phases, in the second group the content exceeded this limit. The metastable beta (B2) phase promotes refinement of the structure upon freezing and cooling and can be dissolved by heat treatment that allows a good balance of mechanical properties to be obtained. The stable beta phase gives rise to some a reduction in high temperature mechanical properties, but improves significantly ductility and workability. Both the alloy groups possess improved processing characteristics in comparison with currently known gamma-TiAl alloys.

5:10 PM

Forging of β-Phase Containing γ-TiAl Alloys: *Jiulai Zhang*[1]; Stefan Schmidt[1]; Fritz Appel[2]; Christoph Leyens[1]; Bernd Viehweger[1]; [1]Brandenburg University of Technology Cottbus; [2]GKSS Research Centre

Forging of conventional γ-TiAl alloys is difficult due to their ordered structure. Up to now the only feasible closed-die forging process is the isothermal forging. The introduction of β-phase in γ-TiAl alloys may make the conventional closed-die forging possible. Upsetting tests were carried out on some newly developed β-phase containing γ-TiAl alloys with warm dies made from a Ni-base superalloy with varied strain rates and die temperatures. Both bare and canned specimens were used. The canning consists of three layers of steel foil (foil thickness 0.1 mm) and a layer of glass. 75% upsetting was reached at optimal forging parameter. The forged specimens were examined with light microscopy and SEM. The results show that the newly developed β-phase containing γ-TiAl alloys can be forged at much higher strain rates than those conventional γ-TiAl alloys. Canning reduces drastically the die temperatures needed for a defect free forging.

Structural Aluminides for Elevated Temperature Applications: Poster Session

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS/ASM: Mechanical Behavior of Materials Committee

Program Organizers: Young-Won Kim, UES Inc; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Rui Yang, Chinese Academy of Sciences; Christoph Leyens, Technical University of Brandenburg at Cottbus

Wednesday, 5:40-8:00 PM Room: 394
March 12, 2008 Location: Ernest Morial Convention Center

Session Chairs: Maria Perez-Bravo, ITP; Chao Jiang, Los Alamos National Laboratory; Christoph Leyens, Technical University of Brandenburg at Cottbus; Rui Yang, Chinese Academy of Sciences; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC

An Electron Microscope Study of Low-Cycle Fatigue of a High Niobium Containing and Precipitation Hardened TiAl Alloy: *Fritz Appel*[1]; Thomas Haeckel[2]; Hans-Jürgen Christ[2]; [1]GKSS Research Centre; [2]Universität Siegen

The fatigue behaviour of an extruded TiAl alloy with the composition Ti-45Al-8Nb-0.2C (at. %) will be described. Fully reversed isothermal tests were performed under strain control at a strain amplitude of 0.7% and the temperatures 25, 550 and 850°C. TEM examination of the samples fatigued at 25 and 550°C revealed dense structures of ordinary dislocations and debris that were accumulated in tangles. The dipole defects apparently serve as additional glide obstacles but may also contribute to dislocation multiplication if the local stress rises. Another important low temperature deformation mechanism is the stress-induced transformation of the orthorhombic B19 phase, which is a significant constituent of the microstructure. The B19 phase is apparently unstable under tetragonal distortion and transforms into gamma phase via distinct shuffle operations. Under high-temperature fatigue the lamellar microstructure degrades by dynamic recrystallization. Details of these processes were investigated by high-resolution electron microscopy.

Fracture Toughness of an Intermetallic TiAl: *Nicolas Barbi*[1]; Russell Goodall[1]; Frédéric Diologent[1]; Andreas Mortensen[1]; [1]Ecole Polytechnique Fédérale de Lausanne

Cast TiAl-based alloys have potential in several applications such as valves or turbocharger wheels; however, the suitability of these materials in critical applications such as gas-turbine blades may be limited by their generally brittle behaviour. We present results from a study of the toughness of gamma/alpha2 titanium aluminides conducted within the framework of a wider collaborative project on these materials. Three-point bend fracture toughness tests conducted under controlled atmosphere on precracked specimens, varying the alloy composition, heat treatment and test temperature are used to assess the suitability of these cast alloys for applications, as well as relations between basic microstructural parameters, such as the alpha2 phase contents and distribution, and their resistance to crack propagation.

Environment Protection of γ-TiAl Alloys Coated with CrAlYN/CrN Nanoscale Multilayer Coatings and EB-PVD Thermal Barrier Coatings: *Reinhold Braun*[1]; David Müßener[1]; Christoph Leyens[2]; Papken Hovsepian[3]; Christina Reinhard[3]; Arutiun Ehiasarian[3]; [1]DLR - German Aerospace Center; [2]Technical University of Brandenburg at Cottbus; [3]Sheffield Hallam University

The oxidation protection capability of nanostructured CrAlYN/CrN multilayer coatings for gamma titanium aluminides was studied at 750 and 850°C under cyclic oxidation conditions. Nitride coatings of about 5 µm thickness with an oxy-nitride top coat were deposited on γ-TiAl samples with Cr^+, $CrAl^+$ and Y^+ etched interfaces. EB-PVD thermal barrier coatings of yttria stabilized zirconia were deposited on some of the samples using CrAlYN/CrN + CrAlYON as bond coat. CrAlYN/CrN coatings exhibited excellent oxidation resistance at 750°C. A significantly reduced mass gain was observed for coated samples in comparison to the bare γ-TiAl alloy. With regard to the different surface pre-treatments, the Y^+ etched interface exhibited the most beneficial effect on oxidation resistance. The multilayer nitride coatings provided also good oxidation protection at 850°C. No spallation of the thermal barrier coating systems was observed during 2000 cycles at 850°C. The TBCs exhibited excellent adhesion to the oxy-nitride top coat.

Fabrication of TiAl Foils by Heat Treatment Only: *Jiulai Zhang*[1]; Chistoph Leyens[1]; Bernd Viehweger[1]; [1]Brandenburg University of Technology Cottbus

TiAl foils were prepared by heat treatment of Ti/Al foil packs. After heat treatment at high temperatures for 0.5h TiAl foils with a thickness of 85 µm were attained. Different from the results in literature, where Ti and $TiAl_3$ instead of γ phase were achieved, the foils are free from Ti, $TiAl_3$ and contain only γ phase and α_2 phase. The as-heat-treated foils show good surface quality too. The foils show a coarse lamellar microstructure through the whole thickness of the foils. A row of pores were found in the middle plane of the foils. The reason of the pore formation was discussed and methods were tried to remove the pores.

Microstructure Characterization and Mechanical Property of Ti/Tial Sheets Prepared by EBPVD: *Liping Shi*[1]; Xiaodong He[1]; Li Ma[1]; Yesheng Zhong[1]; [1]Harbin Institute of Technology

By the double resource method of electron beam physical vapor deposition, Ti and TiAl ingot were evaporated in their given order onto the substrate. And Ti/TiAl sheets with total thickness of 1 mm were fabricated, in which Ti is served as tough phase to improve the fracture toughness property of base phase TiAl. This basic concept of the toughness procedure is defined that tough particles were bridged to connect the main crack surface of the brittle matrix. Based on the results of DSC, XRD and SEM analyses, the microstructure of Ti/TiAl sheets were characterized. Additionally, the mechanical property including tensile, creep and fatigue testing were performed. According to the experimental results, the property of the Ti/TiAl sheets is up to the structure and property of each composition and its main influencing factor includes body content, interlayer spacing and mutual solubility.

Multiple F-Implantation for Improved Oxidation Protection of Gamma-TiAl Alloys: Hans-Eberhard Zschau[1]; Michael Schuetze[1]; *Alexander Donchev*[1]; [1]DECHEMA e. V.

To overcome the insufficient oxidation resistance of Gamma-TiAl above 750°C the fluorine effect is well established. After fluorine doping on the TiAl surface an alumina scale is formed. Several methods of fluorine treatment were considered. The fluorine content at the metal/oxide interface was found to be an important stability parameter. Its time behaviour follows an exponential decay function with a constant term of about 1 at.-% after oxidation for 1000 h at 900°C. To improve the long-time stability of the protective effect a fluorine reservoir was established by using multiple implantation techniques. Implantation energies between 20 keV and 120 keV and ion fluences between 5E16 and 6E17 F cm-2 were considered. The implantation profiles varied between two step and multiple implantation. The fluorine depth profiles were obtained by using the non-destructive PIGE technique showing an optimized shape suitable for long oxidation times.

On the Influence of Nb and C on the Phase Transition Temperatures in γ-TiAl Based Alloys: *Harald Chladil*[1]; Helmut Clemens[1]; Ernst Kozeschnik[2]; Arno Bartels[3]; Rainer Gerling[4]; Sascha Kremmer[5]; [1]Montanuniversity Leoben; [2]Graz University of Technology; [3]Hamburg University of Technology; [4]GKSS Research Centre; [5]Böhler Schmiedetechnik GmbH&Co KG

Intermetallic titanium aluminides are of increasing technical importance for high-temperature applications in automotive and aerospace industries due to their low density, combined with high yield strength, good creep properties and good oxidation behaviour. Current γ-TiAl based alloys are an example of complex multi-phase materials. Therefore, the knowledge of the constituting phases and their solid-state transition temperatures as well as the influence of alloying elements is the basis for thermo-mechanical processing and for smart heat treatments, e.g. for the optimization of mechanical properties. In the present study phase transition temperatures and constituting phases of alloys showing a baseline composition of Ti-45Al-(5-10)Nb-(0-0.5)C (at%) were investigated by means of DSC, scanning electron microscopy, electron back-scattered diffraction and X-ray diffraction. For the prediction of phases and phase transition temperatures, thermodynamic calculations based on the CALPHAD method were performed using a commercially available database, which also was adapted with some corrections.

Phase and Microstructure Evolution and Tensile Properties in a Beta Gamma Alloy Ti-44Al-4Nb-3.5(Mn, Cr)-0.2B: Young-Won Kim[1]; *Christopher Woodward*[2]; Dennis Dimiduk[2]; [1]UES Inc; [2]Air Force Research Laboratory

Beta gamma alloys are under exploration as possibly "more robust" gamma-base alloys. Their compositions exist within a range of Ti-(40-45)Al-(2-8)Nb-(1-9)(Mn, Cr, V, Mo, Cu)-(0-0.3)B-(0-0.4)C. Three groups of beta gamma alloys, rich with gamma phase (>85%) at use temperatures, were determined to exist in and around the ternary phase field (γ-TiAl + ß-Ti + α2-Ti3Al). A three-phase alloy, Ti-44Al-4Nb-3.5(Mn, Cr)-0.2B, was investigated to evaluate its phase transformation pathways and microstructure evolution taking place upon heat treatments for both ISM ingot material and an extruded material. These processes were analyzed using phase and grain size distribution measured as a function of temperature and heat treatment condition. The resulting information and data was used to determine extrusion conditions and also define new useful microstructure types with a desirable phase and microstructure distribution. Property testing is underway, and the results will be correlated to measured microstructures to define their relationships.

Surface Strengthening for Enhanced Fatigue Performance of Gamma Titanium Aluminides: *Maria Glavatskikh*[1]; Janny Lindemann[1]; Christoph Leyens[1]; Michael Oehring[2]; Fritz Appel[2]; [1]Brandenburg University of Technology Cottbus; [2]GKSS Research Centre

The fatigue performance of modern high niobium-containing gamma titanium aluminides can be significantly improved by mechanical surface treatments such as shot peening and roller burnishing. Fatigue strength improvements depend on the process-induced work hardening, the residual compressive stresses in the surface layer and on the surface roughness. While at ambient temperature the improvement of the fatigue strength is mainly caused by the process-induced residual compressive stresses, the residual stresses hardly affect the fatigue performance at elevated temperatures since they are quickly relaxed at service temperatures above 650°C. Nevertheless, surface strengthening can be beneficial for fatigue performance even at elevated temperatures. In the present work the potential of surface strengthening to enhance the fatigue performance of high niobium-containing gamma titanium aluminides at elevated temperatures was assessed. Furthermore, methods for improving the thermal stability of defect structures produced by surface strengthening were investigated.

Thermodynamic and Diffusion Modelling for Nanostructured Coatings Design on Gamma-TiAl Aluminides for High Temperature Applications: *Francisco Perez-Trujillo*[1]; Juan Nieto[1]; Maria Hierro de Bengoa[1]; Sophia Tsipas[1]; Juan Leal[1]; Sonia Mato[1]; [1]Universidad Complutense

Thermodynamic and siffusion modelling on gamma-TiAl have ben applied in order to model the oxidation behviour in different aggresive environments at high temperatures. From these data, solid phases can be model and use it to optimize the coating composition and design. Moreover diffusional apporaches have been done to know the interdiffusion processes and metalic elements depletion in the process to form protective oxidized layer on the nanostructured coated gamma-aluminide. Some validation of this modelling approach will be given for micro/nano structuctured coatings.

Diffusion Bonding between TiAl and Ti_2AlNb: Jianying Zou[1]; Yuyou Cui[1]; *Rui Yang*[1]; [1]Institute of Metal Research CAS

Direct diffusion bonding of orthorhombic Ti_2AlNb-base alloy to TiAl-base alloy was carried out and the interface microstructure, formation of new phase at the interface and joint strength were characterised. At low temperature, a new phase with $AlNb_2$-structure, $Al(Nb,Ti)_2$, was formed at interface adjacent to the O-based alloy. The α_2 was found to be the major reaction product and developed at interface adjacent to the TiAl alloy as well as at interface adjacent to the O-base alloy accompanying $Al(Nb,Ti)_2$. The formation of $Al(Nb,Ti)_2$ has been attributed to the different diffusion velocity of Nb and Al leading to a eutectoid-like reaction. At relatively high temperature, $Al(Nb,Ti)_2$ did not form due to enhanced diffusivity of Nb but a B2-enriched zone formed instead after long holding time. Only when an appropriate phase composition was achieved through diffusion could the shear strength of the joint reach 80% of that of the TiAl base metal.

Deformation in TiAl by the Formation of L11 Pseudotwin: *Dongsheng Xu*[1]; Hao Wang[1]; Rui Yang[1]; [1]Institute of Metal Research, Chinese Academy of Sciences

The shear deformation of twinned and perfect TiAl with L10 structure at various temperature and hydrostatic pressure was investigated using atomistic simulations. By shearing in [211] direction on the (111) plane under hydrostatic compression, an L11 structured pseudo-twin was found to form at low temperature. The critical conditions for activating different twinning system were identified with the help of molecular dynamic and static simulation. The activation of the pseudo-twinning is expected to benefit the strain transfer and the reduction of the stress concentration at the grain or lamella boundary. The L11 pseudo-twin in TiAl was observed only in limited experiments, consistent with the difficulty of its formation. Our results showed that the L11 phase has a higher energy than normal twins, and can only form by shearing under hydrostatic compression. Further first principles calculation showed that some alloying elements may have the potential to promote this transformation.

Anisotropic Properties of Interfaces in Fe-Al: *Vaclav Paidar*[1]; Jaromir Kopecek[1]; Pavel Lejcek[1]; [1]Institute of Physics

The properties of interfaces such as grain and antiphase boundaries are primarily derived from the atomic structure in their core regions. Structural and chemical inhomogeneity of these planar interfaces can play a decisive role for material behaviour including mechanical properties. Dislocation motion is affected by their core structure that depends on the stacking-fault-like defects and by the interaction with barriers as grain boundaries. Fracture is essentially governed by processes on atomic scale when the interatomic bonds are interupt. In polycrystalline materials where the grain boundaries represent regions of different atomic arrangements, the cracks can nucleate and propagate under specific conditions along the boundary planes. The processes identified on interfaces will be discussed from the point of view of different grain boundary types and their behaviour will be deduced from experimentally studied grain boundaries in bicrystals and from theoretical considerations.

Effects of Ru Addition on Phase Equilibria, Microstructures and Mechanical Properties of $E2_1$ Co_3Alc_{1-X} Based Alloys: *Yosuke Uotani*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology

$E2_1$ Co_3AlC_{1-x} is one of the most possible candidates for a new strengthening phase in Co based heat resistant alloys. $E2_1$ Co_3AlC_{1-x} transforms to $E2_1$' $Co_3AlC_{0.5}$ at 1325K due to the extra-ordering of C atoms associated with the APD formation. $E2_1$' consists of alternate stacking of $E2_1$ and $L1_2$ type close-packed {111} planes. Single crystal of Co_3AlC_{1-x} exhibits excellent ductility and high strength in a temperature range from 300 to 1373K. The influence of APD size on room temperature ductility was investigated using single crystals. Co_3AlC_{1-x} with fine APDs shows no plastic deformability. From the viewpoint of the improvement in elevated temperature strength, small amount of Ru addition was conducted and phase equilibria were investigated to establish the pathway to design new Co based heat resistant alloys. We have found that Ru is a B2 former and a solubility of Ru in Co_3AlC_{1-x} is limited to a few at.%.

Effects of Ternary Additions on Mechanical Behaviour of B2-Ordered Fe-40 at.% Al Alloys: *Anna Fraczkiewicz*[1]; Sanaa Najjar[1]; David Colas[1]; Adrien Chapuis[1]; [1]Ecole Nationale Superieure des Mines

Forty years after beginning of development of B2-ordered FeAl for structural applications, their poor room temperature ductility still remains the main locking point. Nowadays, all existing solutions of avoiding this problem involve an extreme grain size refining by P/M-type technologies that make the fabrication cost of FeAl prohibitive. A new way of progress could come with development of optimised, ternary or quaternary, alloy compositions.Fe-40Al alloys, B-doped and containing some ternary elements (Ni, Co, Mo, Ti, Nb, Mn) have been studied. Their behaviour was appreciated from RT tensile tests and high temperature (20 – 900°C) compression tests. Among studied additions, Ni, Co and Mn seem the most interesting. Nickel and cobalt provoke a significant hardening associated with an important RT brittleness. In Mn-alloyed FeAl, an increase of RT ductility is associated with an unchanged YS value. These results will be analysed on the basis of alloys fine microstructure.

Fabrication and Strengthening of Ni-Based Thermostructural Panels: *Sara Johnson*[1]; Tresa Pollock[1]; [1]University of Michigan

Thermostructural panels for use in emerging hypersonic flight systems require the use of advanced materials able to support substantial loads at elevated temperatures. The major challenge in this advancing technology is identifying formable structural materials that are both strong and oxidation resistant. A new processing path beginning with sheets of Ni-based alloys has been developed where the solid-solution alloy is formed and subsequently strengthened by vapor phase aluminization. Fabrication, microstructure, mechanical properties, and oxidation behavior of these panels will be discussed.

Microstructure and Mechanical Properties of a Eutectoid FeNiMnAl Alloy: *Yifeng Liao*[1]; Ian Baker[1]; [1]Dartmouth

The microstructure and mechanical properties of a novel eutectoid alloy with the composition (in at.%) of Fe-20Ni-35Mn-15Al have been studied. The alloy was arc melted from elemental powders, and SEM/EDS and TEM/EDS were used to determine the microstructures and compositions of the phases present. The results revealed that the compound was composed of FCC and B2 lamellae, whose widths were ~300 nm and ~200 nm, respectively. Straining under compression produced yield strengths ranging from 750 MPa at 300 K to 160MPa at 1000K, with a rapid drop in yield strength at ~600K. Elongations of 25%-30% were observed in tensile tests at room temperature, with fracture surface analysis indicating a ductile fracture mode. The dislocation behavior was also investigated. Research supported by NSF grant DMR-0505774.

Ternary Interdiffusion in Ni_3Al with Ir or Ta Additions: *Narayana Garimella*[1]; M. Ikeda[2]; Machiko Ode[3]; Hideyuki Murakami[3]; Yong-ho Sohn[1]; [1]University of Central Florida; [2]Kobe Steel Company; [3]National Institute for Materials Science

Average ternary interdiffusion coefficients in Ni_3Al with Ir or Ta additions have been determined using solid-to-solid diffusion couples annealed at 1200°C for 5 and 25 hours. Disc shaped alloy specimens were prepared by vacuum arc melting at various compositions within the Ni_3Al with minor alloying up to 3 at.% Ir or 1.5 at.% Ta. Concentration profiles of individual components were measured by electron probe microanalysis and interdiffusion fluxes were determined and examined to calculate the average ternary interdiffusion coefficients. Average main interdiffusion coefficients of Ir or Ta were observed to be two orders of magnitude lower than those of Al and Ni. A significant diffusional interaction between Ir and Ni, and between Ta and Al was observed, highlighted by large magnitudes in average cross interdiffusion coefficients. Variation of average ternary interdiffusion coefficients is presented with respect to sublattice sites of Ni_3Al and applications in high temperature alloys and coatings.

The Development of High Specific Strength Wrought Fe3Al-Based Alloys: *Satoru Kobayashi*[1]; Stefan Zaefferer[2]; Takayuki Takasugi[1]; [1]Tohoku University; [2]Max-Planck-Institut Fuer Eisenforschung GmbH

Our recent research on grain refinement of Fe3Al-based alloys through thermomechanical process revealed that coarse κ-Fe3AlC precipitate particles are effective in introducing nucleation sites for recrystallization, and thereby refining grain size.[1, 2] Between room temperature and 600°C, fine-grained materials showed specific strengths as high as Ti-based alloys, indicating that Fe3Al-based alloys might be useful for structural components such as low-pressure turbine blades and compressors. Fatigue properties and tensile properties in water vapor atmosphere will be presented. [1]Kobayashi S, Zaefferer S: Microstructure Control Using Recrystallization in Particle-containing Fe3Al-based Alloys, Materials Science Forum Vol. 558-559. pp. 235-240 (2007). [2]Kobayashi S, Takasugi T: Grain Refinement of a Fe3Al-based Alloy Using κ-Fe3AlC Precipitate Particle Stimulating Nucleation of Recrystallization, Intermetallics, accepted.

The Role of Engineers in Meeting 21st Century Societal Challenges — AIME Keynote Session

Sponsored by: The Minerals, Metals and Materials Society, TMS: Public and Governmental Affairs Committee
Program Organizers: Diran Apelian, Worcester Polytechnic Institute; Iver Anderson, Iowa State University

Wednesday PM — Room: 272
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chair: Dan Thoma, Los Alamos National Laboratory

2:00 PM Introductory Comments by Dan Thoma, Los Alamos National Laboratory

2:10 PM Invited
The Role of Engineers and the Profession in the 21st Century: *Diran Apelian*[1]; [1]Worcester Polytechnic Institute

Globalization of the economy has amplified the impact of technology on modern societies in ways that could not have been predicted. The connectivity provided by the Internet has generated new markets for products and services, but has also made available labor that is often both educated and cheap. This is likely to have a profound impact on the distribution of wealth in both the developed and the developing part of the world and may, in particular, alter the socio-economic structure of countries where the general well- being of the population has been taken for granted. The role of engineers and the profession is critical for the sustainable development of our planet. The 21st Century presents us with challenges (as well as opportunities) that will require a "new" breed of engineers. These issues and the impending actions that we need to take will be discussed in this presentation.

2:30 PM Invited
People First: Global Mission: *Jaleel Al-Khalifa*[1]; [1]Saudi Aramco

In the past, share holders' emphasis on maximizing short-term commercial interest (The bottom line) has steered corporate management. The notion of management by values was introduced to better engage the workforce. Introducing the corporate social responsibility (CSR) to help local communities has also gained significant momentum. The notion of the three bottom-line (share holders, customers, workforce) was also introduced. The next essential reform is to apply "People First" paradigm, i.e. the four bottom-line (share holders, customers, workforce, and humanity at-large). "People First" ensures the commercial success of the organization, while meeting the global mission of advancing growth and prosperity of mankind. It represents a very systematic approach to running business while serving humanity. It aims at ensuring the commercial success while availing adequate food, water, shelter, health and education for the neighbor community. The role of professional societies to promote "People First" paradigm cannot be further emphasized. We, professionals, need to continually sharpen our technical competency and at the same time nurture the human side of our character. It is both technical competency and personal character that ensure excellence in our future careers.

2:50 PM Invited
Energy Sources for the 21st Century - Implications and Challenges: *Tomas Diaz De La Rubia*[1]; [1]Lawrence Livermore National Laboratory

Economic development and population growth trends indicate that global energy demand will double by the year 2050 to 25 TW-yr and will reach 50-60 TW-yr by 2100. At the same time, climate models indicate that continued emissions of greenhouse gases into the atmosphere will likely lead to global surface temperature increase by the year 2100 ranging from 1.1 C to 2.9 C in

the low impact scenario. Fossil fuels, the main culprits behind current increases in CO_2 concentration in the atmosphere, currently provide 85% of the world's energy demand, but are likely to deplete over the next decades for oil and gas, or the next two centuries for coal. Meeting the growth in energy demand while mitigating climate change will demand new energy sources beyond fossil fuels such as solar, nuclear and ultimately fusion. The development and massive worldwide deployment of these clean, alternative sources to produce energy at economically competitive rates will require new, advanced materials capable of performing with high efficiency, safety and reliability, often in very extreme environments. In this talk, I will describe some of the world wide efforts to develop new materials for energy applications with an emphasis on fission and fusion energy.

3:10 PM Break

3:20 PM Invited
Sustainable Mobility: The Grand Challenges: *John Moavenzadeh*[1]; [1]World Economic Forum

Transportation of both people and goods will face a number of interconnected challenges over the next decades, including: Urbanization: The world's urban population will grow from roughly 3.2 billion today to roughly 4.9 billion by 2030, an increase of over 50%.[1] United Nations World Urban Population Prospects: 2005 Revision. How will this fundamental transformation in where we live affect the automotive industry? How will people get around in the city of the future? Global Climate Change: Public awareness of global climate change continues to grow, and governments are increasingly ready to act. Does the automotive industry have the best strategy to engage governments and other stakeholders on the climate change issue? Developing World Motorization: Tata Motors of India is launching a $2500 car for the Indian market. China is now the second largest automotive market in the world. What are the implications of a rapidly motorizing developing world? Other noteworthy challenges include the global volume of vehicle-related fatalities, the overwhelming dependence of the transport sector on fossil fuels, an ageing air transport infrastructure that cannot keep up with increasing demand, and others. Engineers have a critical role to play in developing technology that will enhance the sustainability of the transport network for future generations; however, engineers will need to work closely with other stakeholders. [1]United Nations World Urban Population Prospects: 2005 Revision.

3:40 PM Invited
Housing for the 21st Century – Design, Technology and Construction: *Stephen Lee*[1]; [1]Carnegie Mellon

Traditional American methods of homebuilding are not adequately responding to the need for flexible, affordable, energy effective and resource efficient homes. This session will take at fresh look at the housing delivery process in response to global and regional change. From climate change, to power deregulation, to suburban sprawl to the rapid proliferation of information technology, change is occurring at a more rapid pace than at any other time in our history. Yet, the housing industry is a fragmented, multi-headed beast in which change is slow to occur, if at all. Our houses of today are not meetingthe needs of the users, nor are they performing as good "global" citizens. Applying industrial engineering principles to the housing delivery process could potentially offer solutions to directly solve these problems. History however, shows us that the houses constructed in this fashion, from the panelized houses of Konrad Wachsmann and Walter Gropius to Operation Breakthrough and HUD-code homes, have not been the solution to our housing needs. While these historic efforts introduced new technologies and processes, they were not fully integrated into the structure of the domestic homebuilding industry. Japan and the European Community, faced with higher energy costs and high density housing conditions, have been leading the way globally with innovative ideas and financial incentives to produce better housing. The concepts of "low–", "zero–" and "plus–" house are well known and describe the annual energy balance for homes with respect to the grid. This session will speak directly to our future housing needs and will illustrate potential design, technology and construction process solutions using the 2007 Carnegie Mellon Solar Decathlon house as a case study.

4:00 PM Invited
Biomaterials: Evolving Technologies and Applications: *Arthur Coury*[1]; [1]Genzyme Corporation

Biomaterials, the components of medical devices that interact directly with tissue, have enabled the development of a worldwide industry of over $200 billion. From its infancy a half-century ago, the device industry has evolved from the use of commercially available commodity materials for mainly structural applications to custom-designed materials with specific properties and biological interactions. Accompanying this transition, the complexity and functionality of device designs have increased to include products such as device-drug, device-biologic and device-cell combinations. Therapeutic goals have increasingly evolved from replacement of structure and function of body tissues to their restoration and regeneration using principles of tissue engineering, i.e., systematic control of cells, matrices and fluids of the body. By my estimate, some 25% of device and combination products involve tissue engineering. Regenerative and restorative therapies currently involve expensive development and production protocols, but economic efficiencies to come will improve the availability of advanced medical therapies worldwide.

4:20 PM Invited
Recycling Technologies and Environmental Stewardship: *David Spencer*[1]; [1]wTe Corporation

The ever burgeoning quantities of waste that our society produces are a positive sign of economic growth and human wealth. This new world-wide industrial revolution, however, also spawns new challenges to effective environmental stewardship. Historically, these end-of-life products have been wasted — either disposed in landfills or incinerated — often with adverse environmental consequences. Early methods of recycling were incapable of dismantling and recycling manufactured products whereby the inherent value of the processed materials going into the end-product was preserved. Recently, with support from NSF and NIST/ATP, new optoelectronic recycling processes are under development or being piloted to recycle end-of-life goods in a fully automated way. These advanced technologies can run at high capacity and sort a wide array of materials with extreme accuracy yielding high-value recycled raw materials. As a result, a paradigm shift in recycling, with significant consequent benefits to the environment is both possible and probable, along with the generation of still more opportunities for manufacturing new products with much higher quantities of recycled content..

Ultrafine-Grained Materials: Fifth International Symposium: Structure and Evolution

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Yuri Estrin, Monash University and CSIRO Melbourne; Terence Langdon, University of Southern California; Terry Lowe, Los Alamos National Laboratory; Xiaozhou Liao, University of Sydney; Zhiwei Shan, Hysitron Inc; Ruslan Valiev, UFA State Aviation Technical University; Yuntian Zhu, North Carolina State University

Wednesday PM — Room: 395/396
March 12, 2008 — Location: Ernest Morial Convention Center

Session Chairs: Xiaozhou Liao, University of Sydney; Terry McNelley, Naval Postgraduate School; David Morris, Centro Nacional de Investigaciones Metalurgicas, CSIC; Pei-Ling Sun, Feng Chia University

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2:00 PM Invited
Deformation-Induced Grain Growth and Texture Evolution in Nanocrystalline Materials: Li Li[1]; Yandong Wang[2]; Guojiang Fan[1]; Y. L. Yang[2]; N. Jia[2]; Yang Ren[3]; Hahn Choo[1]; *Peter K. Liaw*[1]; [1]University of Tennessee; [2]Northeastern University, School of Materials and Metallurgy; [3]Argonne National Laboratory, X-Ray Science Division

Grain growth was observed recently in nanocrystalline materials (grain size < 100 nm) during the plastic deformation. However, the underlying mechanisms of this deformation-induced grain growth were not well understood. In our

studies, nanocrystalline Ni-Fe alloys with grain size ~ 23nm were investigated after rolling under both room and liquid-nitrogen temperatures. The high-energy x-ray diffraction technique was used to investigate their mechanical deformation behaviors. Compared with as-deposited one, the nanocrystalline Ni-Fe alloy shows great grain growth (up to ~ 50 nm) after rolling at room temperature. Moreover, a strong texture is found to develop in the deformed nanocrystalline materials during rolling. Based on the above experiments and the associated simulations, deformation mechanisms in nanocrystalline material will be discussed in this presentation. This research is suported by NSF-IMI program (DMR-0231320) and NSF-MRI program (DMR-0421219).

2:20 PM
Towards the Basic Rules for Grain Refinement by SPD Processing: *Ruslan Valiev*[1]; [1]Ufa State Aviation Technical University

Intensive investigations of the past 10-15 years oriented at fabrication of bulk nanostructured materials using severe plastic deformation (SPD) processing have made it possible to conclude that grain refinement is determined by three main factors: the SPD technique in use, SPD processing regimes and routes, as well as the nature of the material processed. In the present report, critical processing parameters (accumulative strains, processing temperature and applied pressure) that determine efficiency of grain refinement are investigated, taking the two most popular SPD techniques (namely, equal-channel angular pressing and high-pressure torsion) as an example. The problem of ultimate microstructure refinement by SPD processing is considered as well.

2:35 PM Invited
High Pressure Torsion in Mg: Effect of Strain and Hydrostatic Pressure on Texture Evolution: Bartlomiej Bonarski[1]; Erhard Schafler[1]; Borys Mikulowski[2]; *Michael Zehetbauer*[1]; [1]University of Vienna; [2]AGH-University of Science and Technology

Single crystals, and polycrystalline samples of 99.8% Mg with different initial grain size, have been subjected to High Pressure Torsion at RT. The evolution of texture has been investigated as a function of shear strain (up to 120) and of hydrostatic pressure (up to 4 GPa). Apart from a compression component which arises from the early stages of HPT deformation, a clear shear texture evolves. However, at the largest strains investigated, a new texture is observed which exhibits clear evidence for recrystallization. As concerns the texture evolution at higher pressures, the same sequence of textures appears but the onset of recrystallization occurs at lower strains. The results are compared with previous investigations of HPT induced texture evolution in hexagonal and cubic face centered metals, and discussed in terms of c/a ratio and of lattice symmetry, respectively.

2:55 PM
The Limiting Grain Size during Severe Deformation of Dilute Aluminium at Cryogenic Temperatures: *Philip Prangnell*[1]; Yan Huang[1]; [1]University of Manchester

The deformation structures formed in a dilute aluminium alloy have been studied down to liquid nitrogen temperatures, with the aim of investigating the factors that limit the formation of a true nano-scale grain structure. It was found that a steady state was approached, where a minimum grain size was reached irrespective of the temperature. Even at -198°C a nanoscale high angle boundary spacing was only approached in the normal direction. It is shown that the minimum grain size achievable in severe deformation is controlled by a balance between the rate of compression of the HABs and dynamic recovery, with a minor effect of texture and changing the deformation mode. The required boundary migration rate to maintain a constant boundary spacing was found to be far higher than can be justified from conventional diffusion controlled grain growth and at low temperatures, can only be explained by invoking an athermal mechanism.

3:10 PM
Microstructure Refinement in Iron Aluminide by Severe Plastic Deformation: *David Morris*[1]; Ivan Gutierrez-Urrutia[1]; Maria Muñoz-Morris[1]; [1]Centro Nacional de Investigaciones Metalurgicas, CSIC

Microstructural refinement of ductile alloys can be achieved by severe plastic deformation carried out to different strain levels using various processing methods. Changes of microstructure of iron aluminides have been examined after severe plastic defomation, with the extremes of low and high strain levels achieved by heavy rolling and by mechanical milling. Processes such as ECAP provide intermediate strain levels. The deformation substructure obtained during the initial stages of straining is inhomogeneous, showing changes that appear to correspond to different textures or grain orientations. At extremely high strain levels, as the structural scale refines towards the nanoscale, the microstructure becomes homogeneous once again. The relationship between grain orientation, local texture, and the microstructure generated by severe plastic deformation will be examined. These factors play also an important role in determined the way the microstructure coarsens or recrystallizes during annealing after deformation.

3:25 PM Invited
Microstructure and Microhardness of FCC Metals Subjected to Ultra-High Strain Deformation: *Alexander Zhilyaev*[1]; Azat Gimazov[2]; Ádam Révész[3]; Terence Langdon[4]; [1]Centro National de Investigaciones Metallurgicas, Consejo Superior de Investigaciones Científicas; [2]Russian Academy of Sciences, Institute for Metals Superplasticity Problems; [3]Eötvös University; [4]University of Southern California, Los Angeles

Microhardness measurements, TEM and X-ray were used to study the microstructure and tensile properties of highly strained nickel and copper processed from bulk master metal strained to different levels and from electrodeposited, ball milled (BM), rapidly quenched (RQ) ribbons and machined chips. High resolution X-ray showed BM+HPT specimens have the smallest coherent domain size of 9 nm and RQ+HPT possesses the highest dislocation density of 6.7×1015 m-2. A gradual increasing strain in bulk nickel gave increasing dislocation density and decreasing coherent domain size. Microstructure and microhardness of pure copper was compared after ECAP (4 passes, route BC), HPT (P=6 GPa, N=5), machining and their combinations, including machining of ECAP specimens, HPT of ECAP copper and HPT of machining chips. Microstructure, dislocation density and microhardness were evaluated by X-ray, transmission and scanning electron microscopy. The influence of different processing routes is discussed in terms of accumulated strain and microstructure refinement.

3:45 PM Break

4:00 PM Invited
Vacancies by Severe Plastic Deformation and Their Effects in Ultrafine–Grained Al: X. L. Wu[1]; B. Li[2]; *Evan Ma*[3]; [1]Institute of Mechanics; [2]Materials Science; [3]Johns Hopkins University

Severe plastic deformation (SPD) introduces into a metal an extraordinarily high population of vacancies, in addition to dislocations that evolve into grain boundaries. These vacancies also cluster to form nanoscale entities that should have major impact on the microstructure-mechanical property relationship; but this aspect has not received adequate attention so far and remains to be systematically studied. Here we report the size, density, distribution, and stability of vacancy clusters in aluminum after SPD-processing at liquid nitrogen temperature. Vacancy-type dislocation loops as large as 16 nm, at a density as high as 1E15/m2, have been observed under transmission electron microscope. Molecular dynamics simulations indicate that such large faulted loops (stacking faults), when at these high densities, contribute in a major way to impeding moving dislocations. These stacking faults should hence be taken into account when interpreting the high strength of ultrafine-grained or nanostructured metals processed by SPD.

4:20 PM
Strain Path and Microstructure Evolution during ECAP of Al-Si Alloys: Juan Maria Garcia de la Infanta Belio[1]; Srinivasan Swaminathan[2]; Alexander Zhilyaev[1]; Fernando Carreño[1]; Oscar Ruano[1]; *Terry McNelley*[2]; [1]Centro Nacional de Investigaciones Metalúrgicas (CENIM), CSIC; [2]Naval Postgraduate School

As-cast Al-Si alloys were subjected to repetitive ECAP at ambient temperature by route A, which involves monotonically increasing strain through a sequence of passes, or by route BC, wherein the strains are redundant after every four passes. The shape change of the primary and eutectic constituents mirror the idealized straining expected for repetitive processing by either of these routes. This leads to strain path dependence of grain refinement in the primary constituent: grain subdivision combined with geometric effects are operative in material processed by route A, while grain subdivision alone predominates in material processed by route BC. Also, the morphology and distribution of the eutectic constituent in processed materials reflect mainly geometrical effects.

4:35 PM Invited
Phase Separation in Ultrafine Grained Materials Produced by Severe Plastic Deformation: *Xavier Sauvage*[1]; Xavier Quelennec[1]; Abdelahad Chbihi[1]; Dmitry Gunderov[2]; François Vurpillot[1]; [1]University of Rouen, GPM; [2]Ufa State Aviation Technical

Ultrafine Grained (UFG) structures produced by Severe Plastic Deformation (SPD) usually exhibit a much higher yield stress than their coarse grained counterparts; however in most cases applications are limited due to the relatively low ductility. It has recently been suggested that a combination of UFG structure with precipitation of a second phase may give rise to some significant strain hardening and ductility. In this context, we have investigated phase separation and precipitation in FeCu, CuCr and FePdAu alloys processed by SPD. Microstructures were characterized thanks to transmission electron microscopy and three-dimensional atom probe. A special emphasis was given on the competition between phase separation and recrystallisation. In such non equilibrium structures, we show that even if the driving force for recrystallisation is extremely high, phase separation usually occurs first. This feature is attributed to an accelerated precipitation kinetics resulting from enhanced atomic mobility of solute elements.

4:55 PM
Grain Refinement Mechanisms and Mechanical Properties in Copper Subjected to Severe Plastic Deformation: *Nairong Tao*[1]; K. Lu[1]; [1]Chinese Academy of Sciences, Institute of Metal Research

The grain refinement in copper subjected to plastic deformation was investigated by means of TEM. Strain-induced three different grain refinement approaches were identified. At low strain rate, plastic deformation is dominated by dislocation activities. Submicro-sized grains were obtained via formation and development of dislocation cells. The final size of the refined grain is usually 100-300 nm. At high strain rate, mechanical twins were induced and deformation twinning plays an important role in grain refinement process. The structure refinement was achieved by the break-up of twin/matrix lamellae. The average transverse size of refined grains is about 47 nm. Nano-sized grains were also formed inside the shear bands. The nanostructured Cu sample prepared at cryogenic temperatures and high strain rate exhibits tensile yield strength of over 600 MPa, evidently higher than that of the Cu prepared at room temperature and low strain rate (~ 400 MPa).

5:10 PM Invited
Gradient Multiscale Structure and Tensile Property of Cobalt by Surface Mechanical Attrition Treatment: *Xiao-Lei Wu*[1]; [1]Chinese Academy of Sciences, Institute of Mechanics

The formation of the gradient multiscale structure (GMS) in hcp-cobalt during surface mechanical attrition treatment was investigated. The GMS showed grain size distribution ranging from 25 nm to 500 nm in the deformed surface. The mechanism of grain refinement was interpreted in terms of the structural subdivision of grains subjected to plastic deformation. Moreover, plastic deformation induced the phase transformation from hcp into fcc structure. The nanocrystalline fcc phase exhibited also gradient distribution in grain size and volume fraction as well. The tensile properties of GMS cobalt were discussed.

5:30 PM
Irradiation Behavior of Nanostructured Austenitic Steels: *Philippe Pareige*[1]; Auriane Etienne[1]; Bertrand Radiguet[1]; Xavier Sauvage[1]; Ruslan Valiev[1]; [1]Rouen University

The evolution of mechanical properties of austenitic stainless steels in internal structures of pressure water reactor is due to complex intragranular and intergranular induced segregation. An atomic description of the phenomena is needed to increase the level of understanding of the mechanisms at the origin. This means for example that careful examination of grain boundaries is needed and elimination of intragranular segregation would be interesting. In order to bring information, austenitic materials have been nanostructured, annealed and ion irradiated. This allows to investigate a large density of grain boundaries and also to understand the effect of nanograins on the elimination of irradiation point defects. Atomic description of the nanostructure and grain boundaries with Tomographic Atom Probe and TEM will be given. Point defects evolution will be followed with rate theory.

5:45 PM
Deformation Behavior during Tensile Straining of Nano/Ultrafine-Grained Structures Formed by Reversion in Metastable Austenitic Steels: Devesh Misra[1]; *B. Ravi Kumar*[1]; Sashank Naik[1]; Mahesh Somani[2]; Pentti Karjalainen[2]; [1]University of Louisiana at Lafayette; [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.