2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Bulk Nanocrystalline Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Wednesday AM — Room: 3018
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Wonbong Choi, Florida International University; Gregory Thompson, University of Alabama

8:30 AM Invited

Bulk Nanostructured Materials via Severe Plastic Deformation: Issues and Scale up: *Yuntian Zhu*[1]; [1]North Carolina State Univ

Severe plastic deformation (SPD) is an approach that refines the grains and microstructures of metals and alloys via extremely large accumulative plastic strain. The most developed SPD techniques include equal channel angular pressing, accumulative roll bonding, high-pressure torsion, etc. The advantage of SPD techniques is that they can produce bulk nanostructured metals and alloys that are not only large enough for structural applications, but also 100% dense and contamination free. Therefore, the SPD has a great potential for commercial applications. This talk will first present issues on the mechanical properties of SPD-processed materials, especially the ductility, and then discuss technologies for the large-scale production of nanostructured materials via SPD. Potential applications of nanostructured materials produced by SPD will also be discussed.

9:00 AM

Role of Severe Plastic Deformation on the Formation of Nanograins and Nano-Sized Precipitates in an Fe-Ni-Mn Steel: *Mahmoud Nili Ahmadabadi*[1]; Hassan Shirazi[1]; Hadi Ghasemi-Nanesa[1]; Tadashi Furuhara[2]; Behrang Poorganji[2]; Syamak Hossein Nedjad[3]; [1]University of Tehran; [2]Tohoku University; [3]Sahand University of Technology

In this research the effect of sever plastic deformation (SPD) on the formation of nano-scaled grains and precipitation of nano-sized particles which consequently control mechanical properties of Fe-Ni-Mn alloy, was investigated. Fe–Ni–Mn martensitic steels show excellent age hardenability but suffer from embrittlement after aging. Discontinuous coarsening of grain boundary precipitates, resulting in the formation of precipitate free zone (PFZ) along prior austenite grain boundaries, has been found as the main source of embrittlement in previous studies. In this paper, severe plastic deformation has been carried out on Fe-10Ni-7Mn steel to improve its mechanical properties. It is found that substantial improvement of tensile properties in cold-rolled steels occurs at thickness reductions larger than 60% where formation of ultra fine grains is realized. According to TEM observations, formation of nano-scaled grains less than one hundred nanometers along with the copious precipitation of nanometer-sized precipitates take place in the severely-deformed steels.

9:15 AM

Ti-Base Nano-/Ultrafine Eutectic Composites: Microstructure and Deformation: Jayanta Das[1]; *Jürgen Eckert*[1]; [1]IFW Dresden

High strength Ti-Fe-Sn nano-/ultrafine eutectic composites have been prepared through arc melting and cold crucible casting. The microstructure consists of a two phase nano-/ultrafine eutectic comprised of FeTi (Pm3m, B2) and ß-Ti (I m3m, A2) phases. The influence of alloying, i.e., addition of Sn, to the Ti70.5Fe29.5 eutectic is assessed in terms of the microstructure variations such as the change of eutectic spacing, morphology, cell size and the resulting mechanical properties in terms of strength and plasticity. The mechanical properties (maximum strength, σm=1939 MPa, fracture strain, εf =13.5%) of the ternary Ti-Fe-Sn are considerably improved compared to the Ti70.5Fe29.5 binary alloy (σm=1733 MPa, εf =3.4%). The change in the morphology of the eutectic, the microstructure refinement, structural fluctuations and supersaturation in the ß-Ti phase, and the elastic properties of nano-phases are crucial factors for improving the plastic deformability of the nano-/ultrafine eutectic alloys without any additional micrometer-size toughening phase.

9:30 AM

A Tough Nanostructured Material: *J. B. Zhang*[1]; A. Y. Chen[2]; H. W. Song[1]; J. Lu[3]; [1]Baosteel Technology Centre, Baoshan Iron and Steel Company, Ltd.; [2]Baosteel Technology Centre, Baoshan Iron and Steel Company, Ltd. - and - School of Materials Science and Engineering, Shanghai Jiao Tong University; [3]The Hong Kong Polytechnic University

Strength and ductility are two key mechanical properties of materials with intense conflictions. We describe a simple and cost-effective way to fabricate this type of material by integrating toughening strategies widely used in ceramics and recently proposed in nanostructured materials. An engineering material stainless steel AISI 304 was selected in this investigation for showing the extensibility of our approach. The surface mechanical attritions treatment (SMAT) is first exerted on the stainless steel sheets for surface nanocrystallization and then the sheet is thinned by warm co-rolling process. This new periodic micro-submicro-nano structured material may reach high strength with exceptional ductility. In contrast to the original counterparts, the yield strength is increased more than twofold with a slightly reducing ductility, and in contrast to the counterparts after work hardening to reach the same strength, the elongation to failure is drastically increased more than threefold.

9:45 AM

Microstructure Evolution of Nano-Structured Bainite Steel during Surface Mechanical Attrition Treatment: Hongyan Li[1]; *Xuejun Jin*[1]; [1]Shanghai Jiao Tong University

Very strong nano-structured bainite has attracted much attention recently for excellent mechanical properties due to the introduction of very thin bainite and films of retained austenite. It is interesting to look into the microstructure evolution of the mixture of such thin bainite and retained austenite under severe plastic deformation, such as the surface mechanical attrition treatment (SMAT). Experimental results show that with increasing the SMATed time retained austenite in the surface layer gradually transforms to martensite under repeated multidirectional loading at high strain rates. Three regions could be identified according to the morphology: nanocrystalline layer, work-hardened layer and the matrix. A distinct boundary between nanocrystalline layer and work-hardened layer was identified, while no visible boundary was observed between work-hardened layer and matrix material. The grain refinement process involves formation of large laths a phase in the work-hardened layer, and formation of randomly orientated equiaxed nanocrystallines in the nanocrystalline layer.

10:00 AM Break

10:15 AM Invited

Nanocrystalline Soft Magnets: Microstructure and Magnetic Properties: *Matthew Willard*[1]; Maria Daniil[1]; Michael Rawlings[1]; Keith Knipling[1]; Ramasis Goswami[1]; [1]US Naval Research Laboratory

Soft magnetic materials consisting of nanocrystallites surrounded by a residual amorphous matrix provide excellent properties, including both low coercivity and high magnetization. They are produced by rapid solidification processing with devitrification by isothermal annealing. Ultimately, to obtain the highest permeability and lowest core losses, the microstructure must be successfully optimized, with grain diameters less than 10 nm and retained amorphous matrix. Typically, this microstructure is developed during an isothermal anneal between 450 and 650°C. This study will examine the influence of composition, kinetics, and microstructure on the magnetic properties of (Fe,Co,Ni)-Zr-B-Cu alloys. Differential thermal analysis, thermomagnetic analysis, x-ray diffraction, and transmission electron microscopy will be used to describe the phase transformations and the resulting structure/property relationships.

10:45 AM

Energy Efficient Magnetic Nanomaterials: *Raju Ramanujan*[1]; S. Bhame[1]; S. Viswanathan[1]; P. Deheri[1]; S. Shukla[1]; Y. Liu[1]; J. Law[1]; Z. Liu[2]; [1]Nanyang Technological University; [2] South China University of Technology

There is an urgent need for energy efficient devices to mitigate climate change and to reduce energy consumption. Nanostructured magnetic materials are being intensively studied for energy efficient permanent magnet systems and novel solid state cooling devices. Ongoing studies on giant energy product magnetic nanomaterials and high temperature magnetocaloric materials will be described.

Melt spun rapidly solidified nanocrystalline RE-TM-B (RE=Nd, Pr, Dy, TM=Fe, Co) alloys with enhanced hard magnetic properties were studied, composition and microstructure dependent elevated temperature magnetic properties were investigated. Reducing grain size and Co or Dy substitution had a significant beneficial effect on thermal stability. Energy product greater than 100 kJ/m3 was obtained in nanophase alloys, attractive low values of temperature coefficients of remanence and coercivity were realized in exchange coupled nanocomposites. Synthesis of rapidly solidified Fe based magnetocaloric materials with high refrigerant capacity was also studied, the magnetocaloric properties and the effect of nanocrystallization were determined.

11:00 AM

Microstructure of Al-Mn in the Nanocrystalline to Amorphous Transition Regime: *Shiyun Ruan*[1]; Christopher Schuh[1]; [1]MIT

We study the microstructure of Al-Mn alloys electrodeposited from a chloroaluminate electrolyte at room temperature. Transmission electron microscopy and x-ray diffraction analyses show that as the Mn content increases across the range 7.5 to 8.0 at%, a single phase crystalline solid solution changes into a two-phase alloy, where a Mn-rich amorphous phase coexists with a Mn-depleted crystalline phase. Concomitant with the appearance of the amorphous phase at 8.0 at% Mn, the crystalline grain size decreases drastically from >1 μm to ~50 nm. Further increase in Mn content results in a further reduction in grain size to ~5 nm and an increase in the amorphous phase fraction. Scanning transmission electron microscopy analysis reveals some detail of the solute distribution in these unique structures, and nanoindentation results show that there is an optimum amorphous/nanocrystalline structure with a hardness exceeding 5 GPa.

11:15 AM

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

Powders of Al-Mg alloys were fabricated by electrodeposition technique under conditions that encouraged dendritic growth. These powders can potentially be used for hydrogen storage applications. XRD analysis revealed that the as-deposited powders consisted of supersaturated fcc-Al(Mg) and/or hcp-Mg(Al). The maximum solubility of Mg in fcc-Al was found to be 20at%, however, up to 40%Al could be dissolved in hcp-Mg. Equilibrium intermetallic phases precipitated upon elevated temperature exposures of the supersaturated phases. The dendrites developed with two distinct morphologies. The feather-like morphology formed at lower Mg concentrations and slower deposition rates. TEM results revealed that the nanocrystalline structure of these dendrites was extensively textured. Majority of dendrites showed a "globular" morphology consisting of spherical units with randomly oriented nanocrystalline grains. In this presentation, the mechanisms responsible for different dendrite morphologies are discussed. The financial support by NSF (grant DMR-0605406) is greatly appreciated.

11:30 AM

Spontaneous Growth of Novel Hexagonal Mn Nanowhiskers from Hydrogen Activated Laves Phase Alloys: *Erdong Wu*[1]; Xiumei Guo[1]; [1]Chinese Academy of Sciences

The spontaneous growth of metal whiskers is a well-established phenomenon. Owing to its significant importance either as a hidden peril for electronic devices or as a potential fabrication technique for complex microstructures, the phenomenon has been extensively studied for decades. However, only the whiskers of soft metals with relatively low melting points, such as Sn, Cd, Zn and In, and primarily on a micrometer diameter scale, can spontaneously grow at room temperature. With the aid of activation of repeated cycles of hydrogenation/dehydrogenation, the crystalline whiskers of transition metal Mn in the shape of nanorod can segregate and grow spontaneously from the crystals of Zr1-xTixMnCr Laves phase alloys at room temperature. Moreover, the Mn atoms in the nanowhiskers form a novel hexagonal structured allotrope. The morphology and structure of the Mn nanowhiskers are exhibited, and the mechanism and potential of the phenomenon are discussed.

11:45 AM

Surface Oxide Selectivity of Nanostructured CoNiCrAlY and NiCoCrAlY Materials: *Dominic Mercier*[1]; George Kim[2]; Mathieu Brochu[1]; [1]McGill University; [2]Perpetual Technologies Inc.

MCrAlYs are used for high temperature application because of their excellent hot oxidation resistance provided by the Cr2O3 oxide layers that develops on the surface. Nanostructured coatings, such as NiCrAlY, are known to promote the formation of the more stable Al2O3, which increases the oxidation resistance, when compared to conventional NiCrAlY. This work was aimed at investigating this phenomenon on other nanostructured MCrAlY systems, namely CoNiCrAlY and NiCoCrAlY. The nanostructured powders were fabricated using the cryomilling technique. The free-standing coatings, obtained by HVOF, were isothermally oxidized in air at 1000°C for 24, 48, 96 and 192 hours in order to monitor the oxide scale evolution. The phase analysis was carried out by XRD, SEM and TEM. The results show that the nanostructured coatings form a more continuous a-Al2O3 layer with very small amount of Cr2O3 and no mixed oxides compared to that of the conventional powders.

Alumina and Bauxite: Methods - Bauxite Characterization, Bayer Chemistry, Alumina Quality

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

Program Organizers: Everett Phillips, Nalco Co; Sringeri Chandrashekar, Dubai Aluminum Co

Wednesday AM — Room: 2002
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: David Kirkpatrick, Gramercy Alumina LLC

8:30 AM Introductory Comments

8:35 AM

Characterisation of Iron Mineralogy in Jamaican Bauxite and Associated Aspects of Alumina and Soda Losses: *Luke Kirwan*[1]; Desmond Lawson[2]; Ab Rijkeboer[3]; Kieran Hodnett[4]; Austin Mooney[2]; Radcliffe Walker[2]; Keddon Powell[4]; [1]Aughinish Alumina Ltd; [2]Windalco; [3]Rinalco B.V.; [4]University of Limerick

The characterisation of bauxite ores, in particular the iron mineralogy, is critical when determining their processability. In this study, various Jamaican bauxite ores have been characterised by Rietveld X-ray powder diffraction (XRD) and Mössbauer spectroscopy. The only forms of iron minerals found are crystalline hematite and crystalline alumino-goethite of relatively small crystallite size. Within digestion, the apatite structure formed is a sodium carbonate hydroxyapatite, and hence a source of soda loss. No evidence of boehmite reversion was found. Post-digestion, the specific surface area of the bauxite residue is strongly correlated with the goethite content, attributed to a decreased particle size, rather than variations in porosity or density. Possibilities for remediation have focussed on the transformation of goethite to hematite, with thermal treatment proving to be most encouraging to date.

9:00 AM

Technological Characterization of Bauxite from Pará-Brazil: *Fernanda Silva*[1]; Francisco Garrido[2]; João Sampaio[3]; Marta Medeiros[2]; Rachel Santos[1]; Manuel Carneiro[3]; Lucimar S. Costa[3]; [1]IQ-UFRJ/CETEM; [2]IQ - UFRJ; [3]Centro de Tecnologia Mineral

The bauxite from Pará-Brazil is a mixture of minerals where the most important are: gibbsite ($Al(OH)_3$), kaolinite ($Al_4[Si_4O_{10}](OH)_8$), quartz, hematite, goethite, rutile and octahedrite. In this work, a bauxite sample from Northeast of Pará-Brazil was crushed and ground in order to get the same size distribution at alumina production industry by Bayer process. After preparation, the sample (90%, < 0.21 mm and 40% < 43 μm) with reactive silica and alumina contents of 5.9 and 47.5%, respectively, was ground with water in a bar mill to 210 min. The ground samples were characterized infrared spectra (IR), X-ray diffraction (XRD), X-ray fluorescence (XRF) and scanning electron microscopy (SEM). Results show that no major bauxite bulk structure modification was observed as a consequence of the grinding process. An analytical method to quantify reactive silica and available alumina contents was also developed. The method is based

on alkaline bauxite digestion, potentiometric titration and atomic absorption spectrometry (F-AAS) techniques. The method accuracy and precision were checked by analysis of the IPT 131 certified bauxite reference.

9:25 AM

Characterisation of Alumina and Soda Losses Associated with the Processing of Goethitic Rich Jamaican Bauxite: *Keddon Powell*[1]; Luke Kirwan[2]; Desmond Lawson[3]; Ab Rijkeboer[4]; Kieran Hodnett[1]; [1]University of Limerick; [2]Aughinish Alumina Limited; [3]Windalco; [4]Rinalco B.V., Netherlands

Iron oxides occur in Jamaican bauxites predominantly in the mineral forms of goethite and hematite. The relative concentrations of these iron minerals, and their morphologies, in conjunction with the soluble phosphate and available alumina content, has a great impact on alumina refinery operations and associated operational costs. Of the iron minerals, goethite in the Bayer process is generally experienced as being adverse, facilitating alumina losses, sequestration of soda, and rendering the mud more difficult to settle. A fundamental knowledge of the components within Jamaican bauxite that contribute to alumina and soda losses is paramount to finding mitigating solutions. This study focuses on the various components of Jamaican bauxite residue material and examines their propensity to promote gibbsite reversion and their soda adsorption capacity.

9:50 AM

Impact of Excess Synthetic Flocculent on Security Filtration: *Jean-Marc Rousseaux*[1]; Pierre Ferland[1]; [1]Rio Tinto Alcan

Synthetic polymers derived from acrylic acid and acrylamide are used in the Bayer process to assist the separation of red mud residues from the liquor. These contemporary flocculants have replaced starch mainly because of their high efficiency found at relatively low dosage. However usage in excess of flocculant can lead to reduced performances in liquor filtration and/or operational problems due to build-up of compacted mud onto the vessel's internals such as the rake. This work outlines the results obtained from an intensive investigation carried out in one of RTA alumina refineries after the commissioning of the new high rate decanter and security filter facilities. The impact of carried-over flocculent and other operating parameters such as filteraid (TCA) dosage and total suspended solids (TSS) on liquor filterability have been evaluated and are discussed. The paper also includes the basis of a laboratory method to quantify flocculent in Bayer liquor at sub ppm level.

10:15 AM

The Effects of Temperature, Hydrate Solids Concentration and Particle Size on Clarity in Laboratory Settling Tests: *Scott Moffatt*[1]; Francis Bruey[1]; [1]Cytec Industries

An experiment with a full factorial design was carried out to estimate the effects of controlled changes in liquor temperature, solids concentration and particle size distribution on supernatant clarity in laboratory-scale settling tests, when the liquor is tested "as is" and after treatment with a flocculant. The experiment was carried out on two separate occasions to provide some information on the magnitude of experimental error in the test setup. Each of the factors had a statistically significant main effect on clarity when varied over a reasonable range such as could be encountered in practice; there were also some statistically significant interactions among the factors. Effect magnitudes and confidence intervals are reported, and the implications of the findings on the conduct and interpretation of laboratory testing and on the assessment of the performance of a plant scale hydrate classification circuit are discussed.

10:40 AM Concluding Comments

Aluminum Alloys: Fabrication, Characterization and Applications: Materials Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizers: Weimin Yin, Williams Advanced Materials; Subodh Das, Phinix LLC; Zhengdong Long, Kaiser Aluminum Company

Wednesday AM Room: 2004
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Sooho Kim, General Motors

8:30 AM

Influence of Heat Treatment on Low-Cycle Fatigue Behavior of an Extruded 6063 Aluminum Alloy: Lijia Chen[1]; Chunyan Ma[1]; Yuxing Tian[1]; Xin Che[1]; *Peter Liaw*[1]; [1]Shenyang University of Technology

Low-cycle fatigue studies were performed under the total strain-amplitude-controlled mode for the extruded 6063 aluminum alloys with different heat-treatment states. The influence of heat treatment on the fatigue behavior of the alloy was determined. The experimental results show that the alloys with different heat-treatment conditions exhibit cyclic hardening, softening and stability. The solution plus aging treatment can increase the fatigue life of the alloy, while the solution treatment leads to a decrease in the fatigue life of the alloy. For the as-extruded 6063 alloy, a single-slope linear relation between the elastic-strain amplitude, or the plastic-strain amplitude, and reversals to failure is observed. However, for the extruded 6063 alloys subjected to both solution and solution plus aging treatments, the single-slope linear relation between the elastic-strain amplitude and reversals to failure is noted while a two-slope linear relation between the plastic-strain amplitude and reversals to failure is noted.

8:50 AM

Coarsening Kinetics of Al-Li Alloys: *Ben Pletcher*[1]; Martin Glicksman[1]; Kegang Wang[2]; [1]University of Florida; [2]Florida Institute of Technology

Phase coarsening in overaged Al-Li alloys is a diffusion-controlled process. Large particles grow by dissolution and mass transfer from smaller particles. Four binary Al-Li alloys were aged for times between 3-240 h at 225C, to yield various distributions of d' (Al3Li) precipitates. Transmission electron microscopy was used to image 10-100 nm diameter, spherical d' 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. TEM results are compared with small angle X-ray scattering analysis. Results, including the particle size distribution and maximum particle size, agree with predictions from diffusion screening theory and a multi-particle diffusion model.

9:10 AM

Multiple Scale FEM Simulation of Deformation and Damage of an Aluminum Alloy Sheet: *Yansheng Liu*[1]; Xiyu Wen[2]; Randall Bowers[1]; Zhengdong Long[2]; Shridas Ningileri[1]; Subodh Das[3]; [1]SECAT Inc; [2]Center for Aluminum Technology, University of Kentucky; [3]Phinix LLC

Particles have significant influence on the formability of aluminum alloys. It is difficult to directly integrate the effect of particles into FEM model. In the current investigation, particle distribution in micro-scale was determined by optical microscope on multiple locations on the surface and along thickness direction of sheet metal. Material heterogeneity properties were derived based on micro-scale analysis and assembled as a macro-scale model to simulate deformation and damage. The result was used to explain some failure examples from industry operation.

9:30 AM

Precipitation under Cyclic Strain in Solution-Treated Al-4wt%Cu I: Mechanical Behavior: *Adam Farrow*[1]; Campbell Laird[2]; [1]Los Alamos National Laboratory; [2]University of Pennsylvania

Solution-treated Al-4wt%Cu was strain-cycled at ambient temperature and above, and the precipitation and deformation behaviors investigated by TEM. Anomalously rapid growth of precipitates appears to have been facilitated by a vacancy super-saturation generated by cyclic strain and the presence of a continually refreshed dislocation density to provide heterogeneous nucleation

sites. Texture effects as characterized by EBSD appear to be responsible for latent hardening in specimens tested at room temperature, with increasing temperatures leading to a gradual hardening throughout life due to precipitation. Specimens exhibiting rapid precipitation hardening appear to show a greater effect of texture due to the increased stress required to cut precipitates in specimens machined from rolled plate at an angle corresponding to a lower averaged Schmid factor. The accelerated formation of grain boundary precipitates appears partially responsible for rapid inter-granular fatigue failure at elevated temperatures, producing fatigue striations and ductile dimples coexistent on the fracture surface.

9:50 AM

Precipitation under Cyclic Strain in Solution-Treated Al-4wt%Cu II: Precipitation Behavior: *Adam Farrow*[1]; Campbell Laird[2]; [1]Los Alamos National Laboratory; [2]University of Pennsylvania

Solution-treated Al-4wt%Cu was strain-cycled at ambient temperature and above, and the precipitation behavior investigated by TEM. In the temperature range 100C to 200C, precipitation of theta-double-prime appears to have been suppressed, and precipitation of theta-prime promoted. Anomalously rapid growth of precipitates appears to have been facilitated by a vacancy super-saturation generated by cyclic strain, with a diminishing effect observed at higher temperatures due to the recovery of non-equilibrium vacancy concentrations. The theta-prime precipitates generated under cyclic strain are considerably smaller and more finely dispersed than those typically produced via quench-aging due to their heterogeneous nucleation on dislocations, and possess a low aspect ratio and rounded edges of the broad faces, due to the introduction of ledges into the growing precipitates by dislocation cutting. Frequency effects indicate that dislocation motion, rather than the extremely small precipitate size, is responsible for the observed reduction in aspect ratio.

10:10 AM

A Continuous Cast AA2037 Al Alloy with Excellent High Cycle Fatigue Properties: *Qiang Zeng*[1]; T. Zhai[1]; X. Y. Wen[1]; Z. Li[2]; [1]University of Kentucky; [2]Aleris International, Inc.

A continuous cast AA2037 Al-Cu alloy was precipitation heat treated at 470°C for 24 hrs before the final peak-aging. It was found that the alloy exhibited a fatigue strength of 210 MP, much higher than that (175 MP) of the same alloy peak-aged using a conventional age-hardening method for an Al-Cu alloy, while their tensile strengths were comparable, though the elongation was somewhat inferior to that of the conventionally peak-aged alloy. The superior high cycle fatigue strength of the alloy was likely to be due to the combination of precipitation hardening predominantly by T (Al20Cu2Mn3) phase and grain refinement by fast heating in salt-bath before the final peak-aging process. The results from this study indicate that it is advantageous to produce Al-Cu alloys with the continuous cast technology over the direct chill cast method, because of the high level of solid solution in the continuous cast Al hot band.

10:30 AM Break

10:45 AM

Alloy Preparation Improvements at Alumar: *Fernanda Silva*[1]; Jarbas Feitosa[1]; Affonso Bizon[1]; Sebastião Silva[1]; Cristino Campos[1]; [1]ALUMAR Consortium

To support Alumar's strategy of increase aluminum alloys production, process of preparing Al-Si alloy was investigated aiming for a higher efficiency on achieving the chemical composition, (measured as percentage of charges where the right chemical composition was obtained in first sample), lower master alloys consumption and reduction on furnace turnaround. The substitution of AlSr10% for AlSr15% on preparation of Al-Si modified alloys and tests regarding the use of cage for master alloy addition were discussed. Therefore, silicon pre-heating was evaluated aiming on a molten aluminum temperature loss reduction and consequently decrease on average furnace turnaround. The usage of copper scrap from anodes on Al-Cu alloys was also investigated showing considerable gains. This paper discusses the issues associated with "First Sample on Grade", and "Furnace Turnaround", for AlSi Alloy preparation, and examines the benefits that may be realized through a different approach to alloying, pre-heating process and standard preparation practice change.

11:05 AM

Effect of Calcium on the Microstructure in Al-Si Alloys: Prediction of the Formation and Identification of Phases by EBSD: *Antonio Zaldivar-Cadena*[1]; Alfredo Flores-Valdés[2]; Francois Brisset[3]; [1]Universidad Autónoma de Nuevo León; [2]CINVESTAV-IPN Unidad Saltillo; [3]CNRS – ONERA

Al–7Si–3Cu–Fe, Al–7Si–3Cu–Fe–0.5Mn, A319, A380 and Al-12Si-Mg-Ni-Cu-0.2Fe alloys containing 0.0020, 0.0040, 0.0080, 0.1 and 0.2 wt.% Ca were used in this research to study the effects of Ca additions on their microstructures and Brinell hardness. The Ca-containing A319 alloy was artificially aged using the T6 thermal treatment to verify the effect of calcium on the response of Al-Si alloys during heat treatment. Samples from the molten alloys were characterized by XRD, SEM/EDS and EBSD analysis. The path of phase formation was predicted using Thermo-Calc® software and validated with experimental results provided from Thermal Analysis DTA runs and microstructural characterization of the samples. Compounds which contain calcium-rich particles were consistent with that of the hexagonal CaAl2Si2 intermetallic phase in all of the alloys used. Finally, it was found that Ca additions refined the eutectic silicon and coarsened the iron-rich intermetallics.

Aluminum Reduction Technology: New Pot Technology and Pot Start-Up

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

Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Wednesday AM — Room: 2012
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Ketil Rye, Elkem Aluminium ANS

8:30 AM

Start-up of New Generation SY350/SY400 Pot: Kangjian Sun[1]; Xiaodong Yang[1]; Yafeng Liu[1]; *Jiaming Zhu*[1]; [1]ShenYang Aluminium and Magnesium Institute (SAMI)

SAMI has designed a number of SY350/SY400 potlines in China in the last 6 years. One of them is the CHALCO Lanzhou branch smelter. Four out of six sections of the SY350 potline at Lanzhou smelter were started up in mid-2007, with the remaining two sections a year later using an improvised start-up method. Several more SY350/SY400 potlines will start-up before the end of this year. This paper will introduce the pre-heat and start-up method used at Lanzhou smelter, which will also be used to start-up other SY350/400 potlines.

8:50 AM

The Hamburg Smelter – A Study of the Cathode Performance: *Till Reek*[1]; [1]Trimet Aluminum AG

In December 2005 the Hamburg aluminum smelter was shut down. In 2006, Trimet Aluminum AG was able to purchase the assets and successfully restarted the potlines in 2007. In December 2007 the plant was operating at full capacity again, after facing serious supply limitation of lining materials throughout the year. To be able to reach full production at record time, unproven Ukrainian and Chinese cathodes had to be acquired. Until August 2008 45 pots with Ukrainian cathodes and 46 pots with Chinese cathodes were started. In addition to these, 149 old linings that had been idle for more than 1 year were restarted. This paper highlights operational experience with these different groups of pots and describes the success story of the first year of operation after the restart without losing a single pot.

9:10 AM

Spent Si_3N_4 Bonded SiC Sidelining Materials in Aluminium Electrolysis Cell: *Zhaohui Wang*[1]; Egil Skybakmoen[2]; Tor Grande[1]; [1]Norwegian University of Science and Technology; [2]SINTEF

Si_3N_4 bonded SiC sidelining materials with different ages in operation have been analyzed by X-ray diffraction, electron microscopy and chemical analysis. The chemical degradation of the upper and the lower part of the sidelining has been demonstrated to be substantially different. The upper part of the sidelining has been subjected to oxidation by the pot gas and the main degradation product

found was Si_2ON_2. The lower part was infiltrated by Na(g) diffusing from the carbon cathode resulting in formation of Na_2SiO_3 as the main oxidation product. Chemical reactions are proposed based on the experimental findings. The diffusion of the degradation species into the side lining has been modeled by a finite element model. The degradation overtime results in the change of the thermal conductivity of the sidelining materials.

9:30 AM
2008: A Milestone in the Development of the DX Technology: B. Kakkar[1]; *Marc de Zelicourt*[1]; Abdulla Zarouni[1]; Abdulla Kalban[1]; Maryam Al-Jallaf[1]; Ibrahim Baggash[1]; Kamel Alaswad[1]; [1]Dubal

From September to December 2005, five prototype DX Reduction Cells were commissioned at the Jebel Ali smelter. They were progressively brought from 325 kA to 345 kA with a current efficiency greater than 96%. In 2008, the DX technology entered the industrial phase of its history with the commissioning of the first commercial Potline of 40 improved DX Reduction Cells from February end to April. The start-up at 340 kA went very smoothly. The pots reached soon 350 kA with excellent operating parameters. The results achieved so far show that there is a potential for further improvement. Meanwhile, in Abu Dhabi, the steady progress of the construction of the 700 kt EMAL phase 1 smelter takes DUBAL latest technology closer to yet another milestone with its implementation on a very large scale.

9:50 AM Break

10:10 AM
AP50 Performances and New Development: *Ben-Aissa Benkahla*[1]; Oliver Martin[1]; T. Tomasino[1]; [1]Rio Tinto Alcan

After 3 years of continuous improvement toward the industrial version which will be implemented in Jonquière (Canada), the AP50 cell has achieved very good technical performances on the LRF platform in Saint Jean de Maurienne (France). The detailed technical results are presented: at the same time, high amperage and low energy consumption have been reached. The reliable ALPSYS control system has demonstrated low Anode Effect rates and tighten thermal control. Pending availability of the future Jonquière development platform, an upgraded new version of the AP50 able to cope with higher amperage has been developed. The results of the first prototype cell are very promising.

10:30 AM
HAL4e – Hydro's New Generation Cell Technology: *Asgeir Bardal*[1]; Christian Droste[1]; Frank Øvstetun[1]; Elin Haugland[1]; Elmar Wedershoven[1]; Morten Liane[1]; Bjørn Erik Aga[1]; Sven Olof Ryman[1]; Albert Berveling[1]; Morten Karlsen[1]; Markus Fechner[1]; Tor Helge Vee[1]; [1]Hydro Aluminium Metal

HAL4e is Hydro's new generation cell technology. The first pilot cells have operated at 420 kA at the Årdal Test Centre since the summer of 2008. The paper covers selected topics of economics (capex and opex), modelling and cell development, early operational experience, as well as elements in the development pipeline, which will be included in future versions of the technology.

10:50 AM
The Advancement of New Generation SY350 Pot: *Zhu Jia Ming*[1]; Yang Xiaodong[1]; Liu YaFeng[1]; Sun KangJian[1]; [1]Shenyang Aluminum and Magnesium Engineering and Research Institute (SAMI)

SAMI's high amperage SY350 pot technology was developed in 2002. Presently, there are three SY350 potlines in operation in China and nine more in design phase. Over the years, SAMI has made further improvement on the SY350 pot technology and has since increase the pot amperage up to 378kA. SAMI has also developed the SY400 pot technology and has installed a total of 16 trial pots at one of SAMI's latest designed potline. The trial pots are currently operating at 402kA and have been in operation successfully for more than 14 months. Good performance obtained from the trial pots with high current efficiency and low energy consumption. Presently, there are two SY400 potlines under design. This paper will introduce the SY350 pot technology and describe some of the key advancements of the technology to date, including aspects of the SY400 trial pots.

Aluminum Reduction Technology: Process Control

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Wednesday AM Room: 2001
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Gary Tarcy, Alcoa Inc

8:30 AM
Correlation between Anode Properties and Cell Performance: *Trond Eirik Jentoftsen*[1]; Hogne Linga[1]; Bjørn Erik Aga[1]; Vidar Geir Christensen[1]; Frode Hoff[1]; Inge Holden[1]; [1]Hydro Aluminium

The performance of a cell operating at high current density depends on heat balance, interpolar distance, adaptive process control system etc. In addition control of the raw materials; alumina, covering material and anodes is necessary. Parallel with current increase Hydro has worked continuously with improving anode quality. To succeed one needs measuring techniques for anode quality which reflect the cell performance. The equivalent temperature, which has been presented earlier, reflects the baking level of the anodes. In addition an internal gravimetric method for measuring the CO2 and air reactivity is applied. Material brushed off after analysis is used together with the weight loss from reactivity to calculate a dust index. Performance data for a modern high amperage cell is presented showing the dependence of current efficiency on equivalent temperature and dust index of anodes. The need for improved anode quality with decreased interpolar distance is also discussed.

8:50 AM
Multivariate Statistical Process Monitoring of Reduction Cells: *Jayson Tessier*[1]; Thomas Zwirz[2]; Gary Tarcy[3]; Richard Manzini[3]; [1]Alcoa Deschambault; [2]Alcoa Inc., Massena West Smelter; [3]Alcoa Inc., Alcoa Technical Center

Modern smelting control systems have large amounts of available data. The information in this data is often underutilized due to inefficient information extraction from databases containing a large number of noisy and highly collinear measurements. The difficulties associated with analyzing such large databases can limit process engineers to analyze a few variables at a time using either univariate control charts or simple x/y correlations to assess the efficiency of reduction cells. Principal Component Analysis has shown the capability to cope with large messy databases (including colinearity, missing data points, and noisy measurements). This technique generates statistics to determine if a pot is in multivariate control by projecting the pot data onto a reference model. It is then possible to extract information that highlights variables with a significant impact on driving the out-of-control pot. Alcoa Inc. is using multivariate Principal Component techniques for potroom problem solving. Several applications will be discussed.

9:10 AM
Development of a Multivariate Process Control Strategy for Aluminium: *Marco Stam*[1]; Mark Taylor[2]; John Chen[2]; Albert Mulder[1]; Renuka Rodrigo[3]; [1]Aluminium Delfzijl B.V.; [2]University of Auckland; [3]Heraeus Electro-Nite LLC.

Process intensification is used worldwide to maximize economic as well as sustainable operation of existing chemical plants. The aluminium reduction process has strong interactive multivariate characteristics with limited process observability and responses which are non-linear and vary over a wide range of time scales. Contrary to application of more compensating single input-single output loops, this paper describes a process control strategy based on passivated responses to common cell behaviours, advanced detection of abnormalities, cause-specific corrective or preventative control actions. Statistical multivariate control surfaces are identified for alumina feed, bath and liquidus temperature measurements. Online root cause analysis and subsequent quality of decision-making have been improved through soft sensors which fingerprint individual failure mechanisms. A module-based approach allows flexible configuration of the overall control philosophy, which has now been tested on industrial

scale resulting in significantly higher current efficiency and reduced energy consumption. These will be discussed in the paper.

9:30 AM

New Feed Control for VSS Side Break Pots: *Nilton Nagem*[1]; Carlos Braga[1]; João Fonseca Neto[2]; Rodrigo Batista[3]; Frenando Costa[1]; Gustavo Andrade[2]; [1]ALUMAR; [2]UFMA - Universidade Federal do Maranhão; [3]ALCOA - Poços de Caldas

Sustainable aluminum production should be archive by reducing green houses gases emissions (GHG) due an anode effect decrease. Reducing anode effect frequency is only possible by improving potrooms operations and controls. A new control design should be develop to improve alumina control. Alumina control for a Vertical Soderberg Side Break is a hard task. The actual feed adjust for the pot is done in a manual daily basis and the feed cycle is every 2 hours, the new control will adjust automatic the amount poured into the pot for each cycle. The algorithm calculates the resistance curvature by a Least Square Regression and a delta from the resistance is calculated too. This information provides the amount of alumina that will be feeding in the next cycle without mucking the pot and avoiding anode effect.

9:50 AM

Controlled Cooling of Aluminium Smelting Cell Sidewalls Using Heat Exchangers Supplied with Air: *Sankar Namboothiri*[1]; Pascal Lavoie[1]; David Cotton[1]; Mark Taylor[1]; [1]Light Metals Research Centre

Aluminium pot shells have increased in temperature and heat flow in the recent years. Removal of heat from cell sidewalls for the purposes of temperature control and ledge maintenance in smelters presently takes the form of compressed air impingement directly on the shell. These air lances cool in a non-uniform way, are extremely energy inefficient, adversely impact on the workplace environment due to the associated noise and dust, and offer no opportunity for energy recovery in the future. The Light Metals Research Centre (LMRC) has developed a technology with the capability of providing controlled cooling to sidewalls using easily installed heat exchangers, with lower air consumption. LMRC has an in-house dedicated testing facility for the development and demonstration of sidewall cooling based on sidewall heat exchangers supplied with air. This paper reports the experimental results obtained in the testing facility and critically analyses the practicality of this technology.

10:10 AM Break

10:30 AM

Challenges in Power Modulation: *David Eisma*[1]; Pretesh Patel[2]; [1]Trimet Aluminium AG; [2]The Light Metals Research Centre, University of Auckland

Due to the increasing power prices and the increase in the spread between hourly power prices, various European smelters have started doing power modulation. Amperage is increased during the usually cheaper night hours, while it is lowered during the day. The maximum leverage for power modulation can be achieved by a constant anode-to-cathode distance (ACD) approach. However, this solution has the biggest negative impact on the cell thermal behaviour. Therefore, it is important to evaluate the effects of extreme scenarios, ranging between a "constant ACD" approach and a "constant heat" approach. Typically, reduction cell operations are tuned to near constant amperage, while the cell voltage is being used to adjust the power input into the cells. No matter what modulation approach is chosen, traditional voltage-based control should be replaced by a purely energy-based control. This paper outlines some of the challenges that TRIMET Essen encountered in this process.

10:50 AM

Electrical Power Availability Optimization at Alcoa Deschambault's Smelter: *Vincent Letellier*[1]; Norman Plante[1]; [1]Alcoa Canada

Today's energy scarcity affects technology and economic choices in most industries. Even if Quebec is a huge producer of hydro-electricity, Alcoa's plants located in this province, are facing the issue. This situation forces optimization of available power in order to increase plants productivity. One way to achieve this goal is to increase power utilization directly at the electrical input of the smelter, allowing an higher operating amperage in the potline which is translated into more aluminum production. Some modifications have been made to the electric control system, at the main substation of the plant. Those modifications allowed Alcoa Deschambault's smelter to use 1.6 MW of power over its previous average consumption, with the same contract allowance. The rebuilt control system results in an utilization factor (percentage of actual power usage over power contract) up to 99.8% on a monthly basis. A significant gain for a very low capital cost!

11:10 AM

Increasing Electrolysis Pot Performances through New Crustbreaking and Feeding Solutions: *Nicolas Dupas*[1]; [1]ECL

Crustbreaking and feeding devices are key elements of the modern aluminium reduction technology. While the crustbreaker's concept has been implemented since the 1970s, its maintainability, reliability and performance monitoring are becoming key subjects for smelters. Increasing cell temperatures and frozen bath crusts put growing strains on the crustbreakers. Their performance is a key factor of the smelter's efficiency and productivity. This is why specific control systems validating the crustbreaking function have been developed. But it is now necessary to go further as the mechanical components of the crustbreakers are submitted to the increasingly aggressive environment of the pot. New functions protecting the crustbreaker's chisel from the acidic attacks of the bath have been developed, also opening new possibilities for pot process control (anode effects frequency control or bath level measurement). The immediate gain is not only on productivity but also on pot operator's health and safety, and environmental impact reduction.

Biological Materials Science: Cell-Biomaterial Interactions

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

Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Wednesday AM — Room: 3014
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: John Nychka, University of Alberta; Devesh Misra, University of Louisiana

8:30 AM Invited

Modulated Delivery of Biomolecules for Manipulating Responses at the Cell-Biomaterial Interface: *David Puleo*[1]; [1]University of Kentucky

The goal of many drug delivery devices has been to obtain zero-order release kinetics. Wound healing, however, is a dynamic process involving numerous biomolecules that trigger a sequence of cellular events, including chemotaxis, proliferation, and differentiation. For example, analysis of growth factor expression in callus during bone fracture healing has revealed a complex sequence of several biomolecules. Therefore, to achieve desired responses at the cell-biomaterial interface, modulated delivery of one or more bioactive agents is expected to generate the greatest effect. To this end, we have been developing different polymeric systems to vary the release profiles of osteotropic (bone-active) molecules. Release periods ranged from days through months, and the molecules delivered ranged from small molecule drugs through plasmid DNA. Results show that cell behavior can be manipulated by modulating the timing of one or more osteotropic biomolecules. Properly designed controlled release devices have the potential to enhance localized tissue repair.

9:00 AM

Cell Behavior on Thickness Graded Polyacrylamide Hydrogels: *James Dahlman*[1]; John Maloney[1]; Krystyn Van Vliet[1]; [1]Massachusetts Institute of Technology

Mechanically compliant substrata provide a model material system for investigating cellular responses to localized mechanical environments. Previous work has demonstrated strong correlations between cellular properties and substrate mechanics by varying properties such as chemical composition and crosslink density to obtain stiffness gradients. Though these approaches are effective, techniques capable of producing mechanical variation without chemical changes are also desired. Here we outline a processing technique by which hydrogelthickness gradients were generated while maintaining constant compositional properties as well as constant biomolecular ligand density. Changes in the thickness of constant-stiffness coatings are predicted to alter the effective stiffness detected by adherent, traction-exerting tissue cells. We evaluate these predictions by quantifying fibroblast cell behavior on thickness-graded polyacrylamide hydrogels.

9:20 AM
Nanograined/Ultrafine-Grained Structures Formed by Phase Reversion in Austenitic Stainless Steel Improves Cellular Activity: *Wah Wah Thein-Han*[1]; Devesh Misra[1]; Mahesh Somani[2]; Pentti Karjalainen[2]; [1]University of Louisiana; [2]University of Oulu

We describe here the combination of fundamental aspects of materials science and engineering with biological sciences in the modulation of cell-substrate response of pre-osteoblasts on ultra-fine grained (UFG)/nanograined (NG) austenitic stainless steels. UFG/NG austenitic stainless steel were processed by a novel procedure involving controlled phase reversion of strain-induced martensite in a cold rolled austenitic stainless steel. The cellular response of UFG/NG austenitic stainless steel is compared with conventional coarse-grained austenitic stainless steel. Interestingly, the proliferation, adhesion, morphology and spread of pre-osteoblasts were significantly different and enhanced on UFG/NG austenitic stainless steel in comparison to conventional coarse-grained austenitic stainless steel. This was implied by cell-density measurements and observations made using fluorescence microscopy and scanning electron microscopy. The improved cellular response ascribed to UFG/NG structures opens up a new avenue for nanostructured materials with combined benefits of biological and mechanical properties such as high strength/weight ratio.

9:40 AM Invited
Biomimetics – Learning from Diamonds: Andrei Sommer[1]; Dan Zhu[1]; Kai Brühne[1]; *Hans Fecht*[1]; [1]Ulm University

There is increasing observational evidence for an implication of the order of interfacial water layers in biology, for instance in processes of cellular recognition and during first contact events, where cells decide upon survival or entering apoptosis. Experimental methods allowing access to the order of interfacial water layers are thus crucial in biomedical engineering. Here we show that interfacial water structures can be nondestructively analysed on nanocrystalline diamond. Results open the gate to a new chapter in the design of biomaterials inspired by biomimetic principles. Recent results on the role of surface modifications and chemical surface termination of nanocrystalline CVD grown diamond layers for biocompatible and biomimetic materials will be discussed.

10:10 AM Break

10:30 AM Invited
Inkjet Printing of Multilayer Structures of Biopolymers and Cells: *Paul Calvert*[1]; Skander Limem[1]; [1]University of Massachusetts, Dartmouth

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.

11:00 AM
Functional Relationship of Collagen Based Biomimetic Composites with Integral Cell Membrane Proteins: Devendra Dubey[1]; *Vikas Tomar*[1]; [1]University of Notre Dame

Integral cell membrane proteins play an important role in structural integrity as well as adhesion properties of eukaryotic cells. An additional function of integral proteins may be the determination of cell response based on interactions with external stimuli from biofunctional materials such as drug delivery nanoparticles etc. In the presented research analyses of the interaction of cell membrane proteins with tropocollagen (COL) based COL-hydroxyapatite (HAP) biomimetic composite structures are presented. The analyses are performed using molecular dynamics (MD) method in a combined quantum mechanical (QM)-molecular mechanics (MM) framework. The focus is on understanding the free energy change and entropic variation based interactions of the nanocomposites with the cell membrane proteins that ultimately lead to variation in cell membrane proteins' adhesion behavior as well as their molecular conformation. Literature experiments related to cell adhesion and protein-protein interactions are also analyzed in light of presented simulation results.

11:20 AM
Interactions of Microbes with Semiconductor Oxides: Adsorption and Membrane Damage: *Qi Li*[1]; P. Wu[1]; J. Shang[1]; [1]University of Illinois at Urbana-Champaign

Microbial interactions with semiconductor oxides were examined on nitrogen-doped titanium oxides (TiONs) by atomic force microscopy, scanning and electron microscopy, fluorescence microscopy, and cell culture analysis. MS-2 virus was found to strongly adsorb onto TiONs and the adsorption was primarily controlled by the electrostatic force between the TiON surface and MS-2 virus. Upon light illumination, charge transfer with water created oxidative radicals on the semiconductor surface. These radicals induced visible damages on the cell membrane in forms of membrane thinning and perforations, leading to cell deaths.

11:40 AM
Quantitative Assessment of Antibacterial and Antifungal Activities of Copper Vermiculite: *Bowen Li*[1]; Jiann-Yang Hwang[1]; Susan Bagley[1]; [1]Michigan Technological University

Copper vermiculite is an excellent antimicrobial agent. To assess the antimicrobial durability of copper vermiculite, the antibacterial and antifungal activities of copper vermiculite against E. coli and Aspergillus niger were quantitatively investigated. The minimum inhibitory concentration of copper vermiculite against E. coli and Aspergillus niger were also determined.

Bulk Metallic Glasses VI: Fatigue and Other Properties

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

Wednesday AM Room: 3007
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Jurgen Eckert, IFW Dresden; Christopher Schuh, MIT

8:30 AM Invited
How to Improve the Deformability of Bulk Metallic Glasses: *Jurgen Eckert*[1]; [1]IFW Dresden

Metallic glasses have mechanical properties that make them attractive candidates for a variety of structural and functional applications. One drawback still limiting such applications is their tendency for shear localization upon deformation. To circumvent such limitations, concepts of creating heterogeneous materials with different type and length-scale of phases have been followed to control the mechanical properties by proper alloy and microstructure design. The recent developments along this line will be summarized and new results for different types of bulk metallic glasses and composites will be presented to illustrate how the mechanical properties can be tuned by appropriate phase and microstructure control. In all these cases the details of the metastable phase formation are closely linked with optimized processing conditions required to form the desired microstructure. The possible mechanisms that govern the deformation behavior will be discussed and linked with the overall plastic deformability and the fracture of the material.

8:45 AM Invited
Optimized Adhesive Bonding to Bulk Metallic Glass Substrates: *Reinhold Dauskardt*[1]; Jeffrey Yang[1]; Mark Oliver[1]; [1]Stanford University

A range of applications of bulk metallic glass alloys involve the need for high-performance adhesive bonds capable of reliably operating in harsh chemical, temperature and loading environments. These bonds cannot be formed with conventional epoxy bonds. We describe a range of strategies for forming strong adhesive bonds using a conditioned oxide on the metallic glass substrate, an optimized sol-gel layer designed to improve adhesion to both the oxide and the metallic glass, and a high-performance epoxy resin. We describe methods to characterize the adhesive properties and also techniques to simulate the metal oxide on silicon wafers so that the subsequent adhesive sol-gel layers and epoxy can be optimized without the need for metallic glass substrates. Adhesive

properties of the joint together with subcritical debonding behavior in moist environments and fatigue loading will be presented.

9:00 AM

Corrosion and Fatigue Behavior of Zr-Based Bulk-Metallic Glasses: *Aaron Wiest*[1]; Gongyao Wang[2]; Marios Demetriou[1]; Peter Liaw[2]; William Johnson[1]; [1]California Institute of Technology; [2]University of Tennessee

The ZrTiBe + Late Transition Metals (LTMs) system was studied for corrosion resistance in chloride-containing acidic environments. The compositional dependence of mass loss versus time in 37% HCl was explored and an empirical parameter based on an average electronegativity was found to adequately correlate the corrosion performance. The best composition was free of LTMs and lost less mass than Ti-6Al-4V or 316L stainless steel. Two alloys with "good" corrosion resistance in 37% HCl were subjected to high cycle fatigue experiments in air and 0.6M NaCl using a compression-compression geometry at 10Hz. An improvement in the corrosion fatigue endurance limit was observed over previously tested Zr-based glasses and this improvement is attributed to the enhanced corrosion resistance of the tested alloys.This work is supported by the NSF International Materials Institutes Program DMR-0231320,with Dr. C Huber as the contract monitor, and by the Office of Naval Research ONR06-251 0566-22

9:10 AM Invited

Cyclic Hardening in Metallic Glasses: *Christopher Schuh*[1]; Corinne Packard[1]; Naser Al-Aqeeli[1]; [1]MIT

This talk will describe our recent systematic nano-scale studies of cyclic loading in metallic glasses, which contribute to a mechanistic understanding of fatigue damage. Using a nanoindenter, we apply load cycles in the elastic regime, and study the effect of their amplitude, rate, and number on the local structure and strength of the glass. In general, we find that such load cycling leads to a shift in the nanoscale strength distribution of the glass, requiring higher loads to initiate a shear band. Our observations also offer some explanation for a number of reported features of glass fatigue, including most notably the fatigue endurance limit and striation spacing. To understand the process of sub-critical cyclic strengthening, we consider the potential for shear transformation zone activation under the point of contact, and specifically along the shear band path. Efforts to directly observe structural changes in cyclically deformed specimens are also discussed.

9:25 AM

Highly Toughened Metallic Glass In-Situ Matrix Composites: *Maximilien Launey*[1]; Douglas Hofmann[2]; Jin-Yoo Suh[2]; William Johnson[2]; Robert Ritchie[1]; [1]Lawrence Berkeley National Laboratory & University of California Berkeley; [2]California Institute of Technology

The potential for catastrophic failure associated with rapid propagation of shear bands is a concern for the utilization of BMGs in structural applications. For more reliable application, shear banding must be controlled using design strategies that involve the introduction of second phases. The fracture and fatigue behavior of a new class of ZrTiNbCuBe BMG matrix composite with in-situ dendritic phase was examined. Semi-solid processing was used to optimize the volume fraction, morphology, and size of dendrites in order to constrain the initial deformation band to the same length scale (~1micron) as the composite microstructure. Toughening mechanisms result in resistance-curve characteristics with a maximum measurable fracture toughness of Kc~145 MPa. m1/2 based on thickness limitations, and a fatigue limit at stress amplitude ~25% of the tensile strength, comparable that of high-strength steel. Such results are considered in the context of understanding the salient mechanisms responsible for the impressive crack arrest capabilities.

9:35 AM

Thermomechanical Behavior of a Cu50Hf41.5Al8.5 Bulk Metallic Glass Following Cyclic Elastic Compression: *Rainer Hebert*[1]; Arif Mubarok[1]; [1]University of Connecticut

Cyclic elastic compression-compression tests have been conducted with amorphous Cu50Hf41.5Al8.5 rods. Test parameters include the strain amplitude, the number of elastic compression cycles, and the frequency. Experimental analyses have focused mainly on differential scanning calorimetry (DSC) and thermomechanical analysis (TMA). Modulated TMA allows for a separation of the reversing true thermal expansion from the non-reversing structural relaxation and viscous effects. Changes in the elastic constants at room temperature following all elastic deformation conditions remain within the uncertainty of the resonant ultrasound measurements. Changes in the isochronal DSC and non-reversing TMA measurements, however, agree with an effect of the cyclic elastic deformation on the amorphous atomic configuration that emulates a sub-Tg annealing. A phenomenological relation between the reduced free volume and the forcing parameters could be established based on the free volume theory. The results indicate that sustained elastic deformation offers a mechanical approach to modifying the amorphous atomic configuration.

9:45 AM Invited

Stress Corrosion and Corrosion Fatigue Crack Growth in Zr-Based Bulk Metallic Glass: *Yoshikazu Nakai*[1]; Yasunori Yoshioka[1]; [1]Kobe University

Crack propagation tests on a Zr-based bulk metallic glass were conducted either in aqueous sodium chloride solutions or high purity water. Crack growth experiments were conducted under cyclic loading at a stress ratio of 0.1 or 0.5 under a loading frequency of 20 or 1.0 Hz. The experiments were also conducted under a sustained load. Although the crack growth rate in high purity water was almost identical to that in air, the rate in NaCl solution was much higher than that in air even in a very low concentration of NaCl such as 0.01%. In 3.5% NaCl solution, the time-based crack propagation rate during cyclic loading, da/dt, was determined by the maximum stress intensity factor, Kmax, but was independent of the loading frequency and the stress ratio, and the rate was almost identical to that of environment-assisted cracking under a sustained load.

10:00 AM Break

10:10 AM

Fatigue Characteristics of Metallic Glass Foam: *Gongyao Wang*[1]; Marios Demetriou[2]; Joe Schramm[2]; Peter Liaw[1]; William Johnson[2]; [1]University of Tennessee; [2]California Institute of Technology

Metallic glasses are able to deform plastically when the specimen dimensions fall below a critical size. Metallic-glasses foams that consist of struts having thicknesses below this critical size demonstrate good plastic deformability and yet retain a considerable fraction of the amorphous metal strength. Metallic-glass foams are presently being considered as energy-absorbing structures to mitigate impact, and as scaffold material for biomedical implants. These considerations require the study of the fatigue behavior. Uniaxial compression-compression fatigue experiments were performed on Pd-based metallic glass foams. Because the strength of foams decreases with increasing porosity, the predicted yield strength is employed to normalize the applied maximum stress. A clear relationship between the ratio of the applied maximum stress to yield strength and the cycles to failure is found, which is similar to the fatigue S (applied stress) - N (lifetime) curves for monolithic BMGs. A mechanistic understanding of the fatigue behavior will be proposed.

10:20 AM Invited

Intrinsic and Extrinsic Factors Affecting the Plasticity/Toughness of Bulk Metallic Glasses: *John Lewandowski*[1]; [1]Case Western Reserve Univ

The flow and fracture behavior of bulk metallic glasses are affected by both intrinsic and extrinsic factors that affect the both the magnitude of mechanical properties as well as the scatter in mechanical properties measured. Examples will be chosen from the authors work as well as those from the literature in order to illustrate some of the various factors that can affect the mechanical properties of these emerging materials.

10:35 AM

Electrochemical Behaviors of Nickel-Based Metallic Glasses in Aqueous Solutions: Lu Huang[1]; Shujie Pang[1]; Ruijuan An[1]; *Peter Liaw*[2]; Tao Zhang[1]; [1]Beihang University; [2]University of Tennessee, Knoxville

Electrochemical behaviors of specific nickel-based metallic glasses including $Ni_{61}Zr_{28}Nb_7Al_4$, $Ni_{40}Cu_5Ti_{6.5}Zr_{28.5}Al_{10}$, $Ni_{59}Zr_{16}Ti_{13}Si_3Sn_2Nb_7$ and $[(Ni_{0.6}Fe_{0.4})_{0.7}{}_5B_{0.2}Si_{0.05}]_{96}Nb_4$ (at.%) were studied in aqueous solutions. Amorphous ribbons were prepared by melt spinning. Potentiodynamic polarization tests and static-immersion tests were performed at room temperature in 0.5M H_2SO_4, 1M HCl, and 3 mass% NaCl. Surface morphologies after immersion tests were observed by the scanning-electron microscopy (SEM). It is found that the $Ni_{59}Zr_{16}Ti_{13}Si_3Sn_2Nb_7$ amorphous alloy exhibited a poor corrosion resistance in different aqueous solutions. Both $Ni_{61}Zr_{28}Nb_7Al_4$ and $Ni_{40}Cu_5Ti_{6.5}Zr_{28.5Al10}$ amorphous alloys exhibited a good corrosion resistance in a 0.5M H_2SO_4 solution, but susceptible to pitting corrosion in 1M HCl, and 3 mass% NaCl due to the existence of chlorine ion. $[(Ni_{0.6}Fe_{0.4})_{0.75}B_{0.2}Si_{0.05}]_{96}Nb_4$ amorphous alloy possesses an excellent corrosion resistance in differnet solutions with wide passive regions and low passive current densities. This work is supported by IGERT,IMI, and NSFC.

10:45 AM

Influence of Laser Melting on Microstructure and Properties of Amorphous Coatings Deposited by High Velocity Oxyfuel Deposition Method: *Greg Kusinski*[1]; Jan Kusinski[2]; [1]Clemson University; [2]AGH

Amorphous coatings (Fe57Cr8Mo12W3C11B11) with thickness varying form 50-450μm were deposited onto 9Cr steel substrates by a high-velocity oxyfuel (HVOF) flame-spraying process. The Nd:YAG laser with a varied output energy was used to remelt the coatings. The microstructure-property relationship (hardness, wear resistance) was studied as a function of laser power and coating thickness. The as-deposited coatings were amorphous (XRD) and were characterized by a high porosity, poor bonding to the substrate and a presence of unmelted, spherical powder particles. The hardness values were ~1350-1450HV for the unmelted particles, ~900-1000HV deformed particles and ~550HV substrate. After laser melting, a completely dense coating layer was formed with a hardness of 1100-1300HV. When the coating was remelted with the substrate, the microstructure (layers were fully crystalline), chemical composition and hardness (lower than 1000HV) of re-crystallized layer was depended on Fe enrichment. However, for all conditions, laser treatment was found to improve the wear resistance.

10:55 AM

Size Effect on the Fatigue Behavior of Bulk Metallic Glasses: *Gongyao Wang*[1]; Peter Liaw[1]; Yoshihiko Yokoyama[2]; Akihisa Inoue[2]; [1]University of Tennessee; [2]Tohoku University

Rod $Zr_{50}Cu_{40}Al_{10}$ and $Zr_{50}Cu_{30}Al_{10}Ni_{10}$ (in atomic percent) bulk-metallic glasses (BMGs) were fabricated by arc-melt tilt-casting technique. The X-ray diffraction and DSC results exhibited that these rod specimens were fully amorphous alloys. Four-point-bend fatigue experiments were performed on these zirconium (Zr)-based BMGs with different size in air. The experiments were conducted at a frequency of 10 Hz, using an electrohydraulic machine with a R ratio of 0.1, where $R = \sigma_{min.}/\sigma_{max.}$, $\sigma_{min.}$ and $\sigma_{max.}$ are the applied minimum and maximum stresses, respectively. The fatigue-endurance limits of these larger size samples were higher than those of the smaller size samples. The result suggested that although BMGs with small size exhibited good ductility, the fatigue resistance of BMGs might degrade when the specimen size decreases. A mechanistic understanding of the fatigue behavior of these Zr-based BMGs is suggested. The present work is supported by the National Science Foundation (NSF).

11:05 AM

Crystallization Phenomena in Novel Ti-Based Bulk Metallic Glasses: *Hesham Khalifa*[1]; Kenneth Vecchio[1]; [1]UC-San Diego

A new glass forming alloy system with excellent thermal stability is introduced of the form Ti-Ni-Cu-Si-Sn. Low density Ti-based bulk metallic glasses have potential as implant materials in biomedical applications. The development of Ti-based BMG composites permits the modulus to be tuned to match that of the bones into which they may be implanted. A series of isothermal and continuous heating experiments were performed to probe nucleation kinetics. Devitrification of the glassy state, and alternative cooling rate-controlled processing from the molten state were used to identify nucleating phases and investigate the role played by microstructure on mechanical properties in BMG composites. Under certain, cooling rate controlled, processing conditions, Ti-Ni-Cu dendrites lead to marked enhancements in mechanical properties. An understanding of nucleation kinetics, phase formation, and microstructural evolution of new metallic glass forming alloys are critical for the advancement of low cost, bioimplantable BMG composites.

11:15 AM

A Study on Mg Flakes in MgCuYZn Bulk Metallic Glass Composite: *Lalu Robin*[1]; Michael Ferry[1]; Greig Kurniawan[1]; Kevin Laws[1]; [1]University of New South Wales

The formation and distribution of Mg flakes in the composites, Mg65+x (Cu0.667Y0.333)30-x Zn5 (12, 14 and 16) were found to be dependent on composition and cooling rate by casting. Higher cooling rate (i.e. the faster the composites solidifies) and lower concentration of the compositions produce less Mg flakes and less likely for crystallisation and morphology change to occur. This was evidenced by the lower volume fraction near the core of the sample. In addition, there is a thickness difference of Mg flakes between in the centre and at the edge of the sample. Furthermore, crystallisation formation was observed in larger compositions (i.e. 14 and 16). More Mg flakes appears during annealing.

Cast Shop for Aluminum Production: Molten Metal Cleanliness

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Pierre Le Brun, Alcan CRV; Hussain Alali, Aluminium Bahrain

Wednesday AM Room: 2005
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Edward Williams, Alcoa Inc

8:30 AM Introductory Comments

8:35 AM Keynote

Improvements in the Molten Metal Process Chain in the Cast House Based on Modeling - Achievements so Far and Challenges Left: *Gerd Ulrich Gruen*[1]; Andreas Buchholz[1]; [1]Hydro Aluminium Deutschland GmbH

Steadily growing demands in productivity and final product quality together with rising prices for energy and basic materials require optimum process conditions within the whole cast house. Although numerical modelling support to the related mandatory continuous improvement of the DC casting process is known for quite some time, the simulation of critical steps in the prior molten metal processing within the cast house is a more recent development. This contribution will give a brief summary of the actual status of activities with respect to molten metal processing including application examples within the area of transport phenomena in the furnace, filtration and launder design. The achieved results and the related conclusions will be used to pinpoint the additional benefits of modelling of related processes. Eventually, still existing challenges for a complete modelling coverage of the molten metal process chain are pointed out and correspondingly necessary future developments are briefly discussed.

9:05 AM

Aluminium Flotation in Stirred Reactor: A Mathematical Model and a Computer Simulation Coupling CFD and Population Balance: *Olivier Mirgaux*[1]; Jean-Pierre Bellot[1]; Emmanuel Waz[2]; Denis Ablitzer[1]; [1]LSG2M - University of Nancy; [2]Alcan Centre de Recherches de Voreppe

Removing inclusions from molten aluminium by flotation in stirred reactors is widely used in liquid aluminium treatments. This process consists in gas injection into the liquid bulk using an impeller: inclusions are attached to the bubbles during their ascension in the liquid bulk and are released in the dross layer. With the aim both of a better understanding of the physical phenomena acting during flotation and of the optimization of the refining process, a mathematical model of the behaviour of the inclusions population has been built up. Transport phenomena, agglomeration of inclusions, sedimentation and flotation are investigated and modelled. The simulation couples Population Balance with convective transport of the inclusions, in the diphasic flow field, in order to predict the evolution of the size distribution of inclusions as a function of time. A laboratory scaled flotation vessel has been modelled and results of a 2D simulation are presented.

9:25 AM

Development and Practical Performance Characteristics of a New Impeller for Metal Treatment in Casting/Holding Furnaces: *Bernd Prillhofer*[1]; Holm Böttcher[2]; Helmut Antrekowitsch[1]; [1]University of Leoben; [2]AMAG Casting GmbH

There are several criteria which characterize melt cleanliness, e.g. hydrogen, alkali metal and the inclusion content. According to the increasing quality demand of materials for high end applications, melt cleanliness has to reach a certain level before starting to cast. Therefore metal treatment has also to be done already in the casting furnaces. Besides adding refining agents like fluxes, a gas purging treatment with an impeller is one of the efficient ways to increase metal cleanliness. Due to of the fact, that commercial rotor types are mostly developed for small vessels, they do not operate well in furnaces. Because of this, impellers used in furnaces have to be custom-made. This paper presents a new impeller for a 33 metric ton channel induction furnace. Based on CFD-calculations a new geometry was developed and tested. The performance behavior regarding inclusion, hydrogen and alkaline metal of all impeller types will be discussed.

9:45 AM

Operational Experience with a Large Capacity Integrated TAC (Treatment of Aluminium in a Crucible) and a Skimmer: Bruno Maltais[1]; *Dominique Prive*[1]; Ahmed Al Hashimi[2]; [1]Société des Technologies de l'Aluminium du Saguenay Inc. (STAS Inc.); [2]Aluminium Bahrain

This paper will present a description of the TAC/Skimmer (Treatment of Aluminium in Crucible/Skimmer), the different layouts that are available and the latest operational results on sodium removal from a TAC user in the Middle East. Needs for the TAC technology have been increasing in aluminium smelters because of higher amperages in the pot rooms, where sodium levels could now exceed up to 200 ppm. With its chlorine-free technology using AlF3 as a reacting agent, the TAC has proven to give process efficiencies in excess of 90% removal, and alkaline levels as low as 1 or 2 ppm can now be achieved. There have been significant improvements in the TAC design over the last couple of years, and one of them has led to the use of the TAC system in conjunction with an automatic skimming system which is used to remove bath before the TAC treatment aswell as before the metal is transferred to the casthouse.

10:05 AM Break

10:25 AM

Comments on the Capture Mechanisms and Surface Forces Acting during Liquid Aluminum Depth Filtration: *Hervé Duval*[1]; Véronique Ghetta[2]; Emilie Laé[3]; Nathalie Ruscassier[1]; Jean Trubuil[1]; Franz Wheling[1]; Jean-Bernard Guillot[1]; [1]Ecole Centrale Paris; [2]LPSC; [3]Alcan CRV

Only a few studies dealing with adhesion forces between inclusions and filter inner walls are reported in the literature. Said studies, which are essentially theoretical ones, predict values of adhesion force which are several orders of magnitude larger than the drag force exerted by the fluid on the inclusions. These predictions are in contradiction with the release of inclusions which can be observed during liquid aluminium depth filtration. In order to understand the discrepancies between the theoretical findings and the industrial observations, a research program combining experiments and theoretical calculations has been presently set up. This paper presents the experimental results of said research program and further develop a detailed discussion around the following points: coexistence of two different populations of inclusions in the melt, massive ones and oxide skin pieces, experimental estimation of the adhesion force magnitude and comparison with recent theoretical calculations, chemical effect of the AT5B addition.

10:45 AM

Wetting of Pure Aluminium on Filter Materials Graphite, AlF_3 and Al_2O_3: *Sarina Bao*[1]; Anne Kvithyld[2]; Sean Gaal[2]; Thorvald Engh[1]; Merete Tangstad[1]; [1]Norwegian University of Science & Technology; [2]SINTEF

The wettability of pure aluminium on filter materials is believed to be an important factor affecting the filtration of aluminium. The contact angle of molten aluminium on graphite, alumina and AlF_3 has been measured in 1 atmosphere of argon with an oxygen partial pressure of about 10^{-17} Pa in the temperature range of 1000-1800ºC. Improved techniques to remove the aluminium oxide layer from a molten aluminium drop have been employed in a horizontal graphite tube furnace. The wettability of aluminium on graphite, alumina and AlF_3 has been discussed and compared with the aim to obtain "new" and more effective filter materials.

11:05 AM

Trial Results with an Improved System of Filtration of Moltem Aluminium Based on a Three Stage Reactor Employing a Cyclone as Its Final Stage: *John Courtenay*[1]; Frank Reusch[2]; [1]MQP Limited; [2]Drache Umwelttechnik GmbH

The development of a new prototype multi stage filter was described at TMS 2008 in which a ceramic foam filter was applied in a first chamber operating in cake mode; grain refiner added in a second chamber and a cyclone deployed in a final chamber to ensure removal of any oxides or agglomerates arising from the grain refiner addition or release events from the foam filter. The first industrial prototype was installed at Trimet Aluminium at Essen in Germany in February 2008 and demonstrated that liquid metal could pass through the cyclone successfully without excessive turbulence or splash. The results of further refinement of the prototype and the initial trial results with respect to inclusion removal efficiencies and operational performance are reported.

11:25 AM

Removal of Iron and Manganese in Aluminium Alloys by Adding Magnesium and Subsequent Centrifuging: *Christian Simensen*[1]; Pierre Le Brun[2]; [1]SINTEF Group; [2]Alcan CRV

The content of Fe and Mn is critical for the final properties of several alloys. The feasibility of the removal of Fe and Mn by primary crystals precipitation has been studied. Different amount of magnesium was added to Al-1%Mn-1%Fe-1%Si melts. The content was varied from 0 to 17 wt% Mg. Then the melts were homogenized and slowly cooled to a temperature about 10°C above the liquidus temperature of Al-crystals and held for four hours. A series of intermetallic particles was formed during this treatment. These particles were separated from the melt by the subsequent centrifuging of the molten metal. Microprobe analysis of the particles showed large particles of α-Al(Mn,Fe)Si, Al6(Mn,Fe), ϕ-(Al,Si)10(Fe,Mn)3 and Al3(Fe,Mn) of size 0.04-0.4 mm. The content of Mn and Fe in the purified metal was correspondingly reduced to 0.10 wt%Mn and 0.13 wt%Fe (X-ray fluorescence measurements) in metal containing more than 12 wt%Mg.

11:45 AM

Molten Metal Quality and Productivity Improvements by Process Optimization in Continuous Casting: *Volker Ohm*[1]; Peter Bauer[2]; Stefan Schormann[1]; Guido Jerusalem[1]; [1]HOESCH Metallurgie GmbH; [2]Friedrich von Neumann GmbH

Process optimization is of increasing importance in order to improve quality, increase production capacity and minimize overall costs for wrought and cast aluminum alloys. The following paper describes a procedure for process optimization in a medium sized casting operation. The first step was to carry out a detailed plant audit of the production process, raw materials, melt treatment efficiency and temperature profiles from furnace to caster. These measurements were carried out with the Prefil® Footprinter melt cleanliness analyzer and with the AlSCAN™ Hydrogen analyzer. Based on the results of this detailed audit the second step was to develop recommendations to optimize the production process. The third and final step was to implement the changes and measure the achieved results against the original targets. In this case study the original targets were to improve quality regarding the hydrogen and inclusion levels to enable more demanding end quality markets to be targeted and to increase daily output by at least 15% to improve overall plant profitability. Both objectives were achieved through the process optimization approach.

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

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

Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Wednesday AM Room: 3009
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Jiann-Yang Hwang, Michigan Technological University; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

8:30 AM

Characterization of Copper Oxides by EDS-SEM Depth Profiling, EIS and XRD: *David Cocke*[1]; Eric Peterson[1]; Jewel Gomes[1]; Dan Rutman[1]; Morgan Reed[1]; Mohammad Akhtar Hossain[2]; [1]Lamar University; [2]Texas A&M University

Study of surface and subsurface interfacial chemistry of metallic and similar systems has its enormous technological importance. Copper, for example, is a common component of many alloys and bulk amorphous systems. It is being increasingly studied today because of its use for electronic interconnect systems where processing is done below 200 ºC. Since several types of copper oxides exist, and the degree of their formation is dependent on temperature, perhaps characterization by EDS-SEM depth profiling and EIS might delineate the structural and stoichiometric differences among them. In this paper, we described our work on transformation of copper oxides with temperature ranging from 70 to 350 °C and their characterization by SEM, EDS, and XRD.

We also showed the use of the phase angle portion of the impedance as a means to identify the predominant structure and the transition through Cu3O2 between the predominant stable CuO and Cu2O.

8:45 AM

Characterizing the Interfacial Properties of SiO2/Si and Hf-Based/Si Gate Stacks: *S.Y. Tan*[1]; Ming-Yuan Wu[1]; Hsing-Hung Chen[1]; [1]Chinese Culture University

As CMOS devices are scaled down into nano-region, SiO2 dielectric is approaching its physical and electrical limits. High-k materials are recently employed by exploiting the increased physical thickness at the same equivalent oxide thickness. HfO2 amd HfSiO were prepared by MOCVD for gate dielectric, we investigated crystal structures properties and thermal stability of materials at various PDA temperatures. The electrical characteristics of HfSiO thin films were explored in comparison with HfO2. In addition to surface treatment could decrease the interfacial trapping density and the incorporation of N could increase dielectric constant and HfO2 film. The XRD was utilized to analyze crystallization of the thin films, and the XPS was applied for surface chemical bounding energy to identify the silicon and dielectric layers. Surface roughness was detected by using AFM. As results, the interfacial layer was grown during annealing processes, this phenomenon lead to capacitance of device decreasing and hysteresis of C-V, oppositely, HfSiO films show the superior performance on thermal stability and electron properties.

9:00 AM

Determination of Low Atomic Number Elements Using SDD in Portable XRF Instrumentation: Alexander Seyfarth[1]; *John Patterson*[1]; [1]Bruker AXS Inc.

In recent years great advances have been made in the capability of portable XRF instruments. This paper will discuss the latest advance – the incorporation of Silicon Drift Detectors. This detector technology provides improved resolution and extremely high count rate capabilities and better sensitivity for low atomic number elements. These capabilities allow the accurate determination of low atomic number elements like Mg, Al, Si, P and S in essentially all alloy families with no special atmosphere as well as the complete analyses of aluminum and titanium alloys not available with current detector technology. The current state of these analyses will be presented.

9:15 AM

A TEM Study of the Transient Stage Scale on the Fe-22wt%Cr Alloy with Reactive Elements Addition: *Jingxi Zhu*[1]; Laura Fernández Díaz[1]; G. Holcomb[2]; P. Jablonski[2]; D. Alman[2]; Sridhar Seetharaman[1]; [1]Carnegie Mellon University; [2]National Energy Technology Laboratory

The chromium-oxide scales formed on Fe-Cr alloys at high temperatures are considered limited in capability of resisting high-temperature-oxidation. Adding a small amount of RE elements (Y, Ce or La) greatly improves the oxidation resistance of the alloys. Yet the precise roles of these reactive elements in oxidation process and the precise mechanisms by which they are incorporated into the scale are not well-established. One difficulty encountered is the location and form of RE in chromium-oxide scales hasn't been extensively explored. Therefore, this paper aims at characterizing selective sites by TEM where the scale and RE particle are both present. Cross section specimens were made via NOVA600-DualBeam system from a Fe-22wt%Cr alloy with addition of elemental La and Ce. The alloy was oxidized at 800°C in dry air for 15 minutes (transient stage). Based on TEM observations, possible effects of elemental La and Ce and RE-oxide particles on oxidation are elucidated.

9:30 AM

Automatic Rietfeld Refinement Based Quantitative Phase Analysis using TOPAS Combined with Same Sample WD XRF: *Alexander Seyfarth*[1]; Rainer Schmidt[1]; [1]Bruker AXS Inc.

The paper will give an introduction to quantitative phase analysis using the TOPAS software based on the Rietveld method. Case studies using examples from the cement industry and mining industry are shown where the method is used in a QC environment in conjunction with WD XRF. The fundamental parameter approach of the TOPAS software allows the creation of a sturdy "recipe" for the refinement which can be used to automate the analysis and also enables the unattended operation. Combined with automated sample preparation this allows for a fully automated QC operation.

9:45 AM

Thermographic Monitoring of Damage Characterization in Thermosetting Plastic Materials: *Jeongguk Kim*[1]; [1]Korea Railroad Research Institute

The thermosetting plastic materials, which are employed for rail pad in railway application, were selected to characterize the damage evolution during tensile testing. The materials, unsaturated polyester resin, are used to buffer the vibration and impact in rail structure. The materials have to possess enough strength and modulus to endure the instant impact and strength from rails. During tensile testing, a high-speed infrared (IR) camera was used to monitor the fracture behavior and failure mode. After final failure, the SEM microstructural analysis was employed for tensile fractured specimens. The SEM characterization results were comparable with in-situ monitored IR camera analysis results. In this investigation, an IR camera and SEM characterization were used to facilitate a better understanding of damage evolution and failure mode of thermosetting plastic materials during monotonic loadings.

10:00 AM Break

10:20 AM

Structural Change of Mesoporous Carbons Studied in an In-Situ Transmission Electron Microscopy: *Dafei Kang*[1]; Mark Aindow[2]; [1]Michigan State University; [2]University of Connecticut

Mesoporous carbons, also known as carbon aerogels, have been studied more commonly for their desirable physical properties such as low density/ high porosity, high surface areas and good electrical conductivity, which in combination make this category of materials very promising for applications ranging from catalytic supports to electrodes in fuel cells, just to name a few. Until recently, the structural modification of mesoporous carbons has received little attention, but in this paper the authors would illustrate such modification on nano-meter scale in a transmission electron microscope (a JEOL 2010F running at 200kV) that also doubles as a source of electron irradiation. The finding is that the original highly tortuous microstructure of mesoporous carbons, upon contact with the energetic electrons, would transform into more ordered graphene-like structures that largely adopt the morphology of a nano-meter sized cage.

10:35 AM

Room and Elevated Temperature Validation and Characterization of a Novel Electrothermal Mechanical Tester: Benjamin Peterson[1]; *Peter Collins*[1]; Hamish Fraser[1]; [1]Ohio State University

The Electrothermomechanical Tester (ETMT) was developed by Instron and NPL and uses direct resistive heating of small rectangular geometries approximately 40x2x1 mm in tension at a wide range of temperatures, including room temperature. Direct resistive heating allows for rapid and accurate temperature control. The mechanical properties of various heat treatments of alpha/beta and beta processed Ti64 were compared in conventional tensile testing and an ETMT at several temperatures to determine a correlation between the two methods. Creep tests were also performed in the ETMT and compared with conventional methods. The ETMT is potentially a realistic alternative to traditional tensile testing due to the material and time saving advantages. Digital image correlation is also employed as the strain measurement system and is also used to characterize the local and macro strain distribution due to the inherent temperature variation along the length of the samples. The statistical variation between samples is discussed.

10:50 AM

EBSD Characterization of Hot Deformed Microstructure in a Ti-Modified Austenitic Stainless Steel: *Sumantra Mandal*[1]; P.V. Sivaprasad[1]; Baldev Raj[1]; V. Subramanya Sarma[2]; [1]Indira Gandhi Centre for Atomic Research; [2]IIT Madras

Hot deformed microstructure of a Ti-modified austenitic stainless steel was investigated using electron backscatter diffraction (EBSD). Hot compression tests were conducted in a Gleeble thermo-mechanical simulator at temperatures in the range 1173K-1373K with a strain rate 10 and 100 s-1 to different strains. Microscopic examination of the samples revealed various kinds of deformation patterns inside the deformed grains. These were inferred as the development of geometrically necessary boundaries or formation of parallel set of micro-bands. Recrystallized grains were partitioned from the deformed grains employing grain orientation spread approach. Extent of dynamic recrystallization (DRX) was found to be minimal at 1173K. DRX was predominantly found to happen at and above 1273K which increases with increase in strain. DRX nucleation and development mechanisms were discussed with respect to bulging of the

parent grains and sub-grain rotation. Special emphasize was given on the role of annealing twins on DRX nucleation and subsequent expansion.

11:05 AM

Detection of Hard Alpha Inclusions in a Titanium Alloy by Magnetic Sensing: *Hector Carreon*[1]; [1]UMSNH

This paper presents experimental data for the magnetic field produced by thermoelectric currents around uncracked hard-alpha inclusions in a Ti–6Al–4V specimen under external thermal excitation for different nitrogen content ranging from 1.6% to 5.9%. According to our preliminary results, the magnetic flux density measurements were found to be rather sensitive to changes in nitrogen content with the exception of the intrinsic material background magnetic signal that affected deeply the detectability of inclusions and imperfections in non-contacting thermoelectric measurements. Hopefully, these preliminary results can help identify a non-destructive test method that can detect material inclusions with a level of nitrogen that could be detrimental to Ti–6Al–4V alloy components.

11:20 AM

Development and Characterization Investigations of Mechanically Alloyed W-Ni/TiC Composites: *Selim Coskun*[1]; Mustafa Ovecoglu[1]; Aziz Genc[1]; [1]Istanbul Technical University

In this study, tungsten matrix composites reinforced with 2 wt% TiC particles were mechanically alloyed for 1h, 3h, 6h, 12h and 24h. 1 wt% Ni is used as sintering aid which is added before and after mechanical alloying and the effect of the amount of mechanical alloying on the microstructural, mechanical and sintering properties were investigated. Microstructure and phase characterizations of composite powders and sintered samples were carried out via SEM and XRD analyses. Furthermore, density and hardness measurements of as-consolidated and sintered samples were carried out. The effect of the Ni addition at different times on the sintering properties of the powders was investigated by DSC experiments.

11:35 AM

Effect of Microalloying Elements and Deformation Mechanisms on the TWIP Steels: *Huseyin Aydin*[1]; Havva Kazdal Zeytin[1]; Huseyin Cimenoglu[2]; [1]TUBITAK MRC; [2]Istanbul Technical University

TWIP (Twinning-Induced Plasticity) steels have exceptionally good combinations of strength, ductility and damage tolerance which satisfy the requirements for automotive industries. Firstly, TWIP has the most beneficial effect on the work-hardening. It is believed that deformation twins increase the work-hardening rate by acting as obstacles for gliding dislocations. Moreover, TWIP steels have low to intermediate stacking fault energy and hence undergo extensive mechanical twinning during deformation, which in turn leads a good combination of both strength and ductility. To explain which are the main characteristics and the behavior of the TWIP steels we have been made some investigations and experiments that show the effects of microalloying elements and deformation mechanisms. In this study, three different compositions of TWIP steels have been produced. The microstructure and mechanical properties are characterized by using optical, electron microscopy and mechanical tests.

Computational Thermodynamics and Kinetics: Integrated Thermodynamic and Kinetic Modeling

Sponsored by: The Minerals, Metals and Materials Society, ASM International, 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

Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M

Wednesday AM — Room: 3002
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Yongmei Jin, Texas A & M University

8:30 AM Introductory Comments

8:35 AM

Large-Scale Three-Dimensional Phase Field Simulation of γ'-Rafting and Creep Deformation in Single Crystal Superalloys: *Ning Zhou*[1]; Chen Shen[2]; Michael Mills[1]; Yunzhi Wang[1]; [1]The Ohio State University; [2]GE Global Research

Three-dimensional phase field modeling of coupled γ/γ' microstructural evolution and plastic deformation was carried at two different length scales. The relative contributions from elastic modulus inhomogeneity and γ channel plasticity were first quantified by the dislocation-level simulations, which showed that the latter plays the dominant role in controlling the rafting process. Then micrometer-scale simulations were carried out that takes into account plastic deformation in γ channels described by local channel dislocation densities from individual active slip systems. The rafting kinetics and the corresponding creep deformation were characterized at different values of applied stress, lattice misfit and precipitate volume fraction. The simulation results were compared with available experiment carried out for Ni-Al-Cr and quantitative agreement has been obtained. The models have the ability to make quantitative predictions to γ' rafting and the corresponding creep deformation in new superalloys (such as the Co- and Pt-based alloys) that are currently under development.

8:55 AM

Computational Modeling and Critical Experiment in High Strength and Toughness Stainless Steel Development: *Ning Ma*[1]; Patrick Ray[1]; Michael Schmidt[1]; Hamish Fraser[2]; [1]Carpenter Technology Corp; [2]The Ohio State University

Martensitic precipitation-hardened (PH) stainless steel has high strength and toughness with good levels of resistance to both general corrosion and stress-corrosion crack. The objective of present study is to understand the strengthening mechanisms during aging and make possible a computer model of the age hardening kinetics based on existing theories. The alloy studied is strengthened by two types of precipitates, ordered NiAl and a close packed intermatellic phase. A systematic experimental study has been conducted to understand the precipitation and growth under various aging conditions. The dislocation/particle configurations and interaction subjected to tensile deformation was reported. A mechanistic precipitation and hardening model was developed based on experimental observation. The model predictions were validated against experimental results from various heat treatment schedules. The high resolution characterization in present study is conducted in the Center of Accelerated Maturation of Materials (CAMM) at the Ohio State University under CAMM characterization membership agreement.

9:15 AM

Influence of Elasticity on Phase Diagrams and Microstructures of Binary Alloys Using Monte Carlo, Lattice Statics and Phase Field Methods: Celine Varvenne[1]; *Mathieu Fevre*[1]; Alphonse Finel[1]; Yann Le Bouar[2]; [1]ONERA; [2]CNRS

The knowledge of phases stability as a function of temperature and composition is essential to improve metallic alloy performances. The competition between chemical and elastic effects results in various type of morphologies and microstructural evolutions, which must be taken into account in numerical models. At atomic scale, we use relaxed Monte Carlo simulations with a position

dependent potential to calculate "exact" phase diagrams for different atomic sizes in the case of phase separation and ordering transformations. These results are compared to those obtained with the lattice statics technique, where elastic effects are embedded into an effective Hamiltonian on a rigid lattice. In this case, the simulation boxes are large enough to reproduce microstructures with several billions of atoms. Finally these two methods are compared to a phase field approach, where calculated phase diagrams are used as input parameters and microstructal evolutions at large time and space scales can be investigated.

9:35 AM

PANDAT Software with PanEngine, PanOptimizer and PanPrecipitation for Multi-Component Phase Diagram Calculation and Materials Property Simulation: *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 software package, with PanEngine for thermodynamic calculation, PanOptimizer for model parameter optimization and PanPrecipitation for precipitation simulation, provides an integrated workspace for phase diagram calculation and materials property simulation of multi-component systems based on CALPHAD (CALculation of PHAse Diagram) approach. The simulation results including thermodynamic, kinetic, thermo-physical properties, and microstructure related information, are critically needed in materials design. In addition to the functionalities provided by PANDAT as a stand-alone program, its calculation engines (PanEngine, PanOptimizer and PanPrecipitation) are built as shared libraries. This simplifies their integration with user's code for broader applications in the framework of Integrated Computational Materials Engineering (ICME) such as phase field modeling, microscopic/macroscopic solidification simulation and other applications where phase equilibrium information and thermodynamic/kinetic properties are needed.

9:55 AM

PanROME: A Phase Field Modeling Tool for Practical Applications: *Kaisheng Wu*[1]; Shuanglin Chen[1]; Fan Zhang[1]; Y. Chang[2]; [1]CompuTherm LLC; [2]University of Wisconsin

Phase field modeling has been demonstrating its powerful capability to simulate complicated microstructural evolutions. Unfortunately, attempts for semi-quantitative or quantitative calculations have been limited, especially for multi-component and multiphase systems, due to the difficulties in relating the energy functionals to the thermodynamic properties of the systems, as well as mobilities to kinetic properties of the alloy elements involved. A phase field modeling tool, PanROME (Research Of Microstructure Evolution), has been developed that offers capabilities to overcome these difficulties. By fully integrating with PanEngine, it is able to use the thermodynamic and kinetic databases that are built based on CALPHAD approach, making it suitable for the simulations in multi-component and multiphase systems. The model is based on Kim-Kim-Suzuki model which is able to carry out the simulation in a practical length scale while maintaining a reasonable interfacial energy. Several examples have been shown to demonstrate its functionalities.

10:15 AM Break

10:40 AM

Virtual Dilatometer Curves and Effective Young's Modulus of a 3D Multiphase Structure Calculated by the Phase-Field Method: *Markus Apel*[1]; Stefan Benke[1]; Ingo Steinbach[2]; [1]Access, RWTH-Aachen; [2]ICAMS, Ruhr-University Bochum

The multiphase-field method allows for the calculation of 3D microstructures in multicomponent and multiphase materials. However, for technical applications the microstructure itself is not of primary interest, but material properties like elastic modules, yield strength etc. In this work we will derive effective mechanical properties directly from phase-field calculations coupled to linear elasticity. We simulated the austenite to ferrite transformation in a Fe-C-Mn steel and calculated the response of the two-phase microstructure on an external load. From these calculations the effective Young's modulus can be derived as a function of the phase fractions which is a useful input for finite element calculations on a larger scale. Furthermore, the external volume change caused by the phase transformation was calculated which leads to virtual dilatometer curves. For constant transformation strains, i. e. neglecting thermal and composition dependent lattice expansion, the volume change depends linearly on the ferrite fraction.

11:00 AM

Phase Field Modelling of Slag Solidification: *Jeroen Heulens*[1]; Nele Moelans[1]; Frederik Verhaeghe[1]; Bart Blanpain[1]; Patrick Wollants[1]; [1]Katholieke Universiteit Leuven

Phase field modelling has proven itself very powerful for simulating microstructural evolutions in materials. In this research, a phase field model is employed to simulate the solidification behaviour of slags, i.e. liquid oxide systems from extractive metallurgy. The prediction of the solidified microstructure of slags is of crucial importance for the application of freeze linings and the slag recycling. The main difference with the well established solidification theory of metals is glass formation at low cooling rates and redox reactions occurring, depending on the oxygen presence in the atmosphere. The model is validated against directional solidification experiments in a Bridgman setup because of its well controlled boundary conditions. Currently, this research is focussed on CaO-Al2O3-SiO2 slags because of the fully optimized phase diagram and this slag is furthermore the basis of nearly all metallurgical slags.

11:20 AM

Ordering and Clustering Instabilities in FCC-Based Alloys: Importance of Second Nearest Neighbors: *Nitin Singh*[1]; William Soffa[1]; David Laughlin[2]; [1]University of Virginia; [2]Carnegie Mellon University

In TMS 2008, we called attention to the importance of 2nd nearest neighbor interactions on the ordering and clustering instabilities in FCC-Based alloys. The Bragg-Williams model including the second-, third- etc nearest neighbor interactions are very often used to describe the energetics and kinetics of precipitation of ordered phases in many FCC-based alloys such as Al-Li, Ni-Ti and Ni-Al. In the present set of results, we describe the synergism that exists between the thermodynamic first-order, ordering transition and spinodal decomposition in such alloys also including the occurrence of the so called conditional spinodal. Specific attention is drawn to the influence of 2nd neighbor interactions on the two-phase region in FCC alloys involving the precipitation of an ordered L12 phase within a supersaturated FCC solid solution. The salient features of the generalized Bragg-Williams model applied to FCC alloys (A1-L12) will be further elucidated and compared to the BCC case (A2-B2).

11:40 AM

Thermodynamic Modeling of the Cr-Ir Binary System Using the Cluster/Site Approximation (CSA) Coupling with First-Principles Energetic Calculation: *Chuan Zhang*[1]; Jun Zhu[1]; Dane Morgan[1]; Fan Zhang[2]; Ying Yang[2]; Y. Austin Chang[1]; [1]UW-Madison; [2]CompuTherm LLC

A thermodynamic description of Cr-Ir was developed in this study by combining first-principles calculation with Calphad approach. The zero-kelvin enthalpies of formation of Cr3Ir (A15), e (hcp) as well as the ordered CrxIr1-x face-centered cubic (fcc) L12 compounds at x(Ir)=0.25 and 0.75 and L10 compound at x(Ir)=0.5 were obtained from first-principles calculation. They were used as the initial values for optimizing the Gibbs energies of the corresponding phases in the Cr-Ir system. The cluster/site approximation (CSA) model was employed to model the phases in the fcc family: ordered L12, L10 and disordered A1 (they are also referred to as the three states of fcc phase). The phase boundaries and thermodynamic properties calculated from the current thermodynamic description are in good agreement with the experimental data as well as the first-principles calculation. The calculated fcc phase metalstable phase diagram using current description reasonably describe the order-disorder transition of L12/A1 and L10/A1.

12:00 PM

A First Principles Study of Hydrogen Trapping by Yttrium in Iron: *Sanket Desai*[1]; Neeraj Thirumalai[1]; Peter Gordon[1]; [1]ExxonMobil Research and Engineering

The interaction of hydrogen with alloying elements in steels has been well-explored experimentally by various groups over the years due to its relevance in hydrogen trapping. With advances in first-principles based modeling methods, it is now possible to complement these experimental studies with atomistic insights. In the 1980s, Myers[1,2] et al. reported that yttrium centers in iron can act as strong binding sites that trap hydrogen. In this talk, we use yttrium in iron as a model system to study its interaction with hydrogen from first-principles. Various interstitial positions around yttrium are examined as potential binding sites for hydrogen, and the calculated binding energies are compared with experimental observations of Myers et al.. The models help provide atomistic insights into their experimental observations. References: (1) Myers S.M., et al., Appl. Phys. Letter 37, 168 (1980) (2) Myers S.M., et al., Rev. Mod. Phys. 64, 559 (1992).

Diffusion in Materials for Energy Technologies: Session III

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Nuclear Materials Committee, TMS: Solidification Committee, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Jeffrey LaCombe, University of Nevada, Reno; Yongho Sohn, University of Central Florida; Carelyn Campbell, National Institute of Standards and Technology; Afina Lupulescu, GE; Ji-Cheng Zhao, Ohio State University

Wednesday AM Room: 3006
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Ji-Cheng Zhao, The Ohio State University; Zhili Feng, Oak Ridge National Laboratory

8:30 AM Invited
Challenges Associated with the Global Nuclear Energy Partnership Nuclear Fuels and Structural Materials: *Maria Okuniewski*[1]; Steven Hayes[1]; Dennis Keiser[1]; Yongho Sohn[2]; Yunzhi Wang[3]; John Morral[3]; [1]University of Illinois at Urbana-Champaign; [2]University of Central Florida; [3]Ohio State University

The vision of the Global Nuclear Energy Partnership (GNEP) is to recycle nuclear fuel by utilizing proliferation-resistant technologies to recover more energy and reduce nuclear waste. To accomplish this mission GNEP is currently focused on utilizing sodium fast reactors (SFRs) to transmute actinides. This mission will require fuel compositions significantly different that traditional SFR irradiated to substantially higher burnup levels. Thus, there are a variety of new fuels and materials related issues to be addressed relative to this mission. This talk will focus on the challenges that will be faced in the nuclear fuels and structural materials development, with specific emphasis on the diffusion related problems. Examples of multi-scale modeling in nuclear fuels and structural materials will also be discussed. The objectives of these multi-scale models are to develop comprehensive, science-based, and predictive tools.

9:10 AM Invited
Interdiffusion in U-Pu-Zr Alloys: *Mysore Dayananda*[1]; [1]Purdue Univ

Concentration profiles of solid-solid diffusion couples investigated with (bcc) U-Pu-Zr alloys at 750°C by Petri and Dayananda in the mid-1990s are reanalyzed with the aid of the *Multi*Diflux program developed at Purdue University for the evaluation of ternary interdiffusion coefficients from individual couples over selected composition ranges. The evaluated data on interdiffusion coefficients are utilized for the regeneration of concentration profiles of the diffusion couples. Also, the interdiffusion coefficients are compared with those determined at the common composition of a couple pair with intersecting diffusion paths. The appreciable diffusional interactions among the components exhibited by the relatively large cross coefficients are discussed in the light of uphill diffusion of U and zero-flux plane development for Zr observed in selected couples. Atomic mobilities of the individual components and vacancy wind effects in these alloys are also briefly discussed.

9:50 AM Invited
Interdiffusion Behavior in U-Pu-Zr Alloy Versus Advanced Cladding Steel Couples Annealed at 700°C: *Dennis Keiser*[1]; James Cole[1]; [1]Idaho National Laboratory

The Advanced Fuel Cycle Initiative is responsible for the development of advanced nuclear energy systems. One of these nuclear energy systems is the Sodium Fast Reactor (SFR). To maximize the performance of this type of nuclear reactor, it will be important to improve on the performance of the nuclear fuel, i.e., allow for higher fuel burnup and/or operation of the fuel at higher reactor operating temperatures. In order to investigate the compatibility of U-Pu-Zr alloys with what are considered advanced claddings, diffusion couples have been annealed at 700°C to investigate the development of diffusion structures at relatively high temperatures. This talk will describe the types of phases that develop in the interdiffusion zones, the partitioning behavior of the various constituents between these phases, and whether or not there is evidence of melting within the diffusion structures of couples annealed at the relatively high temperature of 700°C.

10:30 AM Break

10:45 AM
Interdiffusion in U-Mo-X (X = Nb, Ti, Zr) vs. Al Diffusion Couples: *Ashley Ewh*[1]; Emmanuel Perez[1]; Dennis Keiser[2]; Yongho Sohn[1]; [1]University of Central Florida; [2]Idaho National Laboratory

U-Mo alloys are used for metallic fuels in nuclear research and test reactors due to their high uranium density. However, a diffusional interaction occurs between the U-Mo and the Al cladding alloys producing intermetallic compounds, which may have deleterious effects on the performance and service life of the fuels. Ternary U-Mo-X alloys were examined with an aim to reduce the interdiffusion fluxes, and to attenuate this interaction. This study focused on three such ternary alloys whose compositions in wt.% are U-8Mo-3Nb, U-7Mo-3Ti, and U-7Mo-6Zr. Diffusion couples have been assembled between these U-Mo-X alloys and pure Al to examine the effects of the alloying addition on the intermetallic formation. Using optical and scanning electron microscopy, both the thickness and phase constituents of the interaction layers were determined, and compared to previous studies involving binary U-Mo alloys in order to assess the suitability of ternary U-Mo-X alloys as enhanced metallic fuels.

11:10 AM
Interdiffusion and Microstructural Development of U-7Mo, U-10Mo and U-12Mo Alloys in Contact with Al, Al-2Si, Al-5Si, 6061Al and 4043Al Alloys at 550°C: *Emmanuel Perez*[1]; Dennis Keiser[2]; Yongho Sohn[1]; [1]University of Central Florida; [2]Idaho National Laboratory

Interdiffusion and microstructural development in the U-Mo-Al system was examined using solid-to-solid diffusion couples consisting of U-7wt.%Mo, U-10wt.%Mo and U-12wt.%Mo vs. Al, Al-2wt.%Si, Al-5wt.%Si, 6061Al and 4043Al. These diffusion couples were annealed at 550°C for 1, 5 and 20 hours. Electron microscopy and microanalysis were employed to examine the development of a very fine multi-phase intermetallic layer. Gamma-to-alpha polymorphic transformation in the U-Mo alloys accelerated the interdiffusion in some diffusion couples. Diffusion couples with Si containing Al-alloys showed a decrease in the growth of multi-phase intermetallic layer regardless of polymorphic transformation in U-Mo alloys. Effects of composition (e.g., Mo, Si content and trace-element additions) and polymorphic transformation of U-alloy on the overall interdiffusion behavior are discussed.

11:35 AM
Interdiffusion Microstructure of U-Mo vs. Al Diffusion Couples Annealed at 600°C for 24 Hours: *Emmanuel Perez*[1]; Brian Kempshall[1]; Ashley Ewh[1]; Dennis Keiser[2]; Yongho Sohn[1]; [1]University of Central Florida; [2]Idaho National Laboratory

Electron microscopy and microanalysis were carried out for diffusion couples, U-7wt.%Mo, U-10wt.%Mo and U-12wt.%Mo vs. Al annealed at 600°C for 24 hours. While a slight variation in the thickness of the interdiffusion microstructure was observed as a function of Mo concentrations in the U-Mo alloys, all couples exhibited complex and layered multiphase microstructure. Compositional analysis of the interdiffusion microstructure by electron microprobe showed little variation in the average composition throughout. Transmission electron microscopy with electron diffraction was employed to examine the phase constituents and interdiffusion microstructure for the diffusion couple U-10 wt.% Mo vs. Al. Results are discussed with respect to the equilibrium phases, cubic-UAl3, orthorhombic-UAl4, hexagonal-U6Mo4Al43 and diamond cubic-UMo2Al20 on the Al-rich corner of U-Mo-Al system, which were determined experimentally in our previous study. Preliminary diffusion paths were constructed for the U-Mo vs. Al diffusion couples.

12:00 PM
Phase Field Modeling of Microstructure Evolution under Applied Temperature Gradient: *Rashmi Mohanty*[1]; Yongho Sohn[1]; [1]University of Central Florida

When a temperature gradient is applied to an alloy, a concentration gradient can develop and alter the local microstructure of the initially homogeneous alloy. Thermotransport or the Ludwig-Soret effect can be important in many applications including interconnects of electronic circuits, metallic nuclear fuels, gas turbine component, where a larger temperature gradient is imposed for higher efficiency and performance. A diffuse interface model was devised and employed to predict the effect of thermotransport in single-phase and multi-phase alloys of an ideal binary system. Simulation results show that an applied temperature gradient can cause significant redistribution of constituents

and phases in the alloy. The magnitude and the direction of the redistribution depend on the initial composition, the atomic mobility and the heat of transport of the respective elements. In multi-phase alloys, the thermomigration effect can cause the formation of single-element rich phases at the cold and hot ends of the alloy.

Electrode Technology for Aluminum Production: Anode Production Operations - Focus on Baking

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Wednesday AM Room: 2003
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Stephen Lindsay, Alcoa Inc

8:30 AM Introductory Comments

8:35 AM
Anode Baking: The Underestimated Human Aspect: Felix Keller[1]; Werner Fischer[1]; *Peter Sulger*[1]; [1]R&D Carbon Ltd.

As a result of carbon plant auditing, significant differences in the performance of bake furnaces with similar design were identified. Avoidable direct annual losses of one million USD and more per 100.000 tons of baking capacity have been identified. Most of the problems creating financial losses could have been avoided. The key question, therefore, is why were these opportunities not explored? We identified four main reasons creating these losses: 1)No well-defined objectives and goals set. 2)Persisting errors regarding anode baking and misunderstanding of cause-effect chain. 3)Organizational and motivational aspects underestimated. 4)Inadequate data and information presentation preventing optimum decision making. Propositions are made how losses can be avoided and how management can react in order to optimize bake furnace efficiency while minimizing cost.

9:00 AM
A New Concept for Baking Anodes – Initial Full Scale Test Results and Future Potential: *Rick Lazarou*[1]; Wolfgang Leisenberg[2]; William Morgan[3]; Barry Sadler[4]; Kristen Watson[1]; Dennis Schubert[5]; [1]Lazar Anode Technologies; [2]Transtec Consultancy; [3]Century Aluminum – Hawesville Operations; [4]Net Carbon Consulting Pty Ltd; [5]ANH Refractories Co.

The concepts underpinning a new vertical shaft anode baking furnace technology have been proven and design options assessed in plant scale anode trials conducted continuously over several months. Following these successful trials, the consortium backing the furnace development is constructing a demonstration unit, capable of baking large plant anodes. The new furnace design has many inherent advantages over existing ring furnaces, including improved thermal efficiency, much reduced fuel consumption, lower CO2 gas generation, lower capital costs, and a steady state temperature profile that enhances control of anode heat-up rates and gives longer refractory life. These advantages make the technology ideal for baking high density anodes made using high intensity Paste Plant technologies; such anodes can be difficult to bake without cracking in conventional baking furnaces. This paper outlines the furnace design basics, the concept proving trial results, and plans for the construction of the demonstration unit.

9:25 AM
Resistance Heating of Laboratory Scale Prebake Anodes: *Stein Rørvik*[1]; Trygve Foosnæs[2]; Hogne Linga[3]; Arne Petter Ratvik[4]; [1]Sintef Materials and Chemistry; [2]Norwegian University of Science and Technology; [3]Hydro Aluminium; [4]Sintef Materials & Chemistry

Resisitivity heating of prebaked anode samples has been done in a laboratory study. The samples were heated by passing electric current through the anodes in a cold nitrogen atmosphere. No external heating was applied. Temperature measurements were done on the samples using both thermocouples and an infrared camera to examine differences in the temperature distribution during heating. The samples were also electrically heated in an air atmosphere using the same setup to observe the oxidation of the anode surface. Microscopy investigations were done after cooling to look for selective reactivity on the anode surface. The results are discussed with respect to their relevance to anode dusting problems.

9:50 AM Break

10:00 AM
Which Strategy to Use When Sampling Anodes for Coring and Analysis? - Start with How the Data Will Be Used: *Keith Sinclair*[1]; Barry Sadler[2]; [1]Sinclair Associates Inc.; [2]Net Carbon Consulting Pty Ltd.

Most Prebake Anode Aluminium Smelters take and test anode core samples, using the results for a range of purposes, including: ·Characterizing product quality going to the Potrooms; ·Historical anode performance troubleshooting; ·Analysis of raw materials, paste plant, or baking furnace performance issues. To generate the data consistent with each of these purposes, a specific approach to selecting the anodes for coring should be used. Unfortunately, only rarely is serious consideration given to these sampling strategies, and while a number of different approaches are used in the industry, in most cases these are not consistent with the information desired. This can lead to poor decision making and waste. This paper will outline appropriate sampling strategies and discuss the key issues to be considered when designing a sampling strategy appropriate for the intended use of anode core data.

10:25 AM
Simulation-Based Approach for Validating a Lean Anode Plant Configuration: *Robert Baxter*[1]; Trevor Bouk[1]; Laszlo Tikasz[1]; Robert I. McCulloch[1]; [1]Bechtel

Bechtel's Aluminium Center of Excellence (ACE), in the Mining and Metallurgy Business Unit, intensively worked on developing advanced modeling tools for plant design and operation. Today, process modeling and simulation are integral parts of Bechtel Studies and Proposals on smelter projects. The present paper outlines the steps of applying lean techniques as continuous improvement effort to the anode handling/storing process. For the carbon area operation in a recent Study, sector models of anode fabrication, namely anode storage, anode baking, rodding and pallet storage models were linked to cover the overall anode handling process. Simulation scenarios were performed under projected normal and extreme operation conditions. Early findings were fed back to designers and measured and analyzed with lean criteria. Reduced storage spaces and still appropriate anode inventories were targeted and achieved. Results introduced to Client demonstrated robust plant operation and adequate green, baked and rodded anode inventories.

10:50 AM
Environmental Improvements during the Handling of Packing Coke at the Albras' Bake Furnaces: *Paulo Douglas Vasconcelos*[1]; André Mesquita[2]; [1]Albras Alumínio Brasileiro S.A; [2]Federal University of Pará

ALBRAS operates five open ring-type bake furnaces in two plants with a capacity of 285,000ton anodes/year. Each furnace is composed of sections made with six pits separated by partition flue walls through which the furnace is fired. The pits are about four meters deep and accommodate twelve anodes, around which petroleum coke is packed to avoid air oxidation and facilitate the heat transfer. During anode baking, about 20 kg of coke per baked anode are used. The placement and removal of this coke cause significant problems with a high generation of carbon dust causing consequent pollution in the workplace environment. This is a common "nuisance" problem faced by all aluminum smelters, and since 2004 Albras' Carbon Plant Engineering Department has been working on a solution. This paper presents the existing problem at Albras and shows the technical solutions that were implemented in 2006 to resolve the dusting problems.

Emerging Applications of Neutron Scattering in Materials Science and Engineering: Deformation Behaviors

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Xun-li Wang, Oak Ridge National Laboratory; Brent Fultz, California Institute of Technology; Hahn Choo, University of Tennessee

Wednesday AM Room: 3012
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: James Jones, Northern College of Applied Arts and Technology; Sheng Cheng, University of Tennessee

8:30 AM Invited
Neutron Diffraction Study of the Strain Rate Dependent Development of Microstructure in Beryllium: *Donald Brown*[1]; Thomas Sisneros[1]; Bjorn Clausen[1]; Saurabh Kabra[1]; Diana Donati[1]; [1]Los Alamos National Laboratory

Plastic deformation of hexagonal metals such as Be, Mg, and Zr occurs by a mix of slip and twinning mechanisms. Deformation slip and twinning are controlled by different mechanisms at the atomic scale, and thus respond differently to variations in strain rate. In general, deformation twinning is expected to be favored by high strain rate conditions. We have completed neutron diffraction studies of the evolution of the microstructure of strongly textured and random Be as a function of strain rate, from 0.0001/sec to 5000/sec. The yield point is rate insensitive over 7+ orders of magnitude of strain rate. The hardening, however, is strongly rate dependent, due to the increased role of twinning at increased strain rates. We measure texture to characterize the twin volume fraction, lattice strains to determine load partitioning between different grain orientations, and line profiles to monitor the defect characteristics, all as a function of strain rate.

9:00 AM
Fatigue Deformation Mechanism of Nanocrystalline Metals Studied by Neutron and X-Ray Diffraction: *Sheng Cheng*[1]; Peter Liaw[1]; Hahn Choo[1]; Xun-Li Wang[2]; [1]University of Tennessee; [2]Oak Ridge National Laboratory

Grain size of materials has a great influence on their deformation mechanism. It is also true for fatigue deformation. However, the deformation mechanism of nanocrystalline metals under fatigue was inadequately studied. We have recently performed fatigue studies on a range of materials with grain size from nanocrystalline to ultrafine-grained to conventional coarse-grained samples under both tensile and compressive loading modes. We used in situ and ex situ neutron diffraction and synchrotron X-ray diffraction to study the deformation mechanism during fatigue. Insightful information (including intergranular strain, texture evolutions, peak broadening etc) in connection with the microstructural change was examined during fatigue tests. From the critical information, we show distinctive deformation mechanism. These influential mechanisms will be discussed. This work is supported by the NSF International Materials Institutes (IMI) Program (DMR-0231320) and Major Research Instrumentation (MRI) Program (DMR-0421219) with Dr. C. Huber and Dr. C. Bouldin as the Program Directors, respectively.

9:20 AM
Deformation in a Bulk Amorphous Alloy Investigated by Neutron Scattering: *Dong Ma*[1]; A.D. Stoica[1]; X.-L. Wang[1]; Z.P. Lu[2]; D.W. Brown[3]; B. Clausen[3]; Th. Proffen[3]; [1]Oak Ridge National Laboratory; [2]University of Science and Technology of Beijing; [3]Los Alamos National Laboratory

An in-situ neutron scattering study of deformation in a Zr-based bulk amorphous alloy has been carried out on the Spectrometer for Materials Research at Temperature and Stress (SMARTS). By monitoring the evolution of the structure factor in the reciprocal space and representing microscopic strains in a q-dependent manner, our analysis of the diffraction data reveals distinct variation of strains on multiple length scales. This unusual behavior is interpreted as a manifestation of the interplay of two structural units in the amorphous metal, i.e., short-range order (SRO) and medium range order (MRO), in response to the stresses.

9:40 AM Invited
In Situ Studies of Ferroelectrics Using Neutron Diffraction: *Jacob Jones*[1]; [1]University of Florida

Ferroelectric materials are used in a variety of applications including diagnostic and therapeutic ultrasound, sonar, and vibration and displacement sensors. The electromechanical response in ferroelectric materials is comprised of both intrinsic (lattice strain) and extrinsic (e.g., domain switching) components that are expressed as characteristic changes in the diffraction pattern. This talk presents the results of three in situ techniques applied to a soft lead zirconate titanate (PZT) ceramic. First, the lattice strains and domain switching (texture) behavior is measured under uniaxial compressive stress using HIPPO at LANSCE. Next, a stroboscopic technique is applied using the instrument TASS at ANSTO to characterize the domain switching behavior under dynamic electric field loading. Finally, the lattice strains under dynamic electric field loading are characterized using the instrument ENGIN-X at the ISIS facility. These measurements provide a comprehensive picture of the constitute behavior of ferroelectrics. New techniques and instruments will also be reviewed.

10:10 AM Break

10:30 AM
In-Situ Neutron Diffraction Study of Uniaxial Deformation of Nickel Based Anode Materials for Solid Oxide Fuel Cells: *Ke An*[1]; Alexandru Stoica[1]; Bjorn Clausen[2]; Beth Armstrong[1]; Don Brown[2]; Xun-Li Wang[1]; [1]Oak Ridge National Laboratory; [2]Los Alamos National Laboratory

Anode-supported solid oxide fuel cells (SOFCs) allow lower operating temperature due to the substantially lower ohmic resistance of the thin electrolyte. Multi-physical integrity including the structural and mechanical properties plays an important role on the durability and reliability of anode-supported SOFCs performance. Cylindrical NiO-Y2O3 stabilized zirconia (YSZ) and Ni-YSZ specimens with different porosities are investigated by in-situ neutron diffraction under uniaxial loads at room and high temperatures. With in-situ neutron diffraction, the monotonic and creep deformation mechanisms at micro-structural level of each single phase, inter-granular strain/stress evolution between the two phases and the impact of porosity on the response are characterized. Redox effect on the deformation evolution of the Ni-YSZ anode is also investigated by neutron diffraction. The details of the experiments and results of this approach will be discussed.

10:50 AM
Neutron Diffraction Measurements of Residual Stress in an Electron Beam Welded Uranium Tubular: *Thomas Holden*[1]; D.W. Brown[2]; B. Clausen[2]; T. Sisneros[2]; J. Vaja[3]; [1]Northern Stress Technologies; [2]Los Alamos Neutron Science Center, Los Alamos National Laboratory; [3]AWE Aldermaston

Neutron diffraction measurements have been made of the stresses associated with electron-beam welding a uranium tube. The tube exhibits a large grain size and fairly strong crystallographic texture. To mitigate the effect of the large grain size the tube was rotated about its own axis during the measurements so the results obtained are circumferential averages. High hoop stresses (350±100MPa) were found in the center of the weld close to the outside diameter. A strong hoop stress gradient was observed between the outside, OD, and inside, ID, diameters such that hoop compression (-50±100MPa) was observed close to the ID in the weld center. Hoop compression was also observed away from the center of the weld for all through-thickness positions. An axial tensile stress (150±50MPa) was observed near the OD which decreases to zero at the ends of the tubular. There is also a gradient of axial stress from the OD to the ID. The radial stresses were found to be zero to within the experimental uncertainties. The stresses associated with the weld are conventional in form. However, the unequal coefficients of linear expansion, and the anisotropic elastic and plastic response of orthorhombic uranium means that thermal residual strains are expected as well as mechanically-induced type 2 strains in the weld region. The analysis must seek to minimize the effect of these strains on the derived stresses. The presence of strong crystallographic texture is helpful in this case.

11:10 AM
Multistage Deformation in Uranium 6 Wt% Niobium: *Catherine Tupper*[1]; Don Brown[2]; Bjorn Clausen[2]; Robert Field[2]; Dan Thoma[2]; Rajan Vaidyanathan[1]; [1]University of Central Florida; [2]Los Alamos National Laboratory

The uranium-niobium alloy system demonstrates shape memory behavior mechanistically similar to the nickel-titanium system. In both U-Nb and NiTi, the shape memory behavior is governed by the selection of martensitic variants

in the microstructure in response to enforced mechanical strain. Through in-situ neutron diffraction experiments, we have identified a second twin reorientation in uranium niobium in the post shape memory regime (4-8% strain) under tensile loading. The reorientation is identified through the use of inverse pole figures, which can each be calculated from one neutron diffraction pattern. The reflections are about the (010) plane or the (100) plane, and are only made possible by the monoclinic structure of the U6Nb alloy. This twin system would not be available in orthorhombic uranium because in the higher symmetry crystal structure, i.e. the (010) is a mirror plane.

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: Experimental Studies of Initiation and Growth in Structural Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Wednesday AM Room: 3008
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Robert Ritchie, University of California; Brad Boyce, Sandia National Laboratories

8:30 AM Invited
Fatigue Crack Initiation and Propagation Behavior of Forging Die Steels: *Ryuichiro Ebara*[1]; [1]Hiroshima Institute of Technology

In this paper low cycle fatigue,thermal fatigue and gigacycle fatigue behavior of forging die steel is presented. First, the effects of temperature, hardness and surface treatment on low cycle fatigue strength of hot forging die steels are summarized. Fatigue crack initiation and propagation behavior is discussed with fracture surface morphology. Thermal fatigue crack initiation and propagation behavior of hot forging die steel is also presented with respect to fracture surface morphology. Then low cycle fatigue behavior of representative cold forging die steels is summarized. Fatigue crack initiation and propagation behavior of cold forging die steels is discussed with respect to stress concentration factor, hardness and surface roughness. Crack initiation behavior in very high cycle regime is also discussed for cold forging die steels with respect to the effect of surface roughness and heat treatment. Finally recommended study on fatigue of forging die steels is touched on briefly.

9:00 AM
Fatigue Crack Growth in Inhomogeneous Steel Components: *Donato Firrao*[1]; Paolo Matteis[1]; Pasquale Russo Spena[1]; Giovanni Mortarino[1]; [1]Politecnico Di Torino

Massive low-alloy high-strength steel components often exhibit microstructure variations from surface to core due to decreasing quenching rates when moving towards the interior. Since different steel constituents exhibit different Fatigue Crack Growth (FCG) behaviors, both the overall FCG rate and the crack shape are expected to be influenced by the microstructural changes. The case of slack-quenched components with simple geometries, having a surface flaw, and subjected to mode-I constant-force-amplitude fatigue, is first examined theoretically. The microstructural variations are hypothesized by considering medium steel hardenabilities and quench severities; thereafter the FCG is computed by considering (during each integration step) the stress-intensity-factor amplitude and the FCG behavior of different points of the crack front, the pointwise FCG properties being determined by the local steel constituents fractions.Simulation results are compared with experimental evidences from a recent failure occurred in a 90 mm diameter connection rod of a 2500 kW naval diesel generator.

9:20 AM
Determine of Fatigue Fracture Mechanisms and Modeling of P/M Heterogeneous Steels: *Hamid Khorsand*[1]; [1]K.N.Toosi University

Industrial parts manufactured by P/M process have found a wide spread use in a variety of applications so, investigation of mechanical properties and determine of how improvement of them is very important, under different kinds of service conditions. In this research, mechanical properties of several low alloy steels have been studied. Specimens with different chemical compositions, metallurgical phases and densities were prepared. The green compacts were sintered at 1120 c and 1250 c in a H2-N2 atmosphere for 35 minutes to densities in the range of 6.6 to 7.2. The formation of different metallurgical phases in the research samples with different densities was caused that the authors can present equations for prediction of mechanical properties of P/M steel parts.

9:40 AM
Effects of Processing Residual Stresses on Fatigue Crack Growth Behavior of Structural Materials: *Christopher Lammi*[1]; Diana Lados[1]; [1]Worcester Polytechnic Institute

Fatigue crack growth mechanisms of long cracks through fields with high and low residual stresses were investigated for various structural materials commonly used in transportation applications. Macro residual stresses were introduced within each material first by processing methods, and then replicated in both magnitude and distribution, through original mechanical/geometrical techniques. Residual stresses were measured/paired using microstructure-tailored X-ray diffraction techniques. Compact tension specimens were fatigue crack growth tested at room temperature and stress ratio, R=0.1. Residual stress corrections were done using the Restoring Force Model, and the results were compared to those generated by a real-time compliance correction technique. Qualitative and quantitative effects of residual stress on fatigue crack growth characteristics, such as fatigue crack growth threshold and fracture toughness, will be presented and discussed for each material/class. Recommendations are given for fatigue crack growth data collection and interpretation to facilitate consistent and accurate design in the presence of residual stress.

10:00 AM
Nominal and Local Effects of Surface Treatment on Fatigue Variability: *Sushant Jha*[1]; Reji John[2]; Dennis Buchanan[3]; James Larsen[2]; [1]Universal Technology Corp; [2]US Air Force Research Laboratory; [3]University of Dayton Research Institute

Incorporating surface-treatment induced residual stresses (RS) in life prediction has been hampered by the variability in the RS profile and relaxation, and a lack of physics-based measure of the benefit of RS on the lifetime. Towards this, the fatigue variability behavior of the alpha+beta titanium alloy, Ti-6Al-2Sn-4Zr-6Mo, under low stress ground (LSG) and two shot-peening (SP) intensities was studied at 260°C. It was found, that the competing roles of the nominal RS profile and the local, microstructure – surface-treatment-process interaction was the strongest determinant of the lifetime distribution. In particular, the probability of surface vs. subsurface failure, the predominant crack initiation mechanisms, and the corresponding crack-initiation sizes can be effectively understood in terms of these two competing effects. This hypothesis was applied in modeling the influence of the RS level and the variability in the profile on the lifetime distribution and the probabilistic lifetime limit in Ti-6Al-2Sn-4Zr-6Mo.

10:20 AM Break

10:40 AM Invited
Short Crack Effects in Extrinsically Toughened Materials: *Jamie Kruzic*[1]; [1]Oregon State Univ

Extrinsic toughening mechanisms (crack bridging, transformation toughening, etc.) are effective at providing crack propagation resistance in many materials and composites; however, these mechanisms result in a crack size dependence (i.e., "short crack effect") for the fatigue properties over the crack sizes where the extrinsic toughening zone forms and develops. This presentation examines how fatigue resistance curves (fatigue *R*-curve or fatigue threshold *R*-curve) may be used to understand and predict such crack size effects. Experimental results using compact tension, C(T), and/or beam specimens for several bridging materials (human bone, polycrystalline Al_2O_3, Si_3N_4) are presented, along with discussion of the role the material microstructure. Furthermore, it is demonstrated that the fatigue behavior can be predicted by characterizing the bridging zone and quantifying the effects of bridging using crack tip opening displacement, compliance, and/or Raman spectroscopy experiments. Experimentally measured small crack data agrees well with the predictions based on quantitative bridging zone characterization.

11:10 AM
Fatigue Crack Propagation in New Generation Aluminum-Lithium Alloys: *Sébastien Richard*[1]; Christine Sarrazin-Baudoux[1]; Jean Petit[1]; [1]LMPM ENSMA

Aluminium alloys are widely used in aeronautical industry due to their good specific mechanical properties. Low-density third generation aluminum-lithium alloys are good candidates in view of reducing the fuel consumption. However, a better knowledge of damage properties is required for application. To answer this question, a study of fatigue crack growth behavior of three new aluminum lithium alloys in T8 temper was undertaken. Tests were performed at 20 Hz (CCT specimens) and 35 Hz (CT specimens), at stress ratios R=0.1 and 0.7 under three environments: ambient air, simulated high atmosphere (dew point and temperature of 223K) and high vacuum as reference for an inert environment. Crack closure contribution was systematically evaluated. Experimental results are analyzed in terms of da/dN curves, crack path profiles and SEM examinations. Influence of specimen geometry, texture, stress ratio, environment and alloy composition is discussed in comparison with conventional alloys.

11:30 AM
The Influence of Shot Peening on Alpha-Case Formation and Microcracking in Titanium Alloys: *Meurig Thomas*[1]; Trevor Lindley[1]; Martin Jackson[2]; [1]Imperial College London; [2]The University of Sheffield

Exposure of titanium alloys to air at high temperature leads to the formation of an all-alpha, oxygen-rich, embrittled surface layer termed the alpha-case. Whilst the deleterious effects of the alpha-case on component integrity such as fatigue behaviour have been identified, research examining the link between processing, microstructure and the propensity for alpha-case formation is limited. This paper seeks to explore the relationship between alloy chemistry, thermo-mechanical processing and surface treatments, (notably shot peening) and alpha-case growth kinetics. In addition, the metallurgical aspects of failure as a result of the alpha-case induced microcracking and approaches to improve aero-engine component lifing are also discussed.

11:50 AM
Combination of Safe Life and Fail Safe Concepts to Assess the Lifetime of Ti-6Al-4V Forgings: *Bernd Oberwinkler*[1]; Heinz Leitner[1]; Martin Riedler[2]; [1]University of Leoben; [2]Böhler Schmiedetechnik GmbH & Co KG

Forged parts made of Ti-6Al-4V are generally used in aerospace industry, e.g. for engine mounts, pylon fitting and frame parts, housings, gear boxes, engine disks and so on. To achieve damage tolerant together with light weight design of such parts a combination of the safe life and the fail safe concept is necessary. The characterization of the fatigue behavior of Ti-6Al-4V with different microstructures has been done through low cycle and high cycle fatigue tests for different stress ratios and relative stress gradients (notches) as well as crack propagation tests for small and long cracks. Assuming the smallest detectable flaw size using non-destructive testing methods the residual lifetime can be estimated with fracture mechanics. To combine this fail safe concept with the safe life concept the crack initiation has to be taken into account. This approach has been validated using flawed specimens under high cycle fatigue loading.

12:10 PM
Fatigue Crack Growth Behavior of Long and Small Cracks in Structural Materials: *Anastasios Gavras*[1]; Diana Lados[1]; [1]WPI

Fatigue crack propagation of long and small cracks was investigated for various structural materials. For each material, two microstructures were prepared and tested. Low residual stress was ensured during processing to shed light on microstructural effects on crack growth. Compact tension and single edge tension specimens were fatigue crack growth tested at room temperature and stress ratio, R=0.1. Microstructure related mechanisms were used to explain the near-threshold behavior and crack growth responses in Regions II and III for each material/microstructure. Threshold behavior of long cracks is attributed to closure-dependent mechanisms. In Regions II and III, the changes in crack growth mechanisms were explained by the extent of the plastic zone ahead of the crack tip. Threshold behavior of small cracks is explained through closure-independent mechanisms, specifically through the barrier effects of characteristic features specific to each material/microstructure. Recommendations for integrating materials knowledge in structural design for fatigue performance are given.

Friction Stir Welding and Processing-V: Session V

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Wednesday AM — Room: 2014
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: William Arbegast, South Dakota School of Mines and Technology

8:30 AM Invited
Partitioning of Forces in Friction Stir Welding – Part 1: *John Baumann*[1]; Abe Askari[1]; Robert Landers[2]; [1]The Boeing Company; [2]Misouri University of Science and Technology

In the FSW system built by Broetje Automation for The Boeing Company, retractable pin tool capability has been achieved with an arrangement that allows for independent positioning and force measurement / control of the shoulder and pin, along with radial loads and torque measurement / control capabilities. The goal of the work described is to characterize the weld envelopes for two pin-length regimes, to measure the loads and torques under changing process conditions, and to compare this collected data with predicted values from the Boeing-Sandia FSW modeling code, Cth. A previous paper described progress in refining that code, to be able to resolve the output into separate contributions from the shoulder and pin. This paper will cover the data collected on the Broetje system for the shoulder and pin tools, using a Design of Experiments approach, while making welds in 2024 Al, and complete the comparison with the model.

8:50 AM Invited
Boundary Condition Effects on Friction Stir Welds in 7050-T7 Sheet: Piyush Uphadyay[1]; *Anthony Reynolds*[1]; [1]University of South Carolina

A series of friction stir welds was made in 6.4 mm thick 7050-T7 sheet. Thermal boundary conditions for the welds were varied by (1) using tools with different values of thermal diffusivity and (2) by varying the ambient thermal conditions. Tools with shoulders made from Nimonic 105, H13 tool steel, and Densimet (a tungsten based alloy) were used for welding. The thermal conductivities of the various shoulder materials vary by a factor of 12 (minimum for the Nimonic and maximum for the Densimet). Each tool was instrumented with thermocouples in the probe and at various locations in the shoulders and shanks. Welds were performed in room air and underwater and with varying levels of base plate pre-cooling. Weld response variables, hardness distributions, and nugget grain size were correlated with the thermal boundary conditions. Finite element simulations are used to enhance understanding of the observed phenomena.

9:10 AM Invited
Aging Weapons Systems Repair Using Friction Stir Welding: *Bryan Tweedy*[1]; William Arbegast[2]; Robert Hrabe[1]; [1]H. F. Webster Inc.; [2]South Dakota School of Mines and Technology

Friction stir welding and processing (FSW&P) was identified in the FY07 Aging Aircraft Study conducted by the South Dakota School of Mines and Technology as a technology that is ready to enter into a qualification process for use as standard repair technique on aging weapons systems. FSW has been widely investigated as a manufacturing process with successes reported in the commercial and government sectors, however, little is reported in the literature on the qualification of FSW&P for repair applications. Preliminary analysis in this study utilizing FSP for repair of several components showed technical feasibility. In addition, the demonstration of FSP to refurbish an actual part was successful. Radiographic inspection showed that the volumetric defects and fatigue cracking were processed from the candidate component. The study produced a cost benefits analysis which estimated $31.4M annual savings to the USAF alone.

9:30 AM

The Role of Friction Stir Welding in the Nuclear Fuel Plate Fabrication: *Douglas Burkes*[1]; N. Hallinan[1]; Michael Chapple[1]; Jared Wight[1]; Pavel Medvedev[1]; Indrajit Charit[2]; Peter Wells[2]; Amit Amritkar[3]; [1]Idaho National Laboratory; [2]University of Idaho; [3]University of Utah

The friction bonding process combines desirable attributes of both friction stir welding and friction stir processing. The development of the process is spurred on by the need to fabricate thin, high density, reduced enrichment fuel plates for nuclear research reactors. The work seeks to convert research and test reactors currently operating on highly enriched uranium to operate on low enriched uranium without significant loss in reactor performance, safety characteristics, or significant increase in cost. In doing so, the threat of global nuclear material proliferation will be reduced. Feasibility studies performed on the process show that this is a viable option for mass production of plate-type nuclear fuel. Adapting the friction stir weld process for nuclear fuel fabrication has resulted in the development of several unique ideas and observations. Potential areas in the nuclear field where friction stir welding could continue to play a significant role will be discussed.

9:50 AM

Effects of Parts and Fixture Geometric Errors and In-Process Deformations on the Quality of Friction Stir Welds: *Michel Guillot*[1]; Sébastien Bédard[1]; Isabelle Bouchard[1]; [1]Laval University

Although the friction stir technique is increasingly used for welding extrusions, aircraft and automotive components, its implementation is often delayed by a lack of understanding of the parameters involved. Among these parameters, the large forces exerted by the tool can induce deformations in the parts and in the fixture. Furthermore, this process appears to be very sensitive to any geometric error in the welding joint. In this paper, the effect on weld quality of geometric errors and in-process deformations is investigated on typical AL6063-T6 and AL6061-T6 extrusions. Butt and lap joints are produced in 6061-T6 samples using a stiff fixture with controlled geometric deviations and gaps. The effect of in-process deformations on weld quality is quantified. A method for improving fixture designs is proposed. Effect of heat transfer to the fixture is considered. Finally, this method is applied to improve the assembly of floor panels made of several extrusions.

10:10 AM

Friction Stir Welding of a AA2199 Al-Li Alloys: *Axel Steuwer*[1]; Jens Altenkirch[2]; Myriam Dumont[3]; Philip Withers[4]; [1]ESS Scandinavia; [2]Institute Laue-Langevin; [3]Faculté des Sciences et Techniques de St-Jérôme; [4]University of Manchester

In this paper we discuss friction stir welding of a novel low-density aluminium-lithium alloy AA2199, which is intended for use in the aerospace industry. Nine trial welds were produced to study the effect of varying welding parameters on residual stresses and microstructure with a view tooptimising the welding process. The welds were characterised using a variey of techniques such as hardness measurements, SEM, TEM, DSC and SAXS, which allows a discussion of the effect of FSW on the precipitation kinetics, and its effect on residual stress. Additionally the in-situ global mechanical tensioning (GMT) was applied to produce nearly stress free welds in this alloy. Residual stresses of ~50% of the yield strength were found after weld parameter optimization, while the stress engineering technique (GMT) allowed producing stress free welds.

10:30 AM Break

10:40 AM

Corrosion in 2XXX-T8 Aluminum Alloys: *Christian Widener*[1]; Tze Jian Lam[1]; Dwight Burford[1]; [1]Wichita State University

This paper investigates the apparent trend in 2XXX-T8 aluminum alloys to possess excellent as-welded exfoliation corrosion resistance in the weld zone compared to the parent material. To evaluate this trend, friction stir welds were produced in 0.125 (3.2 mm) 2024-T81, 0.080-in. (2 mm) 2219-T87 and 0.153-in. (3.9 mm) 2198-T851 (Al-Li) material, and then tested in a standard and modified ASTM G34 exfoliation environment. Unlike welding in the –T3 or –T4 tempers, where the weld zone can become anodic to the parent metal and exhibit preferential corrosion, when welded in the –T8 starting temper the weld zone has been found to be relatively cathodic compared to the parent material exhibiting only mild evidence of corrosion attack.

11:00 AM

Correlation between Ultrasonic Phased Array and Feedback Force Analysis of Friction Stir Welds: *Pedro Gimenez Britos*[1]; Christian Widener[1]; Dwight Burford[1]; [1]NIAR - WSU

Ultrasonic phased array has been used as a powerful non-destructive test (NDT), well known for its capability to detect different kinds of FSW defects. A new NDT technique developed at the South Dakota School of Mines and Technology is a FSW Analysis Software, designed to analyze any specific section of the weld in almost real time using discrete Fourier transforms and phase space analysis. With this software, a trained user can detect where potential flaws may exist. The purpose of this study is to determine if the defects found using ultrasonic phased array inspection can be correlated with defects identified by the software. By correlating this software with an ultrasonic phased array inspection, the time and expense associated with 100% inspection of parts could be significantly reduced. The ultimate goal of this research is to support the development of real time quality control to minimize the cost of inspection through statistical process control methods.

11:20 AM

A Novel Artificial Neural Network Model for Evaluating Hardness in Stir Zone of Submerge Friction Stir Processed Al-6061-T6 Plate: *Abbas Ebnonnasir*[1]; F. Karimzadeh[1]; M. H. Enayati[1]; [1]Isfahan University of Technology

The aluminum (Al) alloy 6061-T6 was friction stir processed at submerged condition and different tool rotation speeds (w) and processing speeds (V). The effect of processing parameters on hardness of stir zone was investigated. In order to derive out the relationship between the hardness of stir zone and processing parameters and optimizing them, some test was done and a matrix of variation parameters of process was filled and used for training of an artificial neural network (ANN) model. A sensitivity analysis was carried out using the ANN model. It is shown that, among two process parameters, the processing speed (V) is more important on stir hardness. In addition, a safe zone can be defined by ANN model in which superior hardness can be achieved.

11:40 AM

Corrosion and Fatigue Evaluation of Swept Friction Stir Spot Welding through Sealants and Surface Treatments: *Jeremy Brown*[1]; Dwight Burford[1]; Christian Widener[1]; Walter Horn[1]; George Talia[1]; Bryan Tweedy[2]; [1]Wichita State University; [2]H.F. Webster Engineering

This experiment investigates the capability of welding though sealants and surface treatments with swept Friction Stir Spot Welding (FSSW) in thin gauge 2024-T3 aluminum alloy. The aluminum sheets have a sealant applied and are pre-treated with various surface coatings. The uncured sealants were applied to the faying surface of the test coupons shortly before joining. The results are also compared to bare sheets in the untreated condition. Corrosion testing was performed through alternate immersion in a salt solution. The samples were evaluated through metallography and testing of residual strength. Fatigue testing was performed per the NASM 1312-21 specification. S-N data was collected for 5 load levels for each sample type. Riveted data has also been collected using this method. Work in this area is important to support increased implementation of FSSW in production applications as a replacement for other discrete fastening methods, like riveting and resistance spot welding.

General Abstracts: Materials Processing and Manufacturing Division: Session I

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: Surface Engineering Committee

Program Organizers: Thomas Bieler, Michigan State University; Neville Moody, Sandia National Laboratories

Wednesday AM — Room: 3022
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM

Anisotropic Properties of Tantalum Processed by Equal Channel Angular Pressing: *Joel House*[1]; Philip Flater[1]; James O'Brien[2]; William Hosford[3]; John Bingert[4]; Robert De Angelis[5]; [1]US Air Force; [2]O'Brien and Associates; [3]University of Michigan; [4]Los Alamos National Laboratory; [5]University of Florida/REEF

The current study examines the effect of severe plastic deformation on pure tantalum via equal channel angular pressing (ECAP). After processing, three metallurgical conditions were characterized: as worked, fine-grain annealed, and large-grain annealed. A series of low strain rate, split Hopkinson Pressure Bar, and Taylor Impact experiments were conducted to characterize the mechanical properties. These experiments revealed orientation dependence in mechanical strength as a result of the processing history. This paper will describe the initial microstructures to include grain size and texture. Data will be given on the mechanical properties relative to orientation. The recovered specimens were analyzed to provide in-sight into the evolution of texture for the various experimental conditions. These data sets will be discussed in the contexts of processing by severe plastic deformation and the resulting anisotropic material properties.

8:50 AM

Corrosion Performance of Al/SiCP Composites with Multimodal Distribution under Humid Ambient and Aerated Chloride Solutions: Miguel Montoya-Dávila[1]; *Martin Pech-Canul*[1]; Maximo Pech-Canul[2]; [1]Cinvestav-Saltillo; [2]Cinvestav-Merida

The effect of the multimodal distribution on the corrosion behavior of Al/SiCp composites was investigated. Composites with 0.6 volume fraction of reinforcements were prepared by the infiltration of preforms (silica-coated a-SiC powders of 10, 54, 86, and 146 µm) with the alloy Al- 13.3Mg –1.8Si (wt. %) in Ar□N2 at 1100ºC for 60 min. Corrosion potential measurements were carried out in aerated 0.1M NaCl solutions; humidity tests were performed in 90±3% humidity chambers at 50 °C. Results show that weight gain augments with increase in particle size distribution. A possible explanation to this outcome is the formation of the unwanted Al4C3. Despite the weight gain registered, the composites show no evidence of degradation. Corrosion potential curves are characterized by two stages; in stage 1, Ecorr tends towards the corrosion potential for aluminum; in stage 2 and after 4.33 h, Ecorr oscillates within a well defined and approximately constant range.

9:10 AM

Fast Epitaxial High Temperature Brazing of Single Crystalline Nickel Based Superalloys: *Britta Laux*[1]; [1]Technische Universität Braunschweig

A new brazing technology has been developed for the repair of cracks in turbine components. Conventional diffusion bonding technologies work with nickel based braze alloys which are enhanced by fast diffusing melting point depressants (MPD) like boron or silicon. An epitaxial growth can be achieved by a diffusion controlled isothermal solidification. Due to the poor solubility of the MPD within nickel, entire diffusion of the MPD out of the braze gap is essential. Otherwise, brittle secondary phases form which results in deteriorating mechanical properties. Since the required hold times for epitaxial solidification are very long, new manganese containing alloys were developed as manganese is completely solvable within nickel. Brazing times being up to 100-times shorter could be achieved. By the addition of aluminium, chromium and titanium a microstructure very similar to that of the base material was produced over a gap width of 300µm, whereas a complete epitaxial solidification occurred.

9:30 AM

GTAW-Assisted Laser Welding of Galvanized High-Strength Steel in Gap-Free Lap Joint Configuration: *Shanglu Yang*[1]; Radovan Kovacevic[1]; Robert Ruokolainen[2]; [1]Research Center for Advanced Manufacturing; [2]General Motors Corporation

Laser welding assisted by GTAW preheating is introduced for welding of galvanized DP 980 in gap-free lap joint configuration. The controlled heat management during the preheating by GTAW will transform the zinc coating at the top surface into the zinc oxides, which will dramatically improve the coupling of the laser power to the welded material. The keyhole is readily formed with the help of zinc oxides,which allows the high-pressurized zinc vapor to be vented out. The completely defect-free laser welds have been obtained. Furthermore, a CCD video camera is used to on-line monitor the molten pool. By the analysis of the video film, it is revealed that when the welding process is stable, the keyhole is kept open. However, the keyhole is readily collapsed when the welding process is instable. The results from the micro-hardness and shear tensile tests reveal that the high strength is obtained in the laser welds.

9:50 AM

Uniform Metallic Coatings on High Conductivity Graphite Foams: *Ben Poquette*[1]; Stephen Kampe[2]; [1]Keystone Materials LLC; [2]Virginia Tech

In the late 90's, a novel technique for fabricating high conductivity graphite foam was developed by Oak Ridge National Laboratory With its unique properties, this foam has shown promise to revolutionize the performance of many commercial and defense related systems not limited to: high surface area electrodes and catalysts supports, power electronics cooling, personal cooling, evaporative cooling, radiators, nuclear reactor core, space radiator, brake and clutch cooling, high temperature bearings, EMI shielding, thermal and acoustic signature management. Until recently, difficulties in joining graphite foam to other materials have hindered its incorporation into current platforms. A technique was developed, through cooperation with ORNL and Virginia Tech, which allows a strongly adhered, uniform metallic coating to be applied throughout the thickness of graphite foam. These metal coatings should serve to both solve existing short-falls (brittleness, lack of joinability, etc.) as well as lend their properties (magnetic, catalytic, etc.) to graphitic foam.

10:10 AM

Microstructural Evolution during Grain Boundary Engineering of Stainless Steel: *Benjiman Albiston*[1]; Megan Frary[1]; [1]Boise State University

Grain boundary engineering (GBE) is a method for controlling the microstructure to improve the material properties. The purpose of GBE is to reduce the interconnection of general grain boundaries (i.e., those susceptible to intergranular degradation) and to increase the fraction of "special" boundaries (i.e., those resistant to attack). The objective of the present work project is to modify the microstructure of 316L stainless steel using GBE. An iterative processing technique involving cold working and annealing steps was developed and electron backscatter diffraction (EBSD) is used to characterize the resulting microstructures. The special boundary fraction was increased from 50% to 80%, effectivly reducing the size of connected general boundary clusters. The reduced general boundary cluster size makes it more difficult for cracks to propagate through the material. By controling the properties of the grain boundaries in the material, its performance can be enhanced by increasing its lifetime and reliability.

10:30 AM

Modeling Uncertainty Propagation in Deformation Processes: *Nicholas Zabaras*[1]; Babak Kouchmeshky[1]; [1]Cornell University

We will present a non-intrusive method for modeling the propagation of uncertainty in process conditions and initial microstructure on the final product properties and geometry in a deformation process. The stochastic multiscale deformation problem is modeled using a sparse grid collocation approach that allows the utilization of a deterministic simulator to build interpolants of the main solution variables in the stochastic support space. The ability of the method in estimating the statistics of the macro-scale properties such as ductility and hardness of the product of the metal forming process is shown through examples featuring randomness in initial texture and process parameters. Comparisons are made with the results obtained from Monte-Carlo method.

10:50 AM

Phase Segregation in Semisolid Powder Processing of Micro-Features: *Gap-Yong Kim*[1]; Yufeng Wu[1]; Iver Anderson[1]; Thomas Lograsso[1]; [1]Iowa State University

In near future, micromanufacturing is expected to play a key role in that it will bridge the macro- and nano-worlds. Increasing demand for miniature parts has attracted significant industrial and academic interests on near net-shape processing of complex, 3-D micro-parts. Among various techniques, the paper investigated the potential of metallic powder materials at semisolid state (i.e, "mush state") to fabricate micro-features. The unique behavior of solid and liquid phase mixture is expected to overcome challenges encountered by traditional net-shape methods when applied to micromanufacturing. Recent research results on semisolid powder forming (SPF) of microscale features will be presented. The work investigated phase segregation when semisolid powders were forced into micro-cavity through back-extrusion experiment. Results of microstructural analysis and hardness test will be presented. Finally, potentials of the SPF as a near net-shape micromanufacturing method are discussed.

11:10 AM

Modeling the Critical Conditions for the Rolling of Seamless Pipes: *Roman Pschera*[1]; Jürgen Klarner[2]; Christof Sommitsch[1]; [1]University of Leoben; [2]voestalpine Tubulars

The present work deals with the description of the material behavior in the feed region and in the rolling gap during cross roll piercing. Numerous previous investigations have shown that the "Mannesmann-Effect" (cyclic plastic deformation of the core of the billet before getting in contact with the plug) exists and has a negative impact on the quality of the pipes. In order to model this effect in the presence of the plug, a new empirical damage criterion was tested. This model takes into account the relative position of the middle principal stress with respect to the other principal stresses. In addition to it, a stress triaxiality function is suggested where negative values influence the damage process. The results agree with the theoretical assumptions giving reason to investigate the "Mannesmann-Effect" in more detail.

11:30 AM

Sol-Gel Synthesis and Adsorption Properties Study of Spinel Manganese Oxide Lithium Ion-Sieves: *Dong Li*[1]; Qinghua Tian[1]; Rongyi Liu[1]; Xueyi Guo[1]; [1]School of Metallurgical Science and Engineering

Two types of spinel manganese oxide lithium ion-sieves were derived from LiMn2O4 and Li1.33Mn1.67O4 respectively, which were synthesized by a sol-gel method using an aqueous solution of lithium hydroxide and manganese acetate containing citric acid as a chelating agent. The two ion-sieves were compared in terms of physical characteristics and lithium adsorption properties. The maximum adsorption capacity in a 0.1 mol•L-1 LiOH system by these ion-sieves were 25mg•g-1 and 30mg•g-1, respectively. The mechanisms of adsorption by these two ion-sieves were discussed, indicating that the change of lattice constants in a lithium ion-sieve reaction is mainly caused by the change of manganese valence, rather than the extraction/insertion of lithium ions.

11:50 AM

Surface Treatment by Variable-Polarity Arc to Promote the Energy Absorption in Laser Welding of Aluminum Alloy: *Rouzbeh Sarrafi*[1]; Dechao Lin[1]; Radovan Kovacevic[1]; [1]Southern Methodist Univ

One of the major obstacles limiting the application of laser technology in the welding of aluminum alloys is the low energy absorption. In order to enhance the laser absorption, a practical technique in which a variable polarity arc is used to treat the surface prior to laser welding is introduced. Non-reflective surfaces on Al6061 were produced by using the mentioned technique. The objective was to investigate the effect of surface treatment by arc on both laser spot welding and continuous laser welding. To help understand the welding process, a machine vision system was integrated, and the molten pool images were captured in real time. Results demonstrated that the by surface treatment, deeper spot welds can be produced. However, it does not play a critical role in the continuous laser welding in keyhole mode since the laser beam mostly interacts with the molten pool, and not with the treated surface.

12:10 PM

Synthesis of Oxide Coated Carbon Nano Fibers via CO Dispropotation in Mg/MgO System: *Farhad Golestanifard*[1]; Mohamad Sharif[1]; [1]Iran University of Science and Technology

Synthesis of oxide coated CNFs via CO Dispropotation in Mg/MgO system was investigated. Mg metal and MgO was mixed with specific weight ratio and then heat treatment at 1000 C at in coke bed. the product was characterized using different method i. e. SEM, TEM, XRD, STA and Raman spectroscopy. The results showed that in this system CNFs can be formed and an oxide (MgO) coating cover outer surface of CNFs. aiding Raman spectroscopy and TEM observation high degree of crystallinity of CNFs was concluded and the arrangement of 2D graphene planes were detected parallel to growth axis of CNFs. oxide coated CNFs prepared via such a simple route showed high oxidation resistance and no weight loss related to oxidation of fibers was seen up to 1200 C.

12:30 PM

In-Situ Fabrication of Metal Matrix Composites by Solidification Process under High Magnetic Fields: *Qiang WANG*[1]; Tie Liu[1]; Chunjiang Wang[1]; Changsheng Lou[1]; Donggang Li[1]; Jicheng He[1]; [1]Northeastern University

High magnetic fields were used to in-situ fabricate metal matrix composites during the solidification processes of Mn-Sb, Mn-Bi and Al-Ni alloys. MnSb-MnSb/Sb-Sb and Mn-BiMn-Bi/BiMn functionally graded materials (FGMs) with gradient structures in morphology and composition have been successfully obtained by controlling magnetic gradients. And anisotropic Al-Al3Ni composites in which the Al3Ni crystals were oriented parallel to the imposed magnetic fields and the primary Al3Ni phases with their long axes were aligned perpendicular to the magnetic fields were also fabricated under uniform magnetic field conditions. The effects of both uniform and gradient high magnetic fields on the migration, crystalline orientation and phase alignment of the reinforced or functional phases during the solidification were examined. Furthermore, the physical properties and mechanical performances of these materials were investigated. The experimental results indicate that the high magnetic field is one of promising approaches in producing metal matrix composites with special performances.

General Abstracts: Structural Materials Division: Session 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: Refractory Metals Committee, TMS: Titanium Committee

Program Organizers: Robert Hanrahan, National Nuclear Security Administration; Eric Ott, GE Aviation

Wednesday AM — Room: 2018
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM

Analysis of Lattice Stress Direction Evolution in Copper Polycrystals Due to Mechanical Loading Using Crystal Yield Surface Vertex: *Tong-Seok Han*[1]; Jun-Sang Park[2]; Paul Dawson[2]; Matthew Miller[2]; [1]Yonsei University; [2]Cornell University

Lattice stresses of a polycrystalline copper under uniaxial tension obtained from the x-ray diffraction experiment and the finite element simulation were compared in a crystal orientation space. The lattice stress distributions from experiment and simulation were in good agreement, and showed significant crystal orientation dependence. To provide insights on the mechanism behind the agreement, the preferred stress direction and its evolution during loading process were investigated using single crystal yield surface vertices. It was found that a lattice stress tends to move toward a vertex when the vertex is aligned or close to the loading direction as the plasticity develops. However, if the closest vertex

from the lattice stress direction is not closely aligned with the loading direction, the lattice stress finds its direction where it can accommodate the equilibrium and deformation of aggregates through grain interactions.

8:50 AM

Compressive Properties of a Closed- Cell Aluminum Foam as a Function of Strain-Rate and Temperature: *Carl Cady*[1]; Cheng Liu[1]; George Gray[1]; [1]Los Alamos National Lab

The compressive deformation behavior of a closed-cell Aluminum foam (ALPORAS) manufactured by Shinko Wire. Co. in Japan was evaluated under static and dynamic loading conditions as a function of temperature. High strain rate tests (1000 - 2000/s) were conducted using a split-Hopkinson pressure bar(SHPB). Quasi-static and intermediate strain rate tests were conducted on a hydraulic load frame. Little change in the flow stress behavior as a function of strain rate was measured. The deformation behavior of the Al-foam was however found to be strongly temperature dependent under both quasi-static and dynamic loading. Localized deformation and stress state instability during testing of metal foams will be discussed in detail since the behavior over the entire range of strain rates indicates non-uniform deformation. Additionally, investigation of residual stresses created during manufacturing was investigated.

9:10 AM

Effects of Alloying Elements on Mechanical Properties of API X80 Linepipe Steels: *Seung Youb Han*[1]; Sang Yong Shin[1]; Chang-hyo Seo[1]; Hakcheol Lee[1]; Jin-ho Bae[2]; Kisoo Kim[2]; Sunghak Lee[1]; Nack J. Kim[1]; [1]POSTECH; [2]Technical Research Laboratories, POSCO

This study aimed at investigating effects of alloying elements on mechanical properties of API X80 linepipe steels. Four kinds of steels were fabricated by varying Mo, Cr, and V additions, and their microstructures, tensile and Charpy impact properties, and effective grain size measurement were analyzed. Since the addition of Mo and V promoted to form fine acicular ferrite and granular bainite, while prohibiting the coarsening of granular bainite, it improved strengths and upper shelf energy, and decreased the energy transition temperature. The Cr addition promoted the formation of coarse granular bainite and hard secondary phases, which led to the increased effective grain size, energy transition temperature, and strength, and the decreased upper shelf energy. The steel containing 0.3 wt.% Mo and 0.06 wt.% V had best impact properties because it was composed of fine acicular ferrite and granular bainite, while tensile properties maintained excellent.

9:30 AM

Finite Element Analysis of Viscoelastic Core Sandwich Structures: Dan Watt[1]; *Xiaomin Li*[1]; [1]University of Windsor

The mechanical behavior of a sandwich panel depends on the face and core materials, and on its geometry. Polymers are sometimes used in the core. These viscoelastic materials are subject to creep and stress relaxation. Because many interactive factors have to be considered for designing sandwich constructions, an effective way to evaluate performance is to use FEA. The present work simulated 7 combinations of different materials based on their viscoelastic and elastic properties as given in the literature. The exception is that compression and shear test values for aluminum foam were obtained experimentally. The most important result is that viscoelastic creep in the polymeric sandwich core, which carries only a very small fraction of the applied load, can lead to large strains in the overall structure. This effect is apparently ignored in the literature, so others may not be aware of its significance.

9:50 AM

Investigation on Microstructure and Properties of Ti-45Al-5.5(Cr, Nb, B, Ta) Alloy Prepared by Double Mechanical Milling (DMM) and Spark Plasma Sintering (SPS): *Yuyong Chen*[1]; Hongbao Yu[1]; Deliang Zhang[2]; Fei Yang[1]; Shulong Xiao[1]; Fantao Kong[1]; Dezhong Wu[1]; [1]Harbin Institute of Technology; [2]The University of Waikato

In this paper, Ti-45Al-5.5(Cr, Nb, B, Ta) alloy with sub-microstructure was prepared successfully by DMM and SPS using element powders as starting materials. XRD patterns show that the as-milled powder is mainly composed of nanometer TiAl and TiAl3 phases, and there is still Al3Ti and Ti phases to exist after DMM. The effect of sintering temperature on microstructure and properties of bulk Ti-45Al-5.5(Cr, Nb, B, Ta) alloy has been investigated. All of the samples sintered at different temperatures (900, 1000 and 1100°C) exhibit high density and same phase constitution (plenty of TiAl phase and a small quantity of Ti3Al , Ti2Al and TiB2 phases). With the sintering temperature is 900°C, the sample shows good ductility, excellent yield strength and fracture strength, with the value of 5%, 1899MPa and 2229MPa, respectively. When the sintering temperature increases, the properties of the bulk Ti-45Al-5.5(Cr, Nb, B, Ta) alloy declined slightly.

10:10 AM

Martensite in Quenched Fe-C Steels and the Engel-Brewer Electron Theory of Crystal Structures: *Oleg Sherby*[1]; Jeffrey Wadsworth[2]; Chol Syn[2]; Donald Lesuer[3]; [1]Stanford University; [2]Battelle Memorial Institute; [3]Lawrence Livermore National Laboratory

The transformation step to form martensite in Fe-C steels has been thoroughly explored over the past 70 years. It is based on the formation of a body-centered-tetragonal phase containing retained FCC austenite. These phases are only observed at above 0.6 wt%C. In contrast, these two phases have not been observed below 0.6 wt%C and no transformation models have been proposed in this carbon range. The present authors propose a model based on two transformations taking place during the quenching process. The first step is from FCC austenite to an HCP phase, designated by the authors as hexagonite. The second step is from hexagonite to BCC ferite containing a carbon rich phase. The 0.6 wt%C composition is interpreted as the maximum solubility of carbon in hexagonite. An electron-controlled mechanism is described, based on the Engel-Brewer theory of crystal structures that is shown to confirm the proposed transformation model.

10:30 AM

Microstructure and Mechanical Behavior in High Strength Nanostructured Spinodal FeNiMnAl Alloys: *Xiaolan Wu*[1]; Ian Baker[1]; Yifeng Liao[1]; Michael Miller[2]; [1]Thayer School of Engineering, Dartmouth College; [2]Oak Ridge National Laboratory

An ingot of Fe35Ni15Mn25Al25 was drop cast and directionally solidified under Ar using a Bridgman furnace. TEM showed that the as-cast alloy had a periodic coherent microstructure consisting of alternating B2 and BCC phases. EDS showed that the BCC phase was rich in Fe and Mn, while the B2 phase was rich in Ni and Al, features confirmed by analysis using a Local Electrode Atom Probe. Hardness measurements were performed as a function of annealing time at 550°C. The directionally solidified alloy showed a steady increase in hardness from 437 HV with annealing time, but the as-cast alloy, which was initially harder at 523 HV, showed more complex behavior. The final hardness after 72 h anneals was very similar at 676 HV for the two initial conditions. The paper will relate the mechanical properties to the changes in microstructure. Research was supported by DOE Award #DE-FG02-07ER46392.

10:50 AM

Neutron Scattering Charaterization of TWIP Steels Deformed by Tensile Test: *Jae Suk Joung*[1]; Yang Mo Koo[1]; Il-Kyung Jeong[2]; [1]POSTECH; [2]Pusan National University

TWIP steels have excellent tensile behavior. It is mainly due to planar faults formation during deformation. So, it is important to know the amount of planar-defects as strain increase. Conventionally there was a method to characterize this kind of defects using diffraction line profile analysis by examining the intensity, displacement and broadeing of the Bragg peaks. But, there are some drawbacks to this approach such as the restriction of the quantitative analysis to the effects of faulting on the Bragg peaks only, ignoring the information in the diffuse scattering. So, we have attempted another technique to include both the Bragg peaks and diffuse scattering. we had increased planar defects using tensile tests and then performed neutron scattering experiment to analyse the defects of TWIP steels. With the scattering data, we conducted the Rietveld refinemet and the PDF refinement, Using those refinement methods we've identified planar defects more effectively.

11:10 AM

Relaxation of Shot Peened and Laser Shock Peened Residual Stresses in a Nickel-Base Superalloy: *Dennis Buchanan*[1]; Reji John[2]; Michael Shepard[2]; [1]University of Dayton Research Institute; [2]Air Force Research Laboratory (AFRL/RXLMN)

Shot peening (SP) is a commonly used surface treatment that imparts compressive residual stresses into the surface of components. The shallow depth of compressive residual stresses, and the extensive plastic deformation associated with shot peening, has been overcome by modern approaches such as laser shock peening (LSP). LSP surface treatment produces compressive residual stress magnitudes that are similar to SP, that extend 4-5 times deeper,

and with less plastic deformation. Retention of compressive surface residual stresses is necessary to retard initiation and growth of fatigue cracks under elevated temperature loading conditions. This presentation compares the thermal relaxation behavior of SP and LSP residual stress profiles in a powder metal nickel-base superalloy (IN100) for a range of temperatures and exposure times. Results indicate that the LSP processing retains a higher percentage of the initial (as processed) residual stress profile over that of SP.

11:30 AM

Surface Characteristics of Low-Temperature Gas Nitrided 316 Ti Austenitic Stainless Steels: *Ozgur Celik*[1]; Eyup Sabri Kayali[1]; Huseyin Cimenoglu[1]; [1]Istanbul Technical University

Austenitic stainless steels are widely used in manufacturing of orthopedic implants due to their excellent corrosion resistance and fabricability. However, the application of austenitic stainless steel as a bearing surface is limited by poor wear and friction behaviour. It has been reported that, formation of nitrogen super saturated solid solution with distorted crystal lattice (expanded austenite) at the outer surface of austenitic stainless steel by low temperature plasma nitriding process (below 450 °C) results in a significant improvement in both tribological and corrosion properties besides surface hardness. In the present study, low temperature nitriding was applied to an AISI 316Ti quality austenitic stainless steel in fluidized bed to form nitrogen rich expanded austenite layer. The surface characteristics of nitrided alloy was examined through microstructural 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.

11:50 AM

The Role of Microstructure on the Fracture Behavior of Cast Ti-5Al-1V-1Zr-1Sn-0.8Mo (Ti-5111) Alloy: *Jennifer Gaies*[1]; Amy Robinson[2]; [1]NSWC Carderock Division; [2]ARL Penn State

A set of heat treatments were developed and conducted on Ti-5111 castings. Critical microstructure features, including prior-ß grain size, a-colony size, a-lath length and thickness, and the volume fraction and composition of the a and ß phases were quantified for each heat treatment condition. Additionally, a series of mechanical tests, including tensile and fracture toughness tests, were conducted on each condition to correlate the critical microstructure features with the mechanical properties of the cast Ti-5111 alloy. Fracture surfaces of several tensile and fracture toughness specimens were examined and directly correlated to the microstructure to understand crack propagation and failure mechanisms of this alloy. Results showed significant scatter in ductility and toughness, even within the same casting set, which is a function of the large grain size. A grain refinement heat treatment was developed to optimize toughness and ductility, while decreasing scatter in the data.

Magnesium Technology 2009: Refining and Surface Treatment

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

Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Wednesday AM — Room: 2006
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Susan Slade, US Magnesium LLC

8:30 AM Introductory Comments

8:35 AM

On the Influence of Settling of $(ZrB_2)_p$ Inoculants on Grain Refinement of Mg-Alloys: Experiment and Calculation: *Robert Günther*[1]; Christian Hartig[1]; Norbert Hort[2]; Rüdiger Bormann[1]; [1]Hamburg University of Technology; [2]GKSS Research Centre

A simulation method for heterogeneous nucleation has been developed that enables the prediction of the resulting grain size in Mg-alloys as a function of the inoculant particle size distribution, cooling rate, alloy constitution and volumetric content of inoculants. Experiments with $(SiC)_p$ and $(Al_4C_3)_p$ have been already successfully performed and verified the model assumptions. In view of the considerably smaller lattice mismatch between ZrB_2 and Mg compared to SiC and Al_4C_3, respectively, $(ZrB_2)_p$ should act even more as potent inoculant for grain refinement. However, the larger density of ZrB_2 leads to sedimentation of the inoculants that greatly alters the particle size distribution and therefore the efficiency of grain refinement. A theoretical estimate for the final grain size under consideration of the settling effect has been performed and will be discussed in view of experimental results.

8:55 AM

Grain Refining of AZ91E Alloy Using Ultrasonic Vibration: *Sandeep Poola*[1]; Qingyou Han[1]; [1]Purdue University

Ultrasonic vibration has been used for grain refining in ingot of small sizes but it is unclear if the technique can be used for grain refining in larger ingots. This article discusses the effect of ingot size on grain refining using ultrasonic vibration. Techniques that combine ultrasonic processing of molten metal and grain refining using nano-particles are evaluated for graining. Experimental results indicate that ultra-fine globular grains can be obtained in small ingots using ultrasonic vibration alone. The grain refining effect using ultrasonic vibration decreases with increasing ingot size. Nano-particles that are dispersed into molten metal using ultrasonic vibration can serve as nuclei for grain refining of Mg alloys.

9:15 AM

The Influence of β-$(Mg_{17}Al_{12})$ Phase Distribution on Corrosion Behavior of AM50 Alloy in NaCl Solution: *Surender Maddela*[1]; Yar-Ming Wang[2]; Anil K. Sachdev[2]; Balasubramaniam R[3]; [1]Missouri University of Science and Technology ; [2]GM Research and Development Center; [3]Indian Institute of Technology-Kanpur

The effect of β ($Mg_{17}Al_{12}$) phase distribution on the corrosion behavior of AM50 alloy in NaCl solution was studied using scanning vibrating electrode technique (SVET) in conjunction with potentiodynamic polarization scan. The β phase distribution was modified by casting AM50 alloy at different cooling rates. For all cooling rates, the following phases were present: primary magnesium (α), eutectic mixture of α and β phase, β phase and Al_8Mn_5 phase. In 0.17 wt% and 1.6 wt% NaCl solutions, the free corrosion potential (FCP) of the moderate cooled alloy was more noble than that for the fast and slow cooled alloy. The corrosion current densities calculated from SVET analysis at zero current potential (ZCP) were in agreement with that determined from potentiodynamic polarization method. This study clearly indicated that for a given magnesium alloy composition, the corrosion resistance of the alloy can be greatly affected by size and distribution of secondary phases.

9:35 AM

Study of Cathodic Metal Transfer to Magnesium Surfaces in Aqueous Environments and Engine Coolant Formulations by Surface Analytic Methods: Zhiming Shi[1]; Pankaj Mallick[1]; *Robert McCune*[2]; [1]University of Michigan-Dearborn; [2]Robert C. McCune and Associates

The corrosion performance of magnesium in the presence of aqueous engine coolants is one of the primary technical challenges in the development of magnesium engine blocks. In addition to the aqueous environment, the coolant loop is a source of dissolved metal ions such as Fe and Cu, which can potentially "plate out" on nascent magnesium surfaces, thereby aggravating corrosion as localized cathodes. The present study considers the composition and growth of the surface film produced on pure magnesium and AM-SC1 magnesium alloy due to transition metal transfer in ethylene glycol-water mixtures containing Fe2+ and Cu2+ ions in solution at 20 and 80°C. Surface compositional analysis was conducted using Rutherford Backscattering Spectroscopy (RBS) and Auger Electron Spectroscopy (AES). It was observed that metal ion transfer from the electrolyte to the magnesium surface created local galvanic corrosion cells on the magnesium surface and promoted higher rates of corrosion in localized areas.

9:55 AM

New Surface Treatment for Developing Luster on AZ31 Magnesium Alloy in Industrial Scale: *Miyoshi Ohara*[1]; Yorinobu Takigawa[2]; Kenji Higashi[2]; [1]Kasatani Corp.; [2]Department of Materials Science, Graduate School of Engineering, Osaka Prefecture University

Magnesium alloys are frequently used in the chassis of laptop computers and cellular phones. The surfaces of such chassis are generally characterized by their luster rather than by any geometrical figures. However, the metallic luster of magnesium alloys is lost by the exposal to air and by the application

of general surface treatment because magnesium alloys is active metal. We therefore, developed a new surface treatment, Mgbright, for producing a luster on the surface of AZ31 magnesium alloy. Mgbright consist of two processes, a chemical treatment process and a coating process, and has the characteristics of developing a primary luster on magnesium and imparting a high resistance to corrosion. We produced high quality chassis of the laptop computers by making use of the characteristics.

10:15 AM Break

10:30 AM

Characteristics of Phosphate Chemical Conversion-Coating on Magnesium Alloy: *Yongfeng Jiang*[1]; Yefeng Bao[1]; Fei Chen[1]; [1]Hohai University

The characteristics of phosphate conversion coating on AZ91D magnesium alloy are investigated. Methods of SEM and EDX analysis, as well as salt spray test and potentiodynamic polarization in 5% NaCl solution, reciprocate erase test using alcohol-cotton and mini-ohm meter are employed to investigate mass transfer and structure transformation in surface layer. A compact and dense surface morphology with fine particles cluster of the oxalate coating is presented on magnesium alloy. The particles are homogeneously distributed over the surfaces of the coatings. The salt spray test of coating is evaluated as 9.5 degree according to ASTM B117. Polarization curves reveal that the anti-corrosion of the magnesium after phosphate treatment is better than the magnesium substrate. The reciprocate erase test using alcohol-cotton for the evaluation of adhesion is over 50 cycles, which achieved desires for adhesion in application. The electrical conductivity to substrate of phosphate chemical conversion coating is below 0.1 Ohm.

10:50 AM

Electroless Ni-P Plating on Magnesium-Lithium Alloy: *Hongjie Luo*[1]; Yihan Liu[1]; [1]Northeastern University

A novel process of electroless Ni-P plating on Magnesium-Lithium alloy was discussed in this paper, by which nickel ions were provided by basic nickel carbonate and a new pretreatment method was introduced for obtaining good quality coating. The corrosion behavior of Magnesium-Lithium alloy without and with coating was compared and the bonding strength of the electroless Ni-P coating to the matrix was also measured. The results showed that the process of electroless Ni-P could easily occur on the intermediate layer and a compact Ni-P coating without flaws could be formed, accordingly the Ni-P coating was above 20 μm thickness and its phosphorus content was about 10.501%. The corrosion potential of Magnesium-Lithium alloy containing the Ni-P coating increased obviously (-0.315V) during anodic polarization in 3.5 mass% NaCl solution and indicated an effective protection for the matrix. It was proved that the Ni-P coating combined closely with Magnesium-Lithium alloy through test.

Magnesium Technology 2009: Twin Roll Casting and Semi-Solid Processing

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

Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Wednesday AM — Room: 2007
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Michele Manuel, University of Florida

8:30 AM Introductory Comments

8:35 AM

NanoMag High Strength/Density Mg Alloy Sheet: *Raymond Decker*[1]; Sanjay Kulkarni[1]; Jack Huang[1]; Stephen LeBeau[1]; [1]Thixomat, Inc

Thixomat has developed the Thixomolding Thermomechanical Processing (TTMP) process to generate high strength/density Mg alloy sheet, called NanoMag. The first step of the process is to Thixomold (T) sheet bar with low porosity containing isotropic fine grains - so obtained by the fast cooling rates inherent in near-liquidus molding. The second step is Thermomechanical Processing (TMP) in 1-2 fast passes of warm deformation designed to command continuous dynamic recrystalization to micron grain size. At the same time coarse eutectic intemetallic phases are refined to nanometer dispersoids. The end result is increases in both yield strength and ductility.

8:55 AM

Magnesium Alloy Strips Produced by a Melt Conditioned Twin Roll Casting (MC-TRC) Process: I. Bayandorian[1]; Z. Bian[1]; *Mingxu Xia*[1]; H. Zhang[1]; Z. Fan[1]; [1]BCAST

Twin roll casting (TRC) offers a promising route for economical production of Mg sheets. But unfortunately, it offers coarse and non-uniform microstructure and severe central line segregation as well. To address this problem, we developed a melt conditioned twin roll casting (MC-TRC) process. Compared with the conventional TRC process, MC-TRC process has the following features: (1) emphasising on solidification control at the casting stage instead of on hot rolling in conventional TRC procss. (2) solidification control achieved by melt conditioning under intensive forced convention prior to the TRC process, which allows an enhanced heterogeneous nucleation followed by equiaxed growth; (3) minimized central line segregation. In this paper we present MC-TRC process and the microstructures of Mg-alloy strip produced by the MC-TRC process. The discussion will be focused on the solidification behaviour of the intensively sheared liquid metal in the twin roll casting process.

9:15 AM

Microstructures of the Deforming Zone in Hot Rolling AZ31 Sheet: L. K. Fan[1]; *L.M. Peng*[1]; R. Wang[1]; J. Dong[1]; W.J. Ding[1]; [1]National Engineering Research Center of Light Alloy Net Forming, Shanghai Jiao Tong University

The OM and SEM were used to investigate the microstructures of the deforming zone in hot rolling AZ31 sheet in present study. FEM was also used to analyze the deformation character. The experimental and simulation results show that the microstructure is nonuniform, which is correlated with the nonuniformities of stress and strain in deforming zone. Howerer, the methods of large strain rolling and multiple pass rolling can enhance the uniformity of microstructure and the forming of twins is in favor of uniformity of microstructure.

9:35 AM

Effect of Warm Rolling and Heat Treatment on Microstructure and Mechanical Properties in Twin Roll Cast ZK60 Alloy Sheet: *Suk-bong Kang*[1]; Jaehyung Cho[1]; Hyoung Wook Kim[1]; Hongmei Chen[2]; Huashun Yu[2]; Guanghui Min[2]; [1]Korea Institute of Machinery and Materials; [2]Shandong University

Microstructure and mechanical properties of ZK60 alloy sheets produced by twin roll casting (TRC) and warm rolling were investigated using OM, SEM, TEM and a standard universal testing machine. The microstructure of TRC ZK60 alloy strip consisted of dendrite structure, eutectics and intermetallic compounds located in the interdendritic region. Relatively higher density of shear bands was observable in TRC ZK60 alloy sheet and no obvious dynamic recrystallization was found after warm rolling. The warm rolling induced high strength and low elongation in the TRC ZK60 alloy sheets. Annealing treatment after warm rolling induced the decrease of strength and increase of elongation. Solution treatment at 3750C for 3 hours and subsequent artificial aging treatment at 1750C for 10 hours can be considered to be the optimum T6 treatment. The uniformity of tensile properties was improved and fine equiaxed structure was obtained at this optimum T6 treatment condition.

9:55 AM Break

10:10 AM

Development of Rolling Technology for Twin Roll Casted 1500mm Wide Magnesium AZ31 Alloy: *Ozgur Duygulu*[1]; Selda Ucuncuoglu[1]; Gizem Oktay[1]; Deniz Temur[1]; Onuralp Yucel[2]; Ali Kaya[3]; [1]TUBITAK MRC, Materials Institute; [2]Istanbul Technical University, Department of Metallurgical and Materials Engineering; [3]Mugla University, Engineering Faculty, Department of Metallurgy and Materials

Magnesium alloy AZ31 sheets of 4.5-6.5mm thick and 800 and 1500mm width were produced by twin roll strip casting first time in Turkey. Afterwards, sheets were hot and cold rolled down to less than 1mm both by laboratory and industrial scale rolls. Microstructure of the sheet was analyzed by optical microscope and scanning electron microscope. In addition, pole figures have been obtained by XRD studies. Mechanical properties were investigated by tensile tests and also hardness measurements. Anisotropy ratio of the rolled sheets has been studied. Annealing heat treatments were performed on the produced sheets. Forming trials and deep drawing tests have been done on the sheets having different thicknesses.

10:30 AM

Development of 150cm Wide Wrought Magnesium Alloys by Twin Roll Strip Casting Technique in Turkey: *Ozgur Duygulu*[1]; Selda Ucuncuoglu[1]; Gizem Oktay[1]; Deniz Temur[1]; Onuralp Yucel[2]; Ali Kaya[3]; [1]TUBITAK MRC, Materials Institute; [2]Istanbul Technical University, Department of Metallurgical and Materials Engineering; [3]Mugla University, Engineering Faculty, Department of Metallurgy and Materials

Magnesium alloy AZ31 sheet was produced by twin roll strip casting first time in Turkey. Sheets of 4.5-6.5mm thick and 800 and 1500mm width were successfully achieved. Microstructure of the sheet was analysed by optical microscope and scanning electron microscope, SEM from lenght, width, thickness and wedge views. Semi-quantitative analyses were performed by SEM-EDS. In addition, XRD studies were performed for both characterization and texture purposes. Mechanical properties were investigated by tensile tests and also hardness measurements. Tensile tests were performed at three different directions: rolling direction, 45 degrees to rolling direction and transverse direction. Moreover, micro Vickers and Brinell Hardness test measurements were done on different crosssection directions. Homogenization and annealing heat treatments were performed on the produced sheets.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Advanced Coatings I

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Wednesday AM Room: 3010
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Gerald Meier, University of Pittsburgh; Bruce Pint, Oak Ridge National Laboratory

8:30 AM Invited

Thermo-Mechanical Property Profiles Governing the Performance of Coated Airfoils Used in Aero-Turbines: Opportunities for Materials Innovation: *Anthony Evans*[1]; [1]University of California, Santa Barbara

Oxides and Ni-alloys are present in the hot section of turbines used for propulsion and power generation. By designing and using these materials in optimal combinations, it has been possible to systematically increase the combustion temperature: In turn, enhancing the fuel efficiency. The utility of these materials is greatest in the high-pressure turbine, especially the airfoil. The technology has demonstrated how oxides can be used to protect load-bearing, Ni-based structural members that experience environmental extremes. It involves choices of materials and spatial configurations, as well as survivability upon extreme temperature cycling, without loss of functionality. The newest research on mechanisms governing durability has identified the combinations of material properties that dictate temperature capability. In turn, this identification has provided directions for materials innovation expected to further enhance fuel efficiency. These innovations are described and discussed.

8:55 AM Invited

Opportunities and Trade-offs in Designing Next Generation TBCs: Rafael Leckie[1]; Stephan Krämer[1]; Jessica Koschmeder[1]; Erin Donohue[1]; *Carlos Levi*[1]; [1]University of California, Santa Barbara

Zirconia partially stabilized with 7±1wt%Y_2O_3 (7YSZ) has been the standard material for thermal barrier coatings (TBCs) since their commercial insertion, but it is becoming increasingly limited as engine temperatures continue to rise. Key durability concerns arise from accelerated sintering kinetics, loss of phase stability and attack by calcium-magnesium-alumino-silicate (CMAS) deposits. In addition, improved performance drives the search for materials with lower thermal conductivity. Numerous alternate compositions exist, but every design path involves improvements in some properties and detriment in others. Conceptual guidelines for design and the associated opportunities and trade-offs will be examined in this presentation in the context of current research on new compositions by the authors and their collaborators. Emphasis will be on the interplay between phase stability, thermal conductivity and toughness, as well as on the implications for improved resistance to CMAS attack. Research sponsored by NSF (DMR-0605700) and ONR (N00014-08-1-0522).

9:20 AM

Hf Addition by Sputtering in β-NiPtAl Bond Coating for TBC Systems and Its Effect on Thermal Cycling Behaviour: *Aurélie Vande Put*[1]; Djar Oquab[1]; John Nicholls[2]; Daniel Monceau[1]; [1]CIRIMAT; [2]Cranfield University

During thermal cycling under oxidising conditions, thermal barrier systems undergo microstructural and morphological changes as well as stresses development. These evolutions initiate cracks whose propagation leads to spallation. Works by Streiff *et al.* and Pint *et al.* showed that Hf additions improve spallation resistance of nickel aluminide coatings/materials. Based on these results, Hf is added to a β-NiPtAl thermal barrier system. The chosen manufacturing process consists in depositing, onto the superalloy, alternative Hf and Pt layers by sputtering. A conventional diffusion treatment is performed before out-of-pack aluminising and a final heat treatment. To study the effect of Hf on TGO adherence and rumpling, the systems are thermally cycled, at 1100°C in air, in the same time as conventional Pt-modified nickel aluminide thermal barrier systems. After failure, TGO and bond coating microstructure and composition are analysed by SEM and compared to the "as-processed" systems.

9:40 AM

Cyclic Oxidation Behavior of Multilayer NiCrAlYSi/Ru-Al Coatings on the DZ125 Superalloy: Liang Chen[1]; Limin He[2]; *Qiang Feng*[1]; [1]University of Science & Technology Beijing; [2]Beijing Institute of Aeronautical Materials

Improvement in creep stress of the bond coat could extend the life time of thermal barrier coatings (TBCs). Recently, it was reported that RuAl had shown a superior creep resistance. However, limited research has been devoted to the influence of the addition of Ru to the MCrAlY bond coat. In the present study, the fabrication of the Ru-Al modified multilayer NiCrAlYSi coatings was developed via arc ion plating technique (AIP), including the Ru-Al layer deposited by detonation spraying technology. The cyclic oxidation behavior of NiCrAlYSi coatings containing the Ru-Al multilayer in various positions has been investigated. The results indicated that the coatings with Ru-Al multilayer exhibited better oxidation resistance than the conventional NiCrAlYSi coatings. The evolution of the coating microstructure at various stages of cyclic oxidation and the relevant interdiffusion between the coating and substrate was studied. The corresponding mechanisms for different types of coatings were evaluated and discussed.

10:00 AM

Development of Oxidation Resistant Pt-Based Coatings on γ-TiAl for High Temperature Applications: *Maik Froehlich*[1]; Andrea Ebach-Stahl[1]; Christoph Leyens[2]; [1]DLR-German Aerospace Center; [2]Technical University of Brandenburg at Cottbus

Due to much lower density and excellent mechanical properties γ-TiAl alloys are promising candidates to replace the heavy steels and Ni-based superalloys typically applied in the field of aerospace and automotive industry. Protective coatings are necessary to exploit the full potential of γ-TiAl at temperatures higher than 750°C; however, so far no coating system tested has proven sufficient performance for long-term use.The paper is focused on the development of Pt-based coatings produced by magnetron sputtering. Two Pt containing systems with different aluminum contents - PtAl and $PtAl_2$ - were investigated aiming at the formation of a slow growing and protective Al_2O_3 scale. The oxidation resistance of each coating system was tested under cyclic conditions at 950°C up to 1000 1h-cycles and compared to the oxidation behaviour of pure Pt deposited on γ-TiAl. Investigations of microstructure evolution will be presented observed by means of SEM and EDS analysis after exposure.

10:20 AM Break

10:30 AM

Effects of Reactive Element and Silicon Additions on the High-Temperature Oxidation Behavior of γ-Ni+γ'-Ni3Al-Based Alloys: *Zhihong Tang*[1]; Scott Chumbley[1]; Eren Kalay[1]; Brian Gleeson[2]; [1]Iowa State University; [2]University of Pittsburgh

The effects of Hf and/or Y additions on the isothermal and cyclic oxidation behavior in air of γ-Ni+γ'-Ni3Al-based alloys were investigated. It was found that Hf addition was more effective in slowing scale growth rate, while Y addition appeared to have a greater effect in improving scale adhesion. Co-

doping with Hf and Y significantly improved the cyclic oxidation resistance of alloys compared to single Hf or Y addition. This beneficial effect of co-doping was more apparent for cyclic oxidation to 1150ºC than to 1000ºC. Addition of Si to co-doped alloys further markedly improved the cyclic oxidation performance of γ-Ni+γ'-Ni3Al-based alloys. The oxidized alloys were characterization by SEM, TEM and STEM to study the scale structure, segregation behavior of Hf or Y on the alumina scale grain boundaries to explain the underlying mechanism of co-doping and Si additions.

10:50 AM

Understanding the Role of Dopants in the Structural Evolution of YSZ: *Jessica Koschmeder*[1]; Yan Gao[2]; Don Lipkin[2]; Carlos Levi[1]; [1]University of California at Santa Barbara; [2]GE Global Research, Niskayuna, NY

The evolution of the metastable t'-phase of air plasma sprayed yttria stabilized zirconia (YSZ) coatings was studied using high-resolution synchrotron x-ray diffraction data. In order to observe the phase evolution as a function of time and temperature, the coatings were subjected to a wide range of heat treatments characterized by a Larson Miller Parameter (LMP). Rietveld's refinement method was employed to develop more accurate structural models for Ti- and Ta-doped YSZ. Using this method phase fractions were also quantified enabling the calculation of the resulting tetragonal and cubic phase compositions.

11:10 AM

Lifetime Prediction of Thermal Barrier Coatings Using Computational, Experimental and Non-Destructive Tools: *Andre Luz*[1]; Daniel Balint[1]; Kamran Nikbin[1]; [1]Imperial College London

Thermal barrier coatings (TBCs) deposited on the superalloy turbine blades can lower the temperature of metallic substrate by 100-300°C, allowing an increase in the turbine inlet temperature. As a result, the engine efficiency is improved. However, the TBCs have durability problems due to the significant thermal mismatch between the coating and substrate, which leads to crack nucleation and propagation at the interface and subsequent coating delamination and loss of thermal protection. In order to understand the influence of several non-linear thermo-mechanical and microstructural parameters in the life of the TBC a new finite element model was developed and run in a high-performance distributed computing system. The evolution of material properties with thermal exposure was experimentally determined in collaboration with a number of international research centers and the results from several image- and laser-based non-destructive techniques were merged using a neural network to improve the lifetime prediction of TBCs.

Materials for the Nuclear Renaissance: New Materials and Past Limitations

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS/ASM: Nuclear Materials Committee, TMS: Refractory Metals Committee

Program Organizers: Raul Rebak, GE Global Research; Robert Hanrahan, National Nuclear Security Administration; Brian Cockeram, Bechtel-Bettis Inc

Wednesday AM — Room: 2009
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Raul Rebak, GE Global Research; Robert Hanrahan, National Nuclear Security Administration

8:30 AM Invited

Advanced Materials for Nuclear Reactor Systems: Overcoming Past Limitations: *Jeremy Busby*[1]; Steven Zinkle[1]; [1]Oak Ridge National Lab

Advanced materials have the potential to improve reactor performance via increased safety margins, design flexibility, and fast reactor economics and can overcome traditional limitations. Increased strength and creep resistance can give greater design margins leading to improved safety margins, longer lifetimes, and higher operating temperatures. The use of advanced materials for component replacement in the existing light water reactor fleet may improve safety margins and reduce the frequency of component replacement. The use of advanced materials in nuclear reactor systems requires considerable development and licensing effort, however. Modern materials science tools such as computational thermodynamics and multiscale radiation damage computational models in conjunction with rapid science-guided experimental validation may offer the potential for a dramatic reduction in the time period to develop and qualify structural materials. This paper will discuss the potential impacts of advanced materials on nuclear reactor systems and contrast those gains with the hurdles for alloy development.

9:10 AM

Development of Ni-W-Cr Alloys for Gen IV Nuclear Reactor Applications: *Thierry Auger*[1]; Rafael Cury[2]; Jean-Pierre Chevalier[3]; [1]Ecole Centrale de Paris; [2]CECM-CNRS; [3]Conservatoire National Des Arts Et Metiers

Whether for high temperature gas cooled or molten salt reactor designs, alloys are required to be oxidation and corrosion resistant, to have appropriate high temperature mechanical properties (creep resistance and yield stress) as well as acceptable room temperature toughness. For instance,a Ni based alloys like Hastelloy N was selected for the Oak Ridge National Laboratory experimental molten salt reactor. The related Ni-Cr-W system offers improvements over Ni-Cr-Mo alloys, such as a lower activation or diffusion of W with respect to Mo and potentially higher in-service temperature, while maintaining similar corrosion and oxidation resistance. Here we present results on ternary alloys, with special emphasis on short-range order (SRO), high temperature hardness, phase diagram determination, and oxidation behaviour. Using electron diffraction, the structural state (in terms of short and long range order) of the alloys as a function their composition (especially, the influence of Cr on SRO) will be presented.

9:30 AM

Super ODS Steels R&D towards Gen-IV Systems: *Akihiko Kimura*[1]; [1]Kyoto University

The development of high performance fuel cladding is essential for the realization of Gen-IV systems. The 9Cr-ODS martensitic steel was developed as the cladding material for sodium-cooled first breeder reactor in Japan, and the steel showed a good performance in sodium, while the corrosion resistance is poor in supercritical water (SCW) and lead-bismuth eutectics (LBE). High-Cr ODS steels added with Al showed a drastic improvement in the corrosion resistance in SCW and LBE. High-temperature strength, however, was reduced because of the characteristic changes in the dispersion morphology of aluminum oxide particles. Recently, "super ODS steels" have been developed by means of the third element alloying method, which results in an achievement of high-temperature strength even with Al addition, as well as high-resistance to corrosion in SCW and LBE. The strengthening mechanism of the super ODS steels is proposed on the bases of nano/meso structure observations and analyses by FE-TEM/EDS, FE-EPMA and FE-AES. Present study includes the result of "R&D of corrosion resistant super ODS steel for highly efficient nuclear systems" entrusted to Kyoto University by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

9:50 AM

A Comparative Study of Uniform Corrosion of Refractory Alloys in Supercritical Water: *Mickael Payet*[1]; Patrick Arnoux[2]; Olivier Raquet[2]; Jean-Pierre Chevalier[1]; [1]CNAM; [2]CEA-Saclay

Interest in supercritical water (SCW) for higher efficiency energy production, has led to the supercritical water-cooled reactor (SCWR) concept. SCWR designs lead to coolant temperatures until 620°C at 25MPa. Materials selection criteria concern high temperature yield stress, creep resistance, no embrittlement and resistance to both uniform corrosion and stress corrosion cracking. This paper presents results of a comparative study of uniform corrosion in SCW of several austenitic stainless steels and Ni-based alloys after exposure to deaerated SCW at 610°C and 25 MPa. Observations of cross-sections reveal that two-layer oxides were formed. Steels present thicker films but in some circumstances, thinner films are observed and may be related to surface preparation. Ni-based alloys present much thinner films with similar structures. Cation and anion diffusions would be responsible respectively of the outer and inner oxide layer growths. The effects of alloy composition and surface microstructure on the oxide films will be discussed.

10:10 AM Break

10:20 AM Invited

Challenges of Materials Degradation in Light Water Reactors: *Peter Andresen*[1]; [1]GE Global Research Center

Environmental degradation of materials in high temperature water has been a dominant factor in safe and economic operation of light water reactors. All structural materials, which are comprised of iron- and nickel-base materials, are

susceptible to phenomena such as stress corrosion cracking, corrosion fatigue, irradiation assisted SCC, environmental effects on fracture, and others. This talk summarizes these factors, emphasizing the common underlying factors that determine their response and the associated lifetime of components.

11:00 AM Invited
Mitigation of Environmentally Assisted Cracking in Nuclear Power Plants: *Aladar Csontos*[1]; Lee Fredette[2]; Paul Scott[2]; [1]U.S. Nuclear Regulatory Commission; [2]Battelle Memorial Institute

Environmentally assisted cracking of components in nuclear power plants is an area of continued focused research by the domestic and international nuclear power industry and regulatory bodies. Domestically, the U.S. Nuclear Regulatory Commission (NRC) is currently conducting several research programs evaluating environmentally assisted cracking in pressurized (PWR) and boiling water reactors (BWR) components. One of these programs indicated that residual stresses play a key role in the growth and arrest of stress corrosion cracks (SCC) in PWR piping components containing dissimilar metal butt welds. Residual stresses in these types of components typically arise from fabrication, fit up, joining, and repair processes. This talk will present the results of NRC research programs evaluating the effectiveness of SCC mitigation measures to include using engineered residual stresses to potentially limit SCC initiation and growth in PWR components.

11:40 AM
A PWSCC Mechanism in Alloy 600: *SungSoo Kim*[1]; JoungSoo Kim[1]; [1]Korea Atomic Energy Rsch Inst

A new explanation is proposed base on an order reaction in Alloy 600. Both the existence of order reaction in Alloy 600 and an activation energy for an order reaction in Alloy 600, Q=~46 kcal/mole (~190 kJ/mole), are determined by a differential scanning calorimeter (DSC). The lattice contraction was confirmed by a high resolution neutron diffraction using a series of isothermally treated specimens at 400°C to accelerate the ordering reaction. The ordering reaction in Alloy 600 causes a lattice contraction and produces an additional stress, internally, in components made of Alloy 600 during a reactor operating condition. The stress level by the order reaction would be about 50 ~ 150 MPa according to the lattice planes. This stress level may be doubled by a certain combination of the neighboring grains. It seems that the basic process of a PWSCC is controlled by the additional stress due to an ordering.

WEDNESDAY AM

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Materials for Hydrogen Production and Transport

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Zi-Kui Liu, Pennsylvania State University

Wednesday AM — Room: 3005
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Zi-Kui Liu, Pennsylvania State University

8:30 AM Introductory Comments

8:35 AM Invited
H2 Generation by Solar Water Splitting: *Craig Grimes*[1]; [1]Pennsylvania State University

Abstract not available.

9:10 AM Invited
Effect of Microstructure on Hydrogen Embrittlement of Pipeline Steels: *Govindarajan Muralidharan*[1]; Joe Strizak[1]; Neal Evans[1]; Doug Stalheim[2]; Subodh Das[3]; [1]Oak Ridge National Laboratory; [2]DGS Metallurgical Solutions, Inc.; [3]Phinix LLC

High pressure transport through pipelines is one of the most economical methods for hydrogen delivery. However, hydrogen is known to embrittle steels that would be typically used for this cost-effective transport. Very little information is available on the embrittlement of pipeline steels exposed directly to high pressure hydrogen, and on the effect of microstructure on the extent of embrittlement. This presentation will highlight the effect of high pressure hydrogen on the mechanical properties of pipeline steels from the X-70/X-80 grade, with a ferritic-pearlitic microstructure, and a ferrite-acicular ferrite microstructure. This talk will review the effect of high pressure hydrogen on the mechanical properties of these steels measured in-situ in a hydrogen atmosphere with specific reference to hydrogen pressures, steel compositions, and microstructures. *Research sponsored by the U.S. Department of Energy's Hydrogen, Fuel Cells and Infrastructure Technologies Program.

9:45 AM
Development of a Non-Noble Metal Hydrogen Purification System: *Paul Korinko*[1]; Thad Adams[1]; Kyle Brinkman[1]; George Rawls[1]; [1]Savannah River National Laboratory

High purity hydrogen is essential for the hydrogen economy to be viable as an alternative to fossil fuels. The development of advanced separation membranes to economically remove gas contaminants from hydrogen produced using coal gasification or as front end gas purifiers is needed. In addition, new gas purification devices are needed to replace tube type palladium based purifiers to reduce costs and maintain rapid throughput. While Pd-based systems are highly successful, they suffer from both the high costs of raw materials as well as the high costs associated with thin tube manufacturing. In this work, a multi-phase vanadium-nickel-titanium alloy has been tested for use as a hydrogen separation membrane. It exhibited acceptable permeability as a thin foil, therefore a scaled-up prototype reactor was designed and built. This presentation will discuss the design details and test data obtained from the prototype gas purifier.

10:05 AM Break

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Advanced Materials for PEM Fuel Cells and Batteries - Session I

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Zi-Kui Liu, Pennsylvania State University

Wednesday AM — Room: 3005
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Zi-Kui Liu, Pennsylvania State University

10:10 AM Keynote
The Science and Economics of Materials for Batteries, Capacitors, and Fuel Cells: *Jay Whitacre*[1]; [1]Carnegie Mellon Univeristy

There is now an intense need for alternative clean energy technologies, a key aspect of which is the ability to generate, store and convert power for applications ranging from cell phones to power grid nodes. This talk will offer an overview of the state of the art in materials for energy storage systems for the transportation and stationary sector, as well as examination of the critical materials for fuel cells (both PEM and SOFC). An overview of the economics of scaled materials production for these technologies will be presented and a subsequent analysis of the most promising materials and systems to further focus on will be offered.

10:55 AM Invited
Novel Fabrication Strategies for Control of Electrode Architectures: *Peter Rieke*[1]; [1]Pacific Northwest National Laboratory

More than incremental increases in the energy and power densities of batteries and fuel cells will require improved electrode architectures and new techniques to fabricate those architectures. For example, optimal mass transport in fuel cells requires not only an integration of structural features from the nano to macro scales but also requires variation in structure across the plane of the electrode. As

applied to polymer electrolyte membrane fuel cells, digital fabrication methods can be used to control structures from the micro to macro scales and result in improved performance and elimination of processing steps. In a second example, phase separation methods can be used to control porosity in high power lithium batteries on the nano to micro scales – using in the processing steps only the components needed for the electrolyte. This results in substantial simplification and cost saving in the fabrication of lithium batteries while improving power density. With new electrode architectures and new fabrication methods, batteries and fuel cells can met the increased performance and cost criteria driven by increasing energy costs and demands for portable power.

11:30 AM

Effect of β"-Alumina Electrolyte Thickness on the Performance of Na/$NiCl_2$ Cells: *Amin Mali*[1]; Anthony Petric[1]; [1]McMaster University

Na/$NiCl_2$ batteries use β"-alumina solid electrolyte tubes 1-2 mm thick and operate at elevated temperatures. Reducing the β"-alumina electrolyte thickness offers significant advantages in battery performance due to the reduction in cell internal resistance. Different methods including electrophoresis, sol-gel and slip casting were used to fabricate β"-alumina tubes. Dense electrolyte tubes with reduced thickness of less than 100 µm and supported on a porous substrate were successfully produced. The electrolyte microstructure was examined by scanning electron microscopy. The effect of electrolyte thickness on the performance of Na/$NiCl_2$ cells was investigated in sealed laboratory research cells.

11:50 AM

Synthesis and Electrochemical Properties of Al-Doped Li3V2(PO4)3 Cathode Materials for Lithium Batteries: Shengkui Zhong[1]; Zhoulan Yin[1]; Jiequn Liu[1]; Qiyuan Chen[1]; [1]Central South University

Al-doped Li3V2(PO4)3 cathode materials were prepared by a carbothermal reduction(CTR) process. The properties of the Al-doped Li3V2(PO4)3 were investigated by X-ray diffraction (XRD) and electrochemical measurements. XRD studies show that the Al-doped Li3V2(PO4)3 has the same monoclinic structure as the undoped Li3V2(PO4)3. The Al-doped Li3V2(PO4)3 samples were investigated on the Li extraction/insertion performances through charge/discharge, cyclic voltammogram (CV), and electrochemical impedance spectra(EIS). The optimal doping content of Al was that x=0.04 in the Li3V2-xYx(PO4)3 samples to achieve high discharge capacity and good cyclic stability. The electrode reaction reversibility was enhanced, and the charge transfer resistance was decreased through the Al-doping. The improved electrochemical performances of the Al-doped Li3V2(PO4)3 cathode materials are attributed to the addition of Al 3+ ion by stabilizing the monoclinic structure.

12:10 PM

Electroless Copper Plating on Microcellular Polyurethane Foam: *Qinghua Tian*[1]; Xueyi Guo[1]; Qingming Feng[1]; Shengzhang Liu[1]; [1]Central South University

In order to get a good conductivity substrates for foam zinc materials used in zinc-air battery, a novel method for electroless copper plating on microcellular polyurethane foam with diameter of 0.2mm was proposed. A new salt-based palladium colloid activation solution was compared with conventional acid palladium colloid activation solution. The result show that the salt-based palladium colloid activation solution presented fairly high activity, all property parameters of it were better than acid palladium colloid activation solution. The stability of electroless copper plating solution has been studied in this paper. The effects of the components of the solution and the technological conditions on the stability and the sedimentation rate have been analyzed by experiments. The optimal prescription for the solution and the technological regulation for electroless copper plating on polyurethane foam have been determined.

Materials Processing Fundamentals: Powders, Composites, Coatings and Measurements

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

Wednesday AM Room: 2016
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Prince Anyalebechi, Grand Valley State University

8:30 AM

Preliminary Evaluation of the Processing of Carbon Nanotube Reinforced Aluminum Composites: *K. Morsi*[1]; A. ESAWI[2]; P. Borah[1]; S. Lanka[1]; A. Sayed[2]; A. Gawad[2]; [1]San Diego State University; [2]American University in Cairo

Carbon nanotube (CNT) reinforced metallic composites have been recently generating significant scientific interest, due to their expected superior properties compared with other composites. Out of the metallic matrices investigated for reinforcement with CNTs, aluminum has received considerable attention. A major problem has been the dispersion of CNTs in metallic matrices. This paper discusses preliminary results on the mechanical dispersion of CNTs in aluminum powder, powder processing, stability and characterization of aluminum dispersed with CNTs at reinforcement levels equal to and below 5 wt.%.

8:45 AM

Synthesis of Nanosized Tungsten Powder by a Thermal Plasma Process and Its Sintering Behavior: Taegong Ryu[1]; Kyu Sup Hwang[1]; *Hong Yong Sohn*[1]; Zhigang Fang[1]; [1]University of Utah

Nanosized tungsten powder was synthesized by the hydrogen reduction of ammonium paratungstate (APT) in thermal plasma. The effects of operating conditions on the product composition and particle size were investigated. The particle size of synthesized W powder was less than 30 nm in all cases tested. The sintering behavior of the synthesized powder (25 nm average size) was then investigated and compared with those of commercial W powder of 500 nm average size and W powder of 23 nm average size produced by milling the commercial powder. The sintering was done at 1400°C for 60 minutes. The hardness of the resulting compact from the synthesized W powder (315 VHN) was similar to that from the milled W powder (309 VHN), but the plasma-synthesized powder had a much lower tendency to form cracks. The compacts of both nanopowders were significantly harder than that of the commercial powder (192 VHN).

9:00 AM

Plasma-Assisted Chemical Vapor Synthesis of Tungsten Carbide and Cobalt Nanocomposite Powder: Taegong Ryu[1]; Kyu Sup Hwang[1]; *Hong Yong Sohn*[1]; Zhigang Fang[1]; [1]University of Utah

A thermal plasma process was used to synthesize nanosized tungsten carbide - cobalt composite powder, in which ammonium paratungstate and cobalt oxide were reacted with a gas mixture containing CH_4, H_2 and Ar. The reduction and carburization of vaporized precursors produced nanosized tungsten carbide (WC_{1-x}) - cobalt composite powder, which sometimes contained small amounts of W_2C and/or W phase. The effects of gas composition, plasma torch power, the flow rate of plasma gas, and the addition of secondary plasma gas (H_2) on product composition and grain size were investigated. The grain size of synthesized tungsten carbide powder was less than 20 nm. The synthesized composite powders were also subjected to a hydrogen heat treatment to fully carburize WC_{1-x}, W_2C, and W phases to the WC phase as well as to remove excess carbon in the product. Finally nanosized WC-Co composite powder of grain size less than 50 nm was obtained.

9:15 AM

Highly Stable Modification of Silicon Carbide and Silicon Nitride Surfaces by Covalently Attached Organic Monolayers: *Han Zuilhof*[1]; [1]Wageningen University

Silicon carbide and silicon nitride are both highly attractive materials due to their mechanical robustness and chemical "inertness". It is therefore of interest to be able to modify the surface of these materials by a stable organic monolayer (thickness: 1-2 nm) that persistently changes relevant surface properties. This paper discusses options to make these surfaces either hydrophobic or fully

protein-repelling via the attachment of tailor-made organic molecules (omega-functionalized-1-alkenes) at room temperature. These conditions allow a wide range of bio-functional moieties to be attached to the substrates. The synthesis and detailed characterization of these surfaces will be outlined (e.g. via XPS, IRRAS, contact angle). Subsequently the extremely high stability of the functionalized surfaces in hot acid and base will be discussed, and finally the protein repellence of oligoethylene oxide-functionalized monolayers.

9:30 AM

Processing and Characterization of Hybrid Perform for Composites: *Qiang Zhang*[1]; Henry Hu[1]; Lihong Han[1]; [1]University of Windsor

Hybrid composites are fabricated by adding two or more reinforcements into matrix materials so that the expected excellent properties can be achieved through the combined advantages of short fibers and whiskers and different size particles including nanoparticles, which provide a high degree of design freedom. In this paper, hybrid preforms were produced by mixing Al2O3 short fiber with low volume fraction of micro Al2O3 particles. The composites prepared with the hybrid preforms are characterized by optical and scanning electron microscopy. The results show that the reinforcements distribute homogeneously in the matrix materials.

9:45 AM

In Situ Synthesis of Silicon-Silicon Carbide Composites from SiO2-C-Mg System via Self-Propagating High Temperature Synthesis: *Sutham Niyomwas*[1]; [1]Prince of Songkla Univ

Silicon-Silicon Carbide (Si-SiC) composites were synthesized by self-propagating high temperature synthesis (SHS) from a powder mixture of SiO2-C-Mg. The reaction was carried out in a SHS reactor under static argon gas at a pressure of 0.5 MPa. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of the reacting species. The effects of silica sources and carbon mole ratio in precursor mixture on the Si-SiC conversion were investigated using X-ray diffraction and scanning electron microscope technique. The as-synthesized products of Si-SiC-MgO powders were leached with 0.1M HCl acid solution to obtain the Si-SiC composite powders.

10:00 AM Break

10:15 AM

Spark Plasma Sintering and Post-Sinter Annealing of Alumina: *Lin Huang*[1]; Wenlong Yao[1]; Jing Liu[1]; Dongtao Jiang[1]; Amiya Mukherjee[1]; Julie Schoenung[1]; [1]University of California, Davis

Magnesia doped alumina has been synthesized via spark plasma sintering (SPS) in two-stage sintering schedules. The effects of both dopant contents and sintering conditions, including sintering temperature, heating rate, holding time on microstructure, such as grain size, porosity, and mechanical properties, such as microhardness, fracture toughness and strength have been investigated. In addition, the influences of select post-sinter annealing conditions, including a variety of annealing times and temperatures, on grain growth have also been considered. The concurrent hindrance on grain growth kinetics of dopants and the application of two-stage sintering has been studied using scanning electron microscopy (SEM) and phase transition studies using X-ray diffraction (XRD).

10:30 AM

Effect of Sintering Temperature and Pressure on the Properties of ZrB2-SiC Composites Prepared by Spark Plasma Sintering: *Ipek Akin*[1]; Mikinori Hotta[2]; Takashi Goto[2]; Filiz Sahin[1]; Gultekin Goller[1]; [1]Istanbul Technical University; [2]Tohoku University Institute for Materials Research

The ZrB2-SiC composites were prepared by the spark plasma sintering (SPS) technique. The SPS process was carried out at different sintering temperatures of 1800-2100°C for 180-300 s with a heating rate of 1.7°C/s in vacuum and uniaxial pressures of about 20 to 80 MPa were applied during the entire process. Densities of the composites were determined by the Archimedes' method and more than 99% relative density was obtained for the composites. The hardness of the composites was calculated as 15 to 26 GPa at the loads of 0.9 to 9.8 N and the fracture toughness was calculated from a half-length of crack formed around corners of indentation and the obtained results were in the range of 3.5 to 4.2 MPa•m1/2 at the loads of 2.9 to 9.8 N.

10:45 AM

Development of Powder Injection Molding Process for Sponge Ti Alloy: Ozkan Gulsoy[1]; Pavan Suri[2]; *Seong Jin Park*[3]; Arockiasamy Antonyraj[3]; Randall German[4]; Paul Wang[3]; [1]Marmara University; [2]Heraeus; [3]Mississippi State University; [4]San Diego State University

Newly developed sponge Ti powder and its alloys have price competitiveness suitable for automotive application. In this study, the metal powder injection molding process was developed for sponge Ti alloyed with Fe and Zr. The effort included development of the alloy composition, binder system, mixing process, debinding process, and sintering process. All samples were analyzed in term of density, microstructure, and mechanical properties. The process simulation and microstructures analysis were performed to rationalize the effects of processing conditions on the sintered density and mechanical properties.

11:00 AM

Migration and Interaction Behavior of Electrical-Insulating Particles in a Conductive Melt under Strong Magnetic Field with High Gradient: *Zhi Sun*[1]; Muxing Guo[1]; Tadej Kokalj[1]; O. Vander Biest[1]; Bart Blanpain[1]; [1]Katholieke Universiteit Leuven

Magnetic fields have been used for inclusions removal from melt and preparation of materials with gradient compositions. Fundamental in these applications is that the induced magnetic forces on the particles/melt can enhance migration and change the interaction behavior. In the present paper, migration and interaction behavior of electrical-insulating particles in a conductive melt under strong magnetic field are theoretically analyzed. It is found that the migration can be considerably enhanced for micrometer sized particles by applying a strong magnetic field. In addition, two forces are involved in the interaction between two particles: the interparticle magnetic dipole-dipole attractive force due to magnetization and the repulsive force induced by flow of the melt. This theoretical analysis renders a better understanding of recent experimental results. Additionally, a new method by using strong magnetic field can be proposed to control the particle size distribution for the preparation of metal-matrix composites.

11:15 AM

Investigation into the Effects of LF Bottom Blown Stirring by Two Nozzles: *Zhigang Liang*[1]; [1]Northeastern University

Investigation into the mixing of 150t ladle furnace by two-nozzle jetting was performed through a physical simulation experiment with geometric similarity proportion of 1:4. The effects of nozzle arrangements including separation angle, radial position and asymmetry as well as tracer adding position on the mixing time were studied. The most favorable mixing is achieved under the condition of the two nozzles are symmetrically arranged at half radii in the ladle, with 45° separation angle. The empirical relationship between the mixing time and stirring power intensity has been established for two-nozzle jetting based on the present experimental results. In addition, the swelling above bath surface is also investigated with two-nozzle jetting. Finally, a plant test on industrial scale to compare the different refining effects, including the composition homogenization and desulphurization between the original and optimized nozzle arrangement was also carried out.

11:30 AM

Preparation of a Carbon Free Precast Block for Ladle Lining: *Zhigang Liang*[1]; [1]Northeastern University

A carbon free precast block was prepared by using high-alumina fused corundum as aggregate and fused magnesia, and ultra-fine Al2O3 and ultra-fine SiO2 powders as matrix materials. A small linear expansion for the precast block was achieved through spinel formation from the reaction of MgO with Al2O3. The precast block showed a lower apparent porosity, higher hot modulus of rupture and better slag corrosion resistance compared to the traditional Al2O3-MgO-C brick. Moreover, the practical application of the precast block to the ladle lining proved it to possess a better thermal insulation which could effectively prevent the temperature drop of molten steel during the refining process. It can be predicted that the carbon free precast block could possibly be used as a new type of ladle lining material for the production of low-carbon and ultra-low-carbon steel.

11:45 AM

Vibratory Stress Relief in D-406A Aerospace Alloy: *M. Bilal Khan*[1]; T. Iqbal[1]; [1]School of Chemical and Materials Engineering, National University of Science and Technology

Thermal stress relief by means of a typical heat treatment cycle (660 oC for one hr) is compared with the one achieved through vibratory stress relief at resonant and multiple resonant frequencies for the D-406 HSLA material. Our analysis of the data is based on the micro-hardness profiles measured for the entire length of scales, namely, base metal, heat affected zone (HAZ) and the weld bead itself. The profiles obtained using the two techniques mimic each other, for an equivalent 70% stress relief level. The data are further qualified by impact testing wherein the zone of interest is projected in the retention groove to provide impact by the pendulum striker in the Charpy mode. Higher equivalent energy absorption in the treated specimens confirms the efficacy of the vibratory method. The present work rectifies earlier anomaly where heat treatment has been reported at 310°C for the same material.

Mechanical Behavior of Nanostructured Materials: Plasticity and Deformation Mechanisms at Small Length Scale II

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Wednesday AM — Room: 3024
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Xiaodong Li, University of South Carolina; Ting Zhu, Georgia Institute of Technology

8:30 AM Invited

Strength and Ductility of Nano-Grained Cu With Nano-Scale Twin Bundles: Y. Zhang[1]; N.R. Tao[1]; *K. Lu*[1]; [1]Institute of Metal Research, Chinese Academy of Sciences

By means of dynamic plastic deformation (DPD, with high strain rates) at cryogenic temperature, bulk nanostructured Cu specimens have been prepared, consisting of nano-sized grains embedded with nano-scale twin bundles. The nanostructure characteristics including grain sizes, nano-twin concentration, and grain boundary structure, can be adjusted by subsequent mechanical and thermal treatments such as cold-rolling and annealing. Tensile properties of the as-prepared DPD samples, the cold-rolled samples, and the as-annealed samples have been systematically investigated to reveal the effects of grain size and twin density on strength and ductility. Experimental results revealed that thermal annealing of the cold-rolled DPD samples leads to a superior strength-ductility combination relative to the samples processed via other routes. Analysis of the enhanced tensile properties in these samples was made in terms of grain size effect and nano-twin effect on strength and ductility.

8:50 AM

Epitaxial Nanotwinned Cu Films with High Strength and High Conductivity: *Osman Anderoglu*[1]; Amit Misra[2]; Haiyan Wang[1]; Filip Ronning[2]; Michael Hundley[2]; Xinghang Zhang[1]; [1]Texas A&M University; [2]Los Alamos National Lab

We report on the synthesis of epitaxial (single-crystal like), nanotwinned Cu films via magnetron sputtering. Increasing the deposition rate from 1 to 4 nm/s, decreased the average twin lamellae spacing from 16 to 7 nm. These epitaxial nanotwinned Cu films exhibit significantly higher ratio of hardness to room temperature electrical resistivity than columnar-grain (nanocrystalline), textured, nanotwinned Cu films.

9:05 AM

Nanomechanics of Surface and Interfacial Plasticity in Nanostructured Metals: *Ting Zhu*[1]; Ju Li[2]; [1]Woodruff School of Mechanical Engineering, Georgia Institute of Technology; [2]Department of Materials Science and Engineering, University of Pennsylvania

Dislocation nucleation is central to our understanding of the plastic deformation in nanostructured metals. The free surface and nanostructure interface may act as effective sources of dislocation to initiate and sustain the plastic flow. Here, we develop an atomistic modeling framework to address the statistical nature of dislocation nucleation. Such an approach bridges the timescale gap between atomistic modeling and laboratory experiments by combining transition state theory and atomistic energy landscape exploration. We show dislocation mediated interfacial reactions is the rate-controlling mechanisms in nano-twinned copper, giving rise to an unusual combination of ultrahigh strength and high ductility. Our results also demonstrate a small activation volume associated with surface nucleation dislocation, leading to sensitive temperature and strain-rate dependence of the nucleation stress, and providing an upper bound to the size-strength relation in nanopillar compression experiments.

9:20 AM

Deformation Twinning Behaviors in Al and Cu Single Crystals: *Zhe-Feng Zhang*[1]; W. Han[1]; S. Wu[1]; S. Li[1]; [1]Institute of Metal Research

Twinning is one of the important plastic deformation modes in metals and alloys. The deformation twinning behaviors in various FCC metals and alloys, including pure Cu and Al single crystals, subjected to equal-channel angular pressing (ECAP), were systematically investigated by taking account of crystallographic orientation, stacking fault energy (SFE) and grain size. For Cu and Al single crystals, their orientations were specially designed with one of twinning systems to just match the macroscopic shear deformation of ECAP. For Al single crystal, no deformation twins were observed after one-pass ECAP although a preferential crystallographic orientation was selected for twinning, while for Cu single crystal, profuse deformation twins were found even strained at room temperature and low strain rate. The current experimental results provided clear and comprehensive evidences that SFE, crystallographic orientation have remarkable influence on the deformation twinning behaviors in Al and Cu single crystals.

9:35 AM

Direct Evidence for Detwinning of Nano-Twinned Copper under Low Temperature Deformation: *Yan-Dong Wang*[1]; Wen-Jun Liu[2]; Lei Lu[3]; Yang Ren[2]; Zhi-Hua Nie[1]; Jonathan Almer[2]; Sheng Cheng[4]; Yong-Feng Shen[1]; Peter K. Liaw[4]; Ke Lu[3]; [1]Northeastern University; [2]Argonne National Laboratory; [3]Institute of Metal Research; [4]The University of Tennessee

We report a new mechanism – detwinning, which is found to operate at low temperatures during the tensile deformation of the electrodeposited Cu with a high density of nano-sized growth twins. Both in-situ synchrotron X-ray diffraction methods, i.e., using the three-dimesional (3-D) X-Ray Microscopy with the submicron-resolution white beam and the high-energy X-ray diffraction with the monochromatic beam, provide the direct experimental evidences for detwinning at low temperatures and capture the rich information on the changes of the crystallographic orientation and stress of individual grains. The migration of twin-boundary and other high-angle boundaries under an extremely high stress at low temperatures is distinct from the twinning activity previously observed in metals deformed at low temperatures or during high-temperature annealing. We believe that the detwinning process, as an important deformation mechanism in nanostructured materials, may increase the capacity for accommodation of plastic strain and promote the homogeneous deformation of nanostructured materials.

9:50 AM Invited

Evolution of Intrinsic Stresses in Thin Film Growth via Coupled Surface and Grain Boundary Diffusion: Tanmay Bhandakkar[1]; Eric Chason[1]; *Huajian Gao*[1]; [1]Brown University

In order to explain experimental observations on the evolution of intrinsic stresses during Vomer-Weber growth of thin metal films of high surface mobility, Chason et al. (2002) have proposed a model based on the assumption that a higher chemical potential near the film surface during deposition provides a driving force for a flow of adatoms into the grain boundaries (GB). Here we build upon the previous models of Gao et al. (Acta Mat, 1999), Chason et al. (PRL, 2002) and Guduru et al. (JMPS, 2003) on coupled surface and GB diffusion and extend these models to include the effect of GB diffusion heterogeneity. Our model

considers a layer of active diffusion near the surface during deposition. Inside the active layer, both surface and GB diffusivities are assumed to be higher than their normal values. The simulation results show excellent comparisons with experimentally measured stress evolution in various films.

10:10 AM
Nanoscale Plasticity at Elevated Temperatures in Tantalum (Ta) Single Crystals: *Koteswararao Rajulapati*[1]; Monika Biener[2]; Juergen Biener[2]; Andrea Hodge[1]; [1]University of Southern California; [2]Lawrence Livermore National Laboratory

The deformation behavior of body centered cubic (BCC) materials is very complex and is relatively poorly understood by the scientific community. The main challenge is to understand the operation of the several slip systems active during the deformation in BCC materials. In this study we utilize instrumented nanoindentation in order to investigate the dislocation nucleation and nano scale plasticity of tantalum single crystals with different orientations. Tests were carried out at different loading rates and at different temperatures. The differences between the deformation behavior at room temperature and the deformation behavior at elevated temperatures will be addressed. The associated deformation mechanisms in single crystal tantalum will be explained with the aid of characteristic differences in load-displacement curves and the topographical features of the indents.

10:25 AM Break

10:35 AM
Size-Dependence of Yield Stress in Twinned Gold Nanowires under Uniaxial Tension: *Chuang Deng*[1]; Frederic Sansoz[1]; [1]University of Vermont

In this work, the mechanical properties of [111]-oriented cylindrical gold nanowires containing pre-grown twin boundaries are investigated by molecular dynamics simulations with an embedded-atom method potential. Wires with different diameters (4 nm to 32 nm) and twin-boundary spacings (1.4 nm to 24 nm) are investigated with up to 2 million atoms. We find strong size effects on the yield stress and maximum strength of twinned gold nanowires as a function of both sample diameter and twin boundary spacing. Defect-free nanowires are found to yield at a higher stress as the radius decreases. However, for twinned nanowires, we show that there exists a transition from a decrease to an increase in yield stress as the twin boundary spacing decreases. This crossover effect can provide guidance for the design of nanoscale devices.

10:50 AM Invited
Structural Evolution and Mechanical Response of Nanoporous Noble Metals: Ye Sun[1]; Jia Ye[2]; Andrew Minor[2]; *Thomas Balk*[1]; [1]University of Kentucky; [2]National Center for Electron Microscopy

Nanoporous noble metals exhibit a nanoscale structure of interpenetrating pores and ligaments at a length scale as small as 5 nm. This presentation will focus on microstructural characterization, thin film stress measurements and in-situ nanoindentation in the transmission electron microscope, and will investigate the effects of nanoscale geometric confinement on mechanical properties and dislocation-mediated plasticity. Although some nanoporous films exhibit macroscopic cracking, individual ligament deformation is completely ductile and clearly involves dislocation activity. The film stresses that evolve during thermal cycling correspond to bulk stresses that, according to scaling equations, approach the theoretical strength of the metal. Surprisingly, film stress exhibits a thickness dependence, although the smaller ligament width would presumably govern deformation. This may be due to the finite number of ligament/pore pairs that span the film thickness. These and other observations will be discussed in light of studies on the microstructure and mechanical behavior of nanoporous noble metals.

11:10 AM Invited
Molecular Dynamics Simulations of Shock Compression of Nanocrystalline Nickel: *Marc Meyers*[1]; Hussam Jarmakani[1]; Bruce Remington[1]; Eduardo Bringa[1]; V. Nhon[1]; [1]UC San Diego

Shock compression in nanocrystalline nickel (5 nm and 10 nm grain size) is simulated over a range of pressures (10-80 GPa) and compared with experimental results. Molecular Dynamics simulations are ideal, both in time and length scales, for comparing with laser-shock compression experiments and providing insight on deformation processes involved. Contributions to the net strain from the various mechanisms of plastic deformation such as partials, perfect dislocations, and twins were quantified in the nanocrystalline samples. The effect of release, a phenomenon often neglected in MD simulations, on dislocation behavior is also studied. It is shown that a large fraction of the dislocations generated at the front are annihilated. An analytical model is applied to predict the critical pressures for the cell-stacking-faults transition in single-crystalline nickel and the onset twinning occurs in nanocrystalline nickel. The results resolve a disagreement consistently observed between MD computations and experimental results. Research funding: UCOP ILSA/LLNL.

11:30 AM
High-Velocity Impact Behavior of Ultrafine Grained Tungsten: *Laszlo Kecskes*[1]; Lee Magness[1]; Brian Schuster[1]; Zhiliang Pan[2]; Qiuming Wei[2]; Eric Klier[1]; [1]US Army Research Laboratory; [2]University of North Carolina at Charlotte

Compared to tungsten-based heavy alloy (WHA) penetrators, those fabricated from depleted uranium alloy (DUA) have greater penetration ability. The effectiveness of DUA is attributed to localized shear zones that sharpen its tip during penetration. Based on published evidence of a definite transition in deformation behavior of ultrafine grained (UFG) Fe and W, it was expected that ballistic tests would also reflect this transition from gross plastic to localized shear deformation. Sub-scale projectiles were fired into rolled homogeneous armor steel targets. The targets were sectioned to reveal the embedded projectile remnant, or 'residual', which was metallographically prepared for microhardness measurements, and optical and scanning electron microscopy. Differences in the deformation behavior of the pure UFG W to that of conventional WHA or DUA will be identified and demarcated.

11:45 AM
Dislocation Nucleation Inside Nanoscale Ribbons in Composite Microwires and the Importance for Determining Composite Strength: *David Morris*[1]; Maria Muñoz-Morris[1]; [1]CENIM CSIC

In situ fibre-reinforced composites are prepared by very heavy straining that elongates the ductile minor phase into very fine ribbons, producing extremely high strength. Examples are Cu-bcc metal mixtures and drawn pearlitic steel wires. Strengthening is explained using a rule-of-mixtures approach, modified to take account of the many geometrically-necessary dislocations introduced during formation of the fibre composite or by scale and barrier strengthening. This final term supposes high stresses for dislocation glide inside nanoscale regions and for dislocation sources inside neighbouring regions.Transmission electron microscopy studies have been carried out on a composite wire containing nanoscale Cr ribbons and ultrafine grain Cu matrix. The nanoscale ribbons are initially dislocation free in a matrix too fine to accommodate dislocation substructures. During deformation, dislocations nucleate in the ribbons at sources that are not ribbon size dependent, while randomly-arranged dislocations glide in the matrix. The implications for strengthening are considered.

12:00 PM
Microstructure and Mechanical Property of Gradient Nanocrystalline Cobalt: *Xiao-Lei Wu*[1]; [1]Institute of Mechanics, Chinese Academy of Sciences

A grain-size gradient surface layer on bulk cobalt was generated by means of the technique of surface mechanical attrition treatment. The process of nanocrystalline formation was systematically studied by transmission electron microscopy. The grain refinement was accompanied by the onset of deformation twinning, the operation of prismatic and basal slip, and the successive subdivision of grains to a finer and finer scale, resulting in the formation of highly misoriented nanocrystalline grains. The uniaxial tensile tests were conducted also for the understanding of the deformation mechanisms of gradient nanocrystalline materials.

12:15 PM
Intelligent Viscoelastic Polyurethane Intrinsic Nano Composites: *M. Bilal Khan*[1]; [1]NUST

Polyurethanes are multiphase systems comprising intrinsically variant nanodomains. The material properties can be tailored by adjusting the relative proportions and organizing the structure of the hard and soft segments akin to the spring-dashpot system in an automobile. The paper describes how an intelligent PU system is created to offer smart response to mechanical and viberational stimuli. In this work unidirectional, dynamic (DMTA), acoustic and impact testing results are qualified with the unique viscoelastic charachter that determines the rate-temperature response of the nanocomposite. Attenuated total reflection infrared spectroscopy(ATR-IR) and DMTA offer logical explanation of the observed viscoelastic behavior in terms of the nanodomains. Acoustic and impact attenuation are correlated with the mechanical modulus.

Microstructural Processes in Irradiated Materials: Ceramics and Fuels

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Nuclear Materials Committee
Program Organizers: Christophe Domain, Electricite De France; Gary Was, University of Michigan; Brian Wirth, University of California, Berkeley

Wednesday AM Room: 2008
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Gary Was, University of Michigan; Steven Zinkle, Oak Ridge National Laboratory

8:30 AM Invited
Microstructural Evolution of SiC and ZrC under High Temperature Neutron Irradiation: *Lance Snead*[1]; Yutai Katoh[1]; Sosuke Kondo[1]; Hsin Wang[1]; [1]Oak Ridge National Laboratory

SiC has been used for decades as the pressure vessel and fission product barrier in gas cooled fission reactor TRISO fuels. Under irradiation SiC is quite resilient, as proven by the successful TRISO fuel performance, and both historic and recent studies on the effects of irradiation and irradiation temperature on its mechanical properties. However, two limitations to the SiC-based TRISO system are its maximum temperature, and the poorly understood issue of silver migration through the SiC. ZrC has been suggested and developed as a possible substitute for SiC, or potentially an additive to SiC, to address these issues and to improve the fuel performance. However, the irradiation performance of ZrC is essentially unknown. This paper presents and contrasts microstructural and mechanical property data for high-purity neutron irradiated ZrC and SiC. Irradiations were carried out in the irradiation temperature and neutron dose range of 600-1500°C and 8 dpa, respectively.

9:00 AM
Characterization of Changes in Mechanical Properties of Glassy Polymeric Carbon and Pyrolytic Carbon Following Ion Irradiation – A Comparison: *Malek Abunaemeh*[1]; Bopha Chhay[1]; Cydale Smith[1]; Claudiu Muntele[1]; Yanbin Chen[2]; R. Zhou[2]; Lumin Wang[2]; Gary Was[2]; Daryush Ila[1]; [1]Alabama A&M University; [2]University of Michigan

The TRISO fuel that is planned to be used in some of the Generation IV nuclear reactor designs consists of a fuel kernel of UOx coated in several layers of materials with different functions. Pyrolytic carbon (PyC) is considered for some of these layers. In this study we investigate the possibility of using glassy polymeric carbon (GPC) as an alternative to PyC. GPC is used for artificial heart valves, heat-exchangers, and other high-tech products developed for the space and medical industries. This lightweight material can maintain dimensional and chemical stability in adverse environment and very high temperatures (up to 3000°C). Here we are looking at comparing the changes in physical properties and structures of GPC and PyC after different doses irradiation with 2 MeV proton, 3 MeV N and 5 MeV Si bombardment at 600 and 800 °C by using scanning electron microscopy, transmission electron spectroscopy, microindentation, and three-point bending.

9:20 AM
Phase Stability of Nanostructurally-Stabilized Pure Cubic ZrO2 under Ion Beam Irradiation: *Jie Lian*[1]; Jiaming Zhang[2]; Fereydoon Namavar[3]; Hani Haider[3]; Kevin Garvin[3]; Rodney Ewing[2]; [1]Rensselaer Polytechnic Institute; [2]University of Michigan; [3]University of Nebraska

Zirconia polymorphs display extremely high radiation tolerance and demonstrate no amorphization in bulk sample even under extreme damage level. However, a transformation from monoclinic to tetragonal or cubic phases was induced by irradiation of bulk monoclinic zirconia. In this study, we first report the phase stability of nanostructurally-stabilized pure cubic ZrO2 at room temperature upon 1 MeV Kr2+ irradiation, and pure cubic zirconia was produced by ion beam assisted deposition (IBAD). Cross-sectional TEM examination indicated that there is an amorphous buffer layer (70 nm thick) between Si substrate and cubic nanocrystalline ZrO2 film with an average grain size of 8 nm. Ion irradiation induced a cubic to tetragonal phase transformation for nanocrystalline ZrO2. Furthermore, tetragonal ZrO2 nanocrystals were directly recrystallized from the amorphous layer upon irradiation. These results suggest that tetragonal phase of nanocrystalline ZrO2 are more energetically favorable upon ion irradiation as compared with cubic, monoclinic and amorphous phases.

9:40 AM
Microstructural Evolution in SiC Irradiated above 1273 K in HFIR: *Sosuke Kondo*[1]; Yutai Katoh[1]; Lance Snead[1]; [1]Oak Ridge National Laboratory

Very dense black spots and small loops, which are nano-sized aggregates of displaced Si and C atoms, are the dominating defect microstructures in SiC irradiated in a wide-temperature range of 573-1273K. However, accelerated defect development has been recently demonstrated by authors following neutron-irradiation at >1273K. With the defects which undergo unstable growth, progressive changes in various material properties may commence. This paper reports the recent results of the TEM studies on ß-SiC irradiated at 1273-1733K in HFIR. The dominating defects in the temperature regime are faceted voids and interstitial-type Frank loops. Both the magnitude and the growth rate of void swelling were very limited (<~0.01%) below ~1573K, 9.3dpa, whereas the fluence and temperature dependent increase in the void swelling was observed clearly above ~1673K, and the maximum value was 0.25% at 1733K, 9.6dpa. Of interest results such as unidirectional formation of tetrahedral voids and interstitial diffusivity will also be discussed.

10:00 AM
Structural Modifications in $A_2B_2O_7$ Pyrochlore Induced by Swift Heavy Ions with and without Pressure: *Maik Lang*[1]; Fuxiang Zhang[1]; Jie Lian[2]; Jiaming Zhang[1]; Christina Trautmann[3]; Rodney Ewing[1]; [1]University of Michigan; [2]Rensselaer Polytechnic Institute; [3]Gesellschaft fuer Schwerionenforschung

Pyrochlore, $A_2B_2O_7$, exhibit a variety of properties that find application in a number of different technologies, from electrolytes in solid oxide fuel cells to actinide-bearing compositions that are used as nuclear waste forms and inert matrix fuels. Ion beam irradiations (energy: GeV) have been used to systematically modify the $Gd_2Zr_{2-x}Ti_xO_7$ binary at the nanoscale by radiation-induced phase transitions that include the crystalline-to-amorphous transition and an order-disorder structural transformation to a defect-fluorite structure. Synchrotron XRD, Raman spectroscopy, and TEM provide a consistent understanding of these results of the pure electronic excitation and ionization caused by the heavy ions. When pressure is included as an additional parameter, the response of the pyrochlore structure to these extreme conditions differs significantly. We show that the combination of relativistic ions and pressure can induce novel structural modifications in pyrochlore that cannot be obtained by irradiation or pressure applied separately (e.g., formation of a new phase).

10:20 AM Break

10:40 AM
Effects of Irradiation on the Microstructures of Low-Enriched U-Mo Fuels: *Dennis Keiser*[1]; Jan Fong Jue[1]; Adam Robinson[1]; [1]Idaho National Laboratory

The Reduced Enrichment for Research and Test Reactors (RERTR) program is developing low-enriched U-Mo alloy fuels for application in research and test reactors around the world. A big part of this development effort is actual irradiation testing of a variety of different fuel types in the Advanced Test Reactor (ATR). As part of this testing, microstructural characterization of dispersion and monolithic plate-type fuels is performed before and after irradiation using scanning electron microscopy to help determine the effects of irradiation on the microstructural stability of the different fuels. This talk will discuss the changes that occur in the microstructures of both dispersion and monolithic U-Mo fuel plates that have been fabricated using different techniques. Comments will be made on changes in fuel plate performance that have been observed as a function of changing matrix composition for dispersion fuel plates and changes in fuel/cladding interlayer composition for monolithic fuel plates.

11:00 AM
Kr Ion Irradiation Study of the Depleted-Uranium Alloys: *Jian Gan*[1]; Dennis Keiser[1]; Brandon Miller[2]; Jeffery Rest[3]; Marquis Kirk[3]; Todd Allen[2]; Daniel Wachs[1]; [1]Idaho National Laboratory; [2]The University of Wisconsin; [3]Argonne National Laboratory

Fuel development for the Reduced Enrichment Research and Test Reactor (RERTR) program is tasked with the development of new low enrichment uranium nuclear fuels that can be employed to replace existing high enrichment uranium fuels currently used in some research reactors throughout the world. Radiation stability of the fuel-cladding interaction product has a strong impact on fuel performance. Three depleted uranium alloys were successfully cast for the radiation stability studies of the fuel-cladding interaction product using Kr

ion irradiation to simulate radiation damage from fission products. SEM analysis indicates the presence of the phases of interest: $U(Si,Al)_3$, $(U,Mo)(Si,Al)_3$ and a mixture of UMo_2Al_{20}, $U_6Mo_4Al_{43}$, and UAl_4. Irradiation of TEM disc samples with 500 keV Kr ions at 200ºC to the doses of 1.0 and 10 dpa should provide insight to the microstructural stability of the phases relevant to fuel-cladding products anticipated to form in RERTR dispersion type fuel.

11:20 AM

Microstructural Evolution in Irradiated Uranium-Bearing Delta-Phase Oxides: *Ming Tang*[1]; Kiel Holliday[2]; Yongqiang Wang[1]; James Valdez[1]; Blas Uberuaga[1]; Ken Czerwinski[2]; Kurt Sickafus[1]; [1]Los Alamos National Laboratory; [2]University of Nevada, Las Vegas

Irradiation damage effects in uranium-bearing delta-phase oxides of $A_6U_1O_{12}$ (A = rare earth cations) were characterized using grazing incidence X-ray diffraction and transmission electron microscopy. Polycrystalline $Y_6U_1O_{12}$, $Gd_6U_1O_{12}$, $Ho_6U_1O_{12}$, $Yb_6U_1O_{12}$, and $Lu_6U_1O_{12}$ samples were irradiated with 300 keV Kr^{++} to fluences up to 2×10^{20} ions/m^2 at cryogenic temperature (100 K). The crystal structure of these compounds was determined to be an ordered, fluorite derivative structure, known as the delta phase, a rhombohedral symmetry belonging to space group R-3. Our preliminary results indicate that all these compounds are resistant to amorphization to a displacement damage dose of 50 displacements per atom. In these experiments, we sometimes observed an irradiation-induced order-to-disorder (O-D) phase transformation. We also will discuss the different tendencies of these compounds to experience the O-D transformation. Factors influencing the irradiation damage response of these compounds will be discussed in terms of cation radius ratio and defect formation.

11:40 AM

Proton Irradiation Studies of Depleted-Uranium RERTR Fuels: *Brandon Miller*[1]; Jian Gan[2]; Todd Allen[1]; Dennis Keiser[2]; Dan Wachs[2]; [1]University of Wisconsin-Madison; [2]Idaho National Laboratory

With the concern of fuel-cladding chemical interaction in RERTR dispersion fuels, three depleted uranium alloys were successfully cast to understand their microstructure during irradiation. These alloys simulate the phases that have been seen to form between the fuel and the Al-matrix in various neutron irradiated dispersion fuel experiments. The primary phases present in the three alloys are U(Si,Al)3 and (U,Mo)(Si,Al)3, which are expected to respond well to irradiation, and UMo2Al20, U6Mo4Al43, and UAl4, which are not expected to respond well to irradiation. Irradiations were conducted using 2.6 MeV protons at 200ºC to doses of 0.1, 1.0, and 3.0 displacements per atom, dpa. TEM and XRD studies are being conducted to understand the radiation effects on the microstructure of these alloys. Initial benchmarking been completed and analysis on the proton irradiated alloys is currently being conducted.

12:00 PM

Molecular Dynamics Study of Thermo-Migration of Voids in Single Crystal UO2: *Tapan Desai*[1]; Paul Millett[1]; Dieter Wolf[1]; [1]Idaho National Laboratory

It is well known that within few hours after startup of a nuclear reactor, the temperature gradient within a fuel element causes migration of voids radially inwards to form a central hole. To understand the atomic processes that control thermomigration of voids, we performed molecular dynamics simulations on single crystal UO2 with voids (d=2.2nm). Then, temperature gradient was applied across the simulation cell by supplying additional kinetic energy at the ends and removing it from the center. The system was equilibrated for 1ns at a temperature (T=2800K) well above the oxygen sub-lattice disordering. After a simulation run of 19ns, we found that the voids had moved towards the hot ends. The void mobility is shown to be controlled by the surface diffusion of uranium ions. As the voids migrate, the trailing region on the uranium sub-lattice is completely restored. This work was supported by the DOE-BES Computational Materials Science Network.

Nanocomposite Materials: Characterization and Modeling of Nanocomposites II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee

Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University; Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Wednesday AM — Room: 3020
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Nikhil Gupta, Polytechnic Institute of New York University; Nikhil Koratkar, Rensselaer Polytechnic Institute

8:30 AM Introductory Comments

8:35 AM Invited

Quantitative Morphology Characterization of Polymer Nanocomposites through Electron Tomography: *Lawrence Drummy*[1]; Hilmar Koerner[1]; Richard Vaia[1]; [1]Air Force Research Laboratory, Materials and Manufacturing Directorate

Polymer nanocomposites often display a hierarchical structure that can be accurately described only through the combination of quantitative results from multiple complimentary characterization techniques. Here we compare quantitative analysis of electron tomography results with results from small angle X-ray scattering (SAXS) from the same samples. Nanocomposites were processed by mixing of organically modified montmorillonite (MMT), a layered silicate material functionalized with an octadecylammonium surfactant, and epon 862 epoxy monomer. Electron tomographic reconstruction of HAADF-STEM tilt series data from the nanocompsoites produced a fully segmented 3D data set. The analysis revealed good agreement between tomography and SAXS from the sub-nm regime up to a length scale of 1 micron, however, for certain samples, a representative volume element was not obtained in the electron tomography data. These results are expected to provide a basis for quantitative morphology analysis of nanocomposites and should be applicable to a wide range of materials.

9:00 AM

Nano Holographic Interferometry for Characterization of Nano-Composites: Cesar Sciammarella[1]; Luciano Lamberti[1]; *Federico Sciammarella*[2]; [1]Politecnico di Bari; [2]Northern Illinois University

Properties of nano-composite materials depend not only on the properties of their individual parents but also on their morphology and interfacial characteristics. Currently, a lot of effort is focused on the ability to obtain control of the nanoscale structures via innovative synthetic approaches. Moving down into this regime to measure characteristics to ensure the desired outcome requires the use of expensive and highly sophisticated equipment. A novel approach that uses a conventional far field microscope is presented here as an alternative for measuring nanoscale structures. Going beyond the resolution limits of traditional optics becomes feasible by the use of evanescent wave fronts as a source of illumination. The observed objects here are sodium-chloride nano-crystals. The mean absolute error on the nano-crystals dimensions is 3.06 nm and the standard deviation ±3.7 nm. The measured lengths of the nano-crystals agree very well with integral numbers of the sodium-chloride elementary cell size.

9:20 AM

Morphological and Dielectric Behavior of Carbon Nanotube-Ferroelectric Liquid Crystal Composite: *Deepika Sharma*[1]; [1]Indian Institute of Technology Roorkee, India

The present study deals with the effect of dispersing carbon nano tubes in ferro-electric liquid crystal material. Carbon nanotube dispersed ferroelectric liquid crystals have high dielectric constant, fast switching response, large electro-optic coefficient which makes them ideal for memories, capacitors and display devices etc. An attempt has been made to understand the influence of carbon nanotubes on dielectric and morphological properties of ferro-electric liquid crystal and how the level (wt/wt %) of carbon nanotubes concentration effect the transition temperature of ferro-electric liquid crystal. Hot stage microscope was

used to investigate the texture and phase changes in the composite. Differential Scanning Calorimetry (DSC) confirmed these phase changes. The results are compared with the original ferro-electric liquid crystal material and showed that the dielectric losses are decreased to a great extent.

9:40 AM

Nanocomposite Coatings for Structural Health Monitoring of Materials: Nguyen Nguyen[1]; SengYoon Lee[1]; *Nikhil Gupta*[1]; [1]Polytechnic University

Conducting coatings, containing a network of carbon nanofibers, are developed for structural health monitoring of materials. These coatings can be applied to the material surface or on the structural elements as paints. Applied stress on the material results in strain in the coating and changes its resistance. The resistance of the coating is calibrated with respect to the applied stress. The coatings are optimized for thickness and nanofiber content. In the calibration and validation study the coatings are applied to fiber reinforced laminates. Effect of presence and growth of a crack in the laminate on the coating response is also studied. The results show that the use of nanofibers can provide a low cost and more efficient alternative to other conducting composites which rely on carbon nanotubes.

10:00 AM Break

10:15 AM Invited

Suppression on Fatigue Crack Growth in Carbon Nanotube Composites: *Nikhil Koratkar*[1]; [1]Rensselaer Polytechnic Institute

Fatigue is one of the primary causes for catastrophic failure in structural materials. Here we report an order of magnitude reduction in fatigue crack propagation rate for an epoxy system with the addition of 0.5% weight of carbon nanotube additives. Using fractography analysis and fracture mechanics modeling we show that the crack suppression is caused by crack bridging which results in an effective crack-closing stress due to the pull-out of nanotube-fibers in the wake of the crack tip. Using this model, we show that the suppression of crack growth can be further optimized by reducing the nanotube diameter, by increasing the number density of nanotubes that bridge the crack and by increasing the nanotube pull-out length. Control experiments with nanotubes of different diameters, lengths and dispersion confirmed the model predictions. These results demonstrate that carbon nanotubes can significantly enhance the fatigue-life of structural polymers that are susceptible to fatigue failure.

10:40 AM

Effects of Core-Shell Rubber(CSR) Nanoparticles on the Cryogenic Fracture Toughness of CSR Modified Epoxies: *Jun Wang*[1]; Seth Cannon[1]; Daniel Magee[1]; Judy Schneider[1]; [1]Mississippi State University

This study investigated the effects of core-shell rubber (CSR) nanoparticles on the mechanical properties and fracture toughness of an epoxy resin at ambient and liquid nitrogen (LN2) temperatures. Varying amounts of Kane Ace® MX130 and MX960 toughening agents were added to commercially available EPON 862/Epikure W epoxy resin. Elastic modulus was calculated using quasi-static tensile data. Fracture toughness was evaluated by the resulting breaking energy measured in Charpy impact tests conducted on an instrumented drop tower. The size distribution of the CSR nanoparticles was characterized using Transmission Electron Microscopy (TEM) and Small Angle X-ray Scattering (SAXS). Scanning Electron Microscopy (SEM) was used to study fracture surface morphologies. The addition of the CSR nanoparticles increased the fracture toughness with negligible change in elastic modulus. At ambient temperature the breaking energy increased with increasing additions of the CSR nanoparticles, while at LN2 temperatures, it reached a plateau at lower CSR concentration.

11:00 AM

Effects of Annealing on the Microstructure of Cu-10vol% Alumina Nanocomposite Powders Prepared by High Energy Ball Milling: *Charlie Kong*[1]; Paul Munroe[1]; Aamir Mukhtar[2]; Deliang Zhang[2]; [1]University of New South Wales; [2]University of Waikato

The effects of annealing on the microstructure of a copper matrix nanocomposite with a dispersion of 10vol% alumina nanoparticles prepared by high energy ball milling have been investigated using focused ion beam (FIB) microscopy and transmission electron microscopy (TEM). The ball milled powder was annealed in vacuum at temperatures up to 500ºC. TEM studies of the microstructure of specimens cut from the powder particles using FIB showed no significant changes in the grain size of the copper matrix when annealed at 150ºC, compared to the as-ball milled sample, presumably due to the pinning effects of the alumina nanoparticles, which are typically 20nm in diameter. The average grain size slightly increased from 50nm to 80nm as the annealing temperature increased to 500ºC. However, annealing to this temperature resulted in a significant decrease in dislocation density and the formation of annealing twins, consistent with reductions in the microhardness of the particles.

11:20 AM

Effects of Plasmonic Electric Field in Nano-Metallic Cylinders Chains Lateral and Vertically Coupled on Quantum Dots: *Juan Arias Castro*[1]; Angela Camacho Beltran[1]; [1]Universidad de los Andes

We are interested in the superficial plasmons propagation in a chain of nano-metalic cylinders by studying the size effect and the coupling between the cylinders. Particularly we focus on the main features of electric fields in the inter-cylinder regions due to their relationship with SERS (Surface-Enhanced Raman Scattering). Giant electric fields have been observed in spherical nanoparticles showing an enormous increasing of the cross section, which offers very interesting applications in molecular physics. We discuss the electric field features dependent on geometry and coupling of the nano-metallic particles and extend the results to possible SERS geometric effect. Furthermore, we propose to extend the SERS to quantum dots, which also allows us to study the effect of the plasmons on the quantum dot geometry.

11:40 AM

Processing, Microstructural Characterization and Mechanical Properties of a Ti2AlC/Nanocrystalline Mg-Matrix Composite: *Shahram Amini*[1]; Michel Barsoum[1]; [1]Drexel University

We report on the processing and properties of Ti2AlC/nanocrystalline Mg-matrix composites fabricated by melt infiltration or hot pressing. XRD and TEM both confirmed the Mg grain size was ~35±15 nm. Some Mg was dissolved in the Ti2AlC confirming the existence of a (Ti1-xMgx)2AlC solid solution. A small amount of Ti (3±1 at. %) – postulated to be the nucleating agent of the Mg nano-grains – was also found in the Mg-matrix. This microstructure was also remarkably stable: annealing at 550 °C for 6h did not result in grain growth. At 350±40 and 700±10 MPa, the ultimate tensile and compressive stresses were significantly higher than those of Ti3SiC2-Mg and SiC-Mg composites, in which the Mg-matrix grains were not at the nanoscale. The composites exhibit exceptional damping capabilities. The energy dissipated per cycle per unit volume at 500MPa is believed to be the highest ever reported for a crystalline solid.

Near-Net Shape Titanium Components: Powder Metallurgy I

Sponsored by: The Minerals, Metals and Materials Society, TMS: Titanium Committee

Program Organizers: Rodney Boyer, Boeing Company; James Cotton, Boeing Co

Wednesday AM — Room: 2010
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: James Cotton, Boeing Co

8:30 AM

Powder Metallurgy Titanium Extrusion Billets for Rod Stock: *Jane Adams*[1]; Vladimir Moxson[2]; Volodymyr Duz[2]; Jason Deters[3]; Craig Niese[3]; Christine Suminski[3]; [1]US Army Research Laboratory; [2]ADMA Products, Inc.; [3]General Dynamics Land Systems

Defense vehicle manufacturers use self-tapping threaded mechanical fasteners to mount various components onto the vehicle structure quickly. GDLS' Fredsert® is a unique design in that it has no fixed mechanical locking, which means replacement is easy. Each vehicle uses thousands of inserts that are currently made from aerospace-grade titanium alloys.A high volume powder metallurgy manufacturing process to produce titanium extrusion billets to make rod feedstock for threaded attachment inserts is under evaluation. These aspects will be discussed:1) characterization of titanium and master alloy powders, 2) compaction of the powder into cylinders by cold iso-static pressing and vacuum sintering, 3) microstructure characterization and mechanical testing of performs and extruded bar stock, 4) insert performance comparative testing (tensile, torque, and breakaway) of inserts made from conventional melt-formed industrial titanium stock vs. powder metallurgy titanium, and 5) analysis of properties versus cost and processing factors.

8:50 AM
Development of High Strength Titanium Alloy Bar Stock from TiH2 Powder: *Curt Lavender*[1]; Yuri Hovanski[1]; K. Scott Weil[1]; Vladimir Moxson[2]; Volodymr Duz[2]; Orest Ivasishin[3]; [1]Battelle - Pacific Northwest National Laboratory; [2]ADMA Products Inc.; [3]Institue for Metal Physics National Academy of Science Ukraine

A new method to produce TiH2 for use in blended elemental powder metallurgy has been under development and has been synthesized into many alloys. This paper reports on the results of use the TiH2 powder for the production of the 5Al5Mo5V3Cr and the difficult to cast 1Al8V5Fe alloys via cold iso-static pressing, sintering and rod rolling. Static and dynamic mechanical properties of the as rolled and heat treated materials were characterized by room temperature tensile, compression and fatigue tests. Elastic constants were determined by ultrasonic techniques. This paper will summarize the characterization of the material and discuss the suitability for use in automotive suspension applications.

9:10 AM
Canless Extrusion Process Development for Blended Elemental Powder-Based Titanium Ti-6AL-4V Alloy: *Sami El-Soudani*[1]; Oscar Yu[2]; Fusheng Sun[2]; Michael Campbell[3]; Joshua Phillips[3]; Tony Esposito[3]; Vladimir Moxson[4]; Vlad Duz[4]; [1]The Boeing Company; [2]RTI International Metals Inc.; [3]Plymouth Engineered Shapes; [4]ADMA

The feasibility of canless extrusion in ambient environment of hydride/dehydride blended elemental Ti-6AL-4V ADMA-processed powder previously direct-consolidated by cold isostatic pressing (CIP), followed by vacuum sintering has been successfully demonstrated. Extrusion of these billets was conducted at both RTI International Metals, Inc. and Plymouth Engineered Shapes, Inc. The extrusion processing sequence and parameters were derived separately based on prior extrusion experience at both RTI and Plymouth Engineered Shapes, but were found to be essentially similar to those used for wrought ingot-based Ti-6AL-4V material. Laboratory analysis showed that the canless powder-based billet extrusion processing step conducted in air added no more than 200 ppm oxygen to the as-vacuum-sintered billet oxygen content. Preliminary tensile properties of the blended-elemental ADMA powder-based extrusions of a Ti-6AL-4V composition processed both in the beta or alpha-beta ranges of extrusion temperatures showed equivalent or superior tensile properties as compared to identically processed wrought, ingot-based and extruded Ti-6AL-4V.

9:30 AM
Developments in Die Pressing Strategies for Low-Cost Titanium Powders: *Yuri Hovanski*[1]; K. Scott Weil[1]; Curt Lavender[1]; [1]Battelle - Pacific Northwest National Laboratory

Recent developments in the production of low-cost titanium powders have rejuvenated interest in manufacturing titanium powder metallurgy components by direct press and sinter techniques. However excessive friction typically observed during titanium powder pressing operations leads to numerous problems ranging from non-homogeneous green densities of the compacted powder to excessive part ejection forces and reduced die life due to wear and galling. An instrumented double-acting die press was developed to both investigate the mechanics of titanium powder pressing (particularly for the new low-cost powder morphologies) and to screen potential lubricants that could reduce frictional effects. As will be discussed, the instrument was used to determine friction coefficients and to evaluate a number of candidate lubricants. These results were then used to optimize the lubricant system to reduce die-wall stresses and improve part density uniformity.

9:50 AM Break

10:10 AM
A CIP-HIP Method for the Production of Near Net Shaped Titanium Components: *Richard Dashwood*[1]; Fatos Derguti[2]; Martin Jackson[3]; David Dye[2]; Malcolm Ward-Close[4]; [1]University of Warwick; [2]Imperial College London; [3]University of Sheffield; [4]QinetiQ

The emergence of a number of novel low cost production methods for titanium alloy powders has stimulated significant interest in the near net shape production of titanium components via powder metallurgy. This paper describes work on new route for titanium parts based on a multi-stage forming and consolidation process using both cold and hot isostatic pressing. Commercial purity titanium and Ti-6Al-4V powders of varying particle size distributions were subjected to cold isostatic pressing at a number of compaction pressures using various bagging materials. The resulting compacts were then subject to a special indirect hot isostatic pressing process intended to achieve full material density without the need for expensive metal cans. Different secondary pressing media (SPM) were used to improve pressure transmission and heat transfer and the optimum SPM/workpiece ratio was investigated. The benefits of this process with respect to the microstructure and properties of the final product will be presented.

10:30 AM
LENS™ Deposition of α2+γ+ß2 Structures Based on Ti-Al-Fe: Brian Welk[1]; *Peter Collins*[1]; Mark Gibson[2]; Colleen Bettles[3]; Hamish Fraser[1]; [1]Ohio State University; [2]CSIRO; [3]Monash Univ

Laser Engineered Net Shaping (LENS™) has been used to explore the α2+γ+ß2 of the Ti-Al-Fe system. Three composition gradients have been produced and subsequently heat-treated to effect variations in microstructure. The properties have been measured along the gradients. State of the art characterization tools, including SEM, FIB, (S)TEM, EDS and EELS, have been used to explore the microstructural variations as a function of composition. The results are correlated with alloy composition.

10:50 AM
Enhancement of Densification Kinetics of Ti-6Al-4V Powders by Thermal Cycling: Bing Ye[1]; Marc Matsen[2]; Wesley Crow[2]; Lee Firth[2]; *David Dunand*[1]; [1]Northwestern University; [2]Boeing Co

The densification of Ti-6Al-4V powders is investigated in uniaxial die pressing experiments carried out isothermally at 1000 C (in the beta-field of the alloy) and during thermal cycling between 800 and 1000 C (about the alpha/beta phase transformation range of the alloy). Thermal cycling enhances densification kinetics because transformation-mismatch plasticity becomes the dominant densification mechanism, replacing dislocation creep active for isothermal densification. Cyclic and isothermal hot-pressing densification data for various processing parameters (temperature range, thermal cycling rate and compaction pressure) are compared with analytical and numerical finite-element models of powder densification. The microstructure and mechanical properties of the fully densified Ti-6Al-4V samples are also studied and compared to baseline values.

Neutron and X-Ray Studies of Advanced Materials: Phase Transition

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Wednesday AM — Room: 3016
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Dean Haeffner, Argonne National Laboratory; Ralph Gilles, TU München

8:30 AM Keynote
Characterization of Structure and Dynamics in Metallic Glasses by Scattering: *Takeshi Egami*[1]; [1]University of Tennessee

Whereas the structure of glasses can be modeled by a computer, understanding the salient and relevant features of the model requires a special consideration. But we have not gone much beyond using the nearest neighbor distance, coordination number and the topology of the local clusters, and it is not easy to link them to real properties. In this talk I propose to consider the width of the first peak of the atomic pair-density function (DPF) and its anisotropy as an important measurable parameter. It is related to the local atomic-level strains, and we now know how it depends upon temperature. The relations to structural relaxation, glass transition, mechanical deformation and other properties will be demonstrated. 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 DE-AC05-00OR-22725 with UT-Battelle.

9:00 AM Invited

Probing the Relationship of Ordering in Antiphase Nanodomain FeCo Alloys with Ternary Additions Using Neutron Diffraction: *Ralph Gilles*[1]; Michael Hofmann[1]; Yan Gao[1]; Frank Johnson[2]; Luana Iorio[2]; Markus Hoelzel[3]; Bruno Barbier[4]; [1]TU München; [2]GE Global Research; [3]TU Darmstadt / FRM II; [4]Universität Bonn

FeCo alloys are industrially important engineering materials due to their very high saturation magnetization and Curie temperature. These alloys play an important role in applications requiring soft magnetic materials, such as electrical generators, motors and transformers. For many industrial applications, the challenge involves increasing the tensile strength and ductility of FeCo alloys while maintaining magnetic performance. Methods used to meet this challenge include alloy design (eg. addition of certain ternary metals such as Ni, V, Nb, Ta, Cr, Mo etc.), annealing, and advanced deformation processing. However, modern applications require even better mechanical and magnetic performance. The effects of alloying FeCo with Pt, Pd, Mn, Ir, and Re have been investigated using neutron diffraction as part of this work. In the composition range of about 30% - 70% Co, FeCo alloys undergo a continuous order-disorder phase transformation at a maximum temperature of 730°C at the equiatomic composition.

9:20 AM Invited

Polarization Switching in Ultrathin Ferroelectric Film by Changing the Chemical Environment: *Carol Thompson*[1]; Matthew Highland[2]; Dillon Fong[2]; Jeffrey Eastman[2]; Paul Fuoss[2]; Timothy Fister[2]; Stephen Streiffer[2]; G. Brian Stephenson[2]; [1]Northern Illinois University; [2]Argonne National Laboratory

We have used grazing-incidence x-ray scattering to show that polarization orientation in an ultrathin ferroelectric film can be switched through control of the chemical environment above the surface. Monodomain epitaxial PbTiO3 films of typical 5 nm thickness are grown by metal-organic chemical vapor deposition onto conducting SrRuO3/SrTiO3 (001) substrates. Our experiments have found that changing the partial pressure of oxygen in the gas above the film can induce inversion in the sign of the polarization. At lower temperatures in thicker films, switching occurs by the mechanism of nucleation and growth of 180o domains, while at higher temperatures in thinner films the polarization switching is continuous; i.e., the polarization magnitude decreases to zero and changes sign uniformly without domain formation. Work supported by the U. S. Department of Energy under Contract No. DE-AC02-06CH11357.

9:40 AM

Multi-scale Analysis during the Mechanically-Induced Martensite Phase Transformation by Synchrotron X-Ray Radiation and Neutron Diffraction: *Benoit Malard*[1]; Guillaume Geandier[2]; Jon Wright[3]; Sophie Berveiller[4]; Etienne Patoor[4]; [1]Institute of Physics; [2]LMP - SP2MI; [3]ESRF; [4]LPMM

Martensitic transformation in Shape Memory Alloys (SMAs) can be induced and controlled by external stress and temperature. The transformation is accompanied by the reversible evolution of large high successive inelastic strains. This paper summarizes three in-situ experimental studies of the superelastic behaviour associated to stress induced martensite transformation in Cu-12%Al-0.5%Be [wt. %] polycrystalline samples with the complementarities between synchrotron X-ray radiation and neutron diffraction on three different length scales: 1. The macroscopic scale involves the analysis of small-grain alloys. A new experimental method to determine in-situ the deformation average strain will be explained. 2. The mesoscopic scale takes us down to in the grain to measure the evolution of the rotation in individual grains with different orientations with the 3DXRD method. 3. In the microscopic scale, the changes of grain orientation inside the austenite between two martensitic variants were studied by synchrotron x-ray microdiffraction.

9:50 AM

High Pressure Deformation of Zirconium: Sven Vogel[1]; Donald Brown[1]; N. Nishiyama[2]; Helmut Reiche[1]; Thomas Sisneros[1]; Heather Volz[1]; Yanbin Wang[2]; Yusheng Zhao[1]; *David Weldon*[1]; [1]Los Alamos National Laboratory; [2]Argonne National Laboratory

In situ deformation studies using diffraction have become routine for materials stable at ambient conditions. However, some materials only exist in extreme conditions, leaving in situ devices the only means to study their structure and mechanical properties. Here, we report results of uni-axial deformation studies of zirconium at hydrostatic pressures of several GPa. The experiments were performed at the BM13 beam line at the Advanced Photon Source. The deformation-DIA apparatus generates a confining hydrostatic pressure causing a phase change (α-Zr to ω-Zr). Uniaxial deformation is superposed on the hydrostatic pressure to obtain the mechanical response at pressure. The applied stress is measured from the lattice response while the macroscopic strain is determined from radiography. Deviations from Debye rings from circular allow for determination of the full stress tensor, that is de-convolution of the hydrostatic and uniaxial components and intensity variations along the ring allow quantitative texture analysis.

10:05 AM

In-Situ Study on Phase Transition of NiMnGa Alloy under Magnetic Field by Synchrotron High-Energy X-Ray Diffraction: *Gang Wang*[1]; Yandong Wang[1]; Yang Ren[2]; Yandong Liu[1]; Liang Zuo[1]; [1]Northeastern University; [2]Argonne National Laboratory

Full information on crystallographic aspects during phase transition under magnetic field is essential for understanding the effect of magnetic field on the 'memory' characteristics in the ferromagnetic shape-memory alloys (FSMA) related to texture and stress. In the present paper, the detailed local information of the microstructural evolution of NiMnGa alloy under magnetic field was measured in-situ on the high energy synchrotron beam line 11-ID-C of APS. The transformation between parent phase and martensitic phase was traced by means of the instantaneously recorded 2D images of diffraction results. According to these results, the mechanism of phase transition of NiMnGa under magnetic field is concluded, which can enrich our knowledge for controlling the microstructure and performances of FSMA.

10:20 AM Break

10:25 AM Invited

Industrial Applications of X-Ray Characterization for Advanced Materials: *Assunta Vigliante*[1]; [1]Bruker-AXS

Films of few nanometers thicknesses, quantum dots, quantum wires are the basis of the modern electronic industry and X-ray diffraction techniques is starting to play a very important role as a basic characterization tool for determining the detailed structural information of ultra-thin film such as: the evolution of strain relaxation, defect formation, interfacial properties between film/substrate, the effects of the reduced dimensionality and their correlation to the electrical properties. Materials of technological interests are: high and low K dielectric materials which will substitute SiO2, materials for interconnects, new materials for memory storage, materials for spintronics. Traditional optical and opto-acustic metrology methods, which have been used in industry for decades, are being strongly challenged to meet the new characterization requirements of complex materials. In this talk, I will give an overview of the state-of-the-art of commercial laboratory instrumentation and applications to the electronic industry.

10:45 AM

Atomic-Scale Studies of Phase Transition in Ni-Mn-In Nanoparticles: *Zhihua Nie*[1]; Dongmei Liu[1]; Yang Ren[2]; Yandong Wang[1]; Gang Wang[1]; Dennis Brown[3]; Liang Zuo[1]; [1]Northeastern University, Key Laboratory for Anisotropy and Texture of Materials (MOE); [2]X-ray Science Division, Argonne National Laboratory; [3]Department of Physics, Northern Illinois University

The Ni-Mn-In ferromagnetic shape-memory alloys have received great interest due to their potential applications as magnetic-field-driven actuators or sensors. The shape memory effect, large magnetoresistance and magnetocaloric effect have been reported in the ternary or Co-doped quaternary alloy systems, which is due to the magnetic field-driven reversible transition from martensite to Heusler parent phase, associated with a ferro- to antiferro- magnetic transformation. The NiMnIn nanoparticles were prepared by a high-energy ball-milling method. The atomic-scale study of phase transition in Ni50Mn34In16 nanoparticles were carried out using the high-energy X-ray diffraction and the atomic pair distribution function (PDF) method. Our results show that the as-milled nanoparticles were of highly disordered cubic structure at room temperature, which can dramatically change to the ordered Heusler structure at 523 K with a large heat-release. This disorder-to-order transition is highly temperature sensitive and the annealed nanoparticles are tailored to 14M modulated structure at room temperature.

11:05 AM
Precipitation Behaviour of Fe-25wt%Co-15wt%Mo Investigated by In-Situ SANS and Complementary Methods: *Elisabeth Eidenberger*[1]; Erich Stergar[1]; Thomas Schmölzer[1]; Harald Leitner[2]; Peter Staron[3]; Helmut Clemens[1]; [1]Montanuniversität Leoben; [2]CD Laboratory "Early Stages of Precipitation"; [3]GKSS Research Centre Geesthacht

The precipitation of a nano-scaled intermetallic phase in a martensitic Fe-25wt%Co-15wt%Mo alloy was investigated by in-situ small-angle neutron scattering (SANS). Solution annealed samples were heated to 700°C using different heating rates while successively measuring the scattered intensity. The size distribution and volume fraction of precipitates as well as the ratio of nuclear and magnetic scattering cross-section, sensitive to changes in the chemical composition of precipitates, were analyzed. Differential scanning calorimetry (DSC) was performed using identical heating rates to gain knowledge of the kinetics of the precipitation reaction. Complementarily, three-dimensional atom probe (3DAP) measurements were conducted to characterize size, shape, and chemical composition of the precipitates. A combination of the results obtained by SANS, DSC, and 3DAP leads to a thorough understanding of the precipitation reaction in the investigated alloy. Specifically, the question if spinodal decomposition takes place in this alloy is addressed.

11:15 AM
In-Situ Synchrotron Investigations and Finite Element Modeling of Microstrains and Mesoscopic Phase Interfaces During Localized Deformation of Pseudoelastic NiTi Shape Memory Alloys: *Martin Wagner*[1]; Marcus Young[1]; Christian Grossmann[1]; Jan Frenzel[1]; Susanne Gollerthan[1]; Mahamudul Hasan[2]; Wolfgang Schmahl[2]; Gunther Eggeler[1]; [1]Ruhr-University Bochum; [2]Ludwig-Maximilians-University Munich

NiTi Shape memory alloys are used in various biomedical and actuator applications. Pseudoelastic materials (in particular wires that possess favorable textures) can recover macroscopic strains of the order of 10 % by virtue of a reversible stress-induced martensitic phase transformation. We report on an in-situ synchrotron diffraction study during tensile straining of pseudoelastic NiTi, where the deformation is associated with the formation of distinct transformation bands. The diffraction data allow documenting how localization of transformation / deformation is related to the macroscopic stress-strain behavior. Moreover, we characterize microstrains in transformation bands and in the adjacent austenitic regions, and we perform a detailed analysis of shapes and widths of boundaries between martensitic bands and austenite. Synchrotron data and complementary finite element simulations of these mesoscopic phase interfaces demonstrate how phase fractions and strain gradients change more sharply in the bulk of a specimen than near the surface.

11:30 AM Invited
Modeling Ductility and Failure Modes of Dual Phase Steels Using Phase Properties Characterized by In-Situ High Energy X-Ray Diffraction: *Xin Sun*[1]; Kyoo Sil Choi[1]; Yang Ren[2]; Yandong Wang[3]; [1]Pacific Northwest National Laboratory; [2]Argonne National Laboratory; [3]Northeastern University

Ductile failure of dual phase steels is predicted in the form of plastic strain localization resulting from the incompatible deformation between the harder marternsite phase and the softer ferrite matrix. Failure modes and ultimate ductility of two dual phase steels are analyzed based on the actual steel microstructures. The plastic work hardening properties for the constituent phases are determined by the in-situ synchrotron-based high-energy X-ray diffraction technique. Under different loading conditions, different failure modes and ultimate ductility are predicted in the form of plastic strain localization. It is found that the local failure mode and ultimate ductility of DP steels are closely related to the stress state. Under plane stress condition with free lateral boundary, one dominant shear band develops and leads to final failure of the material. However, if the lateral boundary is constrained, splitting failure perpendicular to the loading direction is predicted with much reduced ductility.

11:50 AM Invited
The Effect of Secondary Gamma-Prime on the Primary Creep Behavior of Single Crystal, Ni-Base Superalloys: *Gerhard Fuchs*[1]; Brandon Wilson[1]; [1]University of Florida

Some second, third and fourth generation single crystal Ni-base superalloys (i.e., Re-containing alloys) have demonstrated a propensity for excessive primary creep at intermediate temperatures. This behavior has been attributed to the presence of secondary gamma-prime precipitates in the gamma matrix channels, as well as on the Re content of the alloys. This investigation examined creep behavior for a common first generation alloy, PWA 1480, a common second generation alloy, PWA 1484, as well as an modified first generation alloy, PWA 1480 with 3 weight percent rhenium added. Additionally, two different aging heat treatments were given to each alloy to either precipitate or prevent the formation of, fine (nm scale) secondary gamma-prime in the gamma channels. The microstructures of these samples were characterized by SEM, TEM, XRD and LEAP. The role of the fine scale microstructure and the alloy composition on primary creep deformation will be discussed.

12:10 PM
Stress-Induced Martensitic-Transformation Behaviors in Ni-Co-Mn-In-Polymer Composites: *Dongmei Liu*[1]; Zhihua Nie[1]; Yang Ren[2]; Guoshuai Zhou[1]; Peter K. Liaw[3]; Yandong Wang[1]; [1]Northeastern University; [2]X-ray Science Division, Argonne National Laboratory; [3]The University of Tennessee

The ferromagnetic shape-memory composite has been prepared with the Ni-Co-Mn-In particles embedded in a polymer matrix. $Ni_{45}Co_5Mn_{36.6}In_{13.4}$ particles ~30 μm were obtained by a mechanical ball-milling process. The martensitic phase-transformation temperature of the composite is around room temperature. The stress-induced martensitic transformation in the composite was studied by the in-situ high-energy synchrotron x-ray diffraction technique under the uniaxial compressive deformation. After a stress of 50 MPa was applied, the textured martensite with the 14 M modulated structure appeared, and the volume fraction raised with increasing the stress, characterized by preferred distributions of martensitic twin variants with the (0 4 0) and (0 4 14) crystallographic planes parallel to the loading direction (LD) and the (1 3 0) and (0 1 23) planes perpendicular to the LD. The applied magnetic field can certainly lead to the revised phase transition from the martensite to parent phase in the deformed Ni-Co-Mn-In composite.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: 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: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Wednesday AM — Room: 2020
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Iver Anderson, Iowa State University; Darrel Frear, Freescale Semiconductor

8:30 AM Invited
Microstructure and Reliability Comparison of Different Pb-Free Alloys Used for Wave Soldering and Rework: *Polina Snugovsky*[1]; Craig Hamilton[1]; Zohreh Bagheri[1]; [1]Celestica

This paper will describe the results of a intensive microstructural and reliability study of PTH and SMT components which were wave solder assembled using various Pb-free alloys. Both primary attach and reworked solder connections using solder fountain and hand rework were studied. The PTH connector types and SMT resistors were assembled on a test vehicle using the Sn-Ag-Cu, Sn-Cu-Ni-Ge, Sn-Ag-Cu-Bi, Sn-Cu-X, and Sn-Pb solders. Accelerated thermal cycling was conducted at 0 to 100°C. The difference in microstructures, intermetallic formation, Cu dissolution, grain coarsening, and crack formation will be shown. The influence of the microstructure after assembly and rework on Weibull plot parameters and failure mode will be described. Interconnect defects such as non-uniform phase distribution, and void formation will be discussed. Recommendations on alloy applications will also be given.

8:50 AM
Effect of Surface Finish, Package Size, and Rework on Pb-free Solder Ball Grid Arrays During Thermal Cycling: *Fengjiang Wang*[1]; Matt O'Keefe[1]; [1]Missouri University of Science and Technology

The current transition to Pb-free solders raises issues for high reliability applications, such as military and aerospace, where there is a lack of long

term field service data to predict performance lifetime. This work focused on the performance of Sn-3.0Ag-0.5Cu (SAC) Pb-free solder ball grid array (BGA) packages soldered to a high Tg polyimide printed circuit board during temperature cycling. Parameters varied included of the board surface finish, electroless nickel immersion gold (ENIG) vs. Sn-Pb hot air solder level (HASL), the size and location of the BGA components on the board, and whether the BGAs were as-reflowed or removed and replaced (reworked). Results indicate there was no measurable difference in performance with surface finish but SAC, as-assembled, larger BGAs had fewer failures than Sn-Pb, reworked, and smaller BGAs. After thermal cycling assemblies were cross-sectioned to determine failure mechanisms and changes in microstructure.

9:05 AM

Effects of Sn Orientation on Stress Evolution during In-Situ Thermal Cycling of SAC Ball Grid Array Solder Joints from Synchrotron Measurements: *Thomas Bieler*[1]; Tae-Kyu Lee[2]; Kuo-Chuan Liu[2]; [1]Michigan State University; [2]Cisco Systems, Inc.

The stress-strain histories in each joint in a lead-free solder joint array in a package differs depending on the Sn crystal orientations and the location within the array. A slice containing a single row of solder joints was thermally cycled from 0 to 100°C in a period of about an hour with concurrent acquisition of Laue patterns using synchrotron radiation, which indicated that most joints are single or multi-crystals with no more than a few Sn grain orientations. Laue patterns were analyzed using Fit2D, Maud, and Beartex to obtain orientation distribution functions as well as the state of stress in each orientation, allowing the volume fraction and stress history to be tracked in each Sn grain. The same specimens were subsequently given several hundred additional thermal cycles and measured in the synchrotron again to assess how the stress history, volume fraction of Sn phases, and precipitate size evolved.

9:20 AM

A Phenomenological Study of the Effect of Microstructural Evolution on Pb-Free Solder Joint Fatigue Resistance: *Richard Coyle*[1]; Claire Ryan[2]; Steven Kummerl[3]; Peter Read[1]; Michael Reid[2]; [1]Alcatel-Lucent; [2]Stokes Institute, Limerick University; [3]Texas Instruments

This paper presents a phenomenological study of the relationship between the initial Sn-Ag-Cu (SAC) solder joint microstructure, the evolving microstructure, and the thermal fatigue performance during accelerated temperature cycling (ATC). Commercial SMT components with different SAC alloy compositions are evaluated using daisy chained test vehicles. The initial microstructures are altered by varying the solder joint cooling rate, using a single or double pass SMT reflow, and with isothermal preconditioning. Different ATC temperature extremes and dwell times are used to vary the rate of microstructural evolution during testing. The microstructural evolution is tracked and characterized with optical metallography and scanning electron microscopy. Unlike SnPb solders, the thermal fatigue reliability of the SAC solders is influenced significantly by both the initial and evolving microstructures. These results could have practical implications in terms of limiting the ability to develop acceleration factors and effective strain-based models for predicting Pb free solder joint life.

9:35 AM

The Effect of Ni Additions on the Impact Strength of Tin-Copper and Tin-Silver-Copper Lead-free Solders: *Keith Sweatman*[1]; Shoichi Suenaga[1]; Masuo Koshi[1]; Tetsuro Nishimura[1]; [1]Nihon Superior Co., Ltd.

The problem of failure of near eutectic SnAgCu alloy joints to area array components when portable devices such as cell phones are accidentally dropped has prompted consideration of alloys with lower Ag levels with and without microalloying additions. The authors report a study of a range of lead-free alloys that explore these options including SAC305+Ni, SAC1205, SAC1205+Ni and Sn-0.7Cu-0.05Ni+Ge in high speed shear and pull testing. The results for these alloys are benchmarked against those for the tin-lead eutectic and SAC305. 0.5mm spheres were reflow soldered to solder-mask-defined pads on OSP and ENIG substrates and fracture energies measured in shear at speeds up to 2000mm/sec and in tension at speeds up to 200mm/sec. Failure modes were characterised by the fracture surfaces and overall performance correlated with the joint microstructure. Impact strength was found to be sensitive to both the alloy composition and the substrate.

9:50 AM

The Microstructure and Crystal Orientation of Sn-Ag and Sn-Cu Solders Affected by their Interfacial Reactions with Cu and Ni(P): *Sun Kyoung Seo*[1]; Sung K. Kang[2]; Moon Gi Cho[1]; Da-Yuan Shih[2]; Hyuck Mo Lee[1]; [1]KAIST; [2]IBM T.J. Watson Research Center

Recently, it has been reported that the crystal orientation and grain size of the β-Sn phase in Sn-rich solders have profound effects on the reliabilities of Pb-free solder joints, such as thermo-mechanical fatigue, electromigration, and among others. It is also known that the microstructure of Sn-rich solders is strongly affected by their alloy composition. In this study, the grain size and orientation of the β-Sn phase are investigated in terms of their alloy composition and interfacial reactions with two different under bump metallurgy (UBM), Cu vs. Ni(P). The Cu content investigated varies from 0.5 to 2.0wt% in Sn-Cu, while the Ag content varies from 0.5 to 3.5wt% in Sn-Ag. After reflowed at 250°C for 2min, the microstructure of solder joints is analyzed by cross polarization light microscopy and electron backscatter diffraction (EBSD). In addition, the microstructure changes of both solders are examined after aging at 150°C for 1000h.

10:05 AM

Heterogeneous Intragranular Inelastic Behaviour of an SnAgCu Alloy: Jicheng Gong[1]; *Paul Conway*[2]; Changqing Liu[2]; Vadim Silberschmidt[2]; [1]University of Oxford; [2]Loughborugh University

Sub-100μm scale SnAgCu joints may be formed with only one or a few grain. Its mechanical behaviour shifts from polycrystalline aggregate to single crystal. The latter exhibits anisotropic properties since the ß-Sn matrix has a contracted body-centred tetragonal structure. Shear tests have shown one slip system is activated in each grain, indicating lattice-dependent inelastic behaviour. The simple shearing may not be the only mechanism for intragranular behaviour. To capture the intragranular deformation principles, micromechanical behaviour has been investigated for SnAgCu grains under low loading. A reflowed alloy is machined to a block of 25x5x2mm. One surface is ground, polished and examined under polarized light to characterize the structure. FIB milling is used to prepare microspecimens that are tested on an Instron 5848 with a low loading rate. TEM is employed to study crystallographic characteristics within a grain to examine the evolution of the substructure and dislocations.

10:20 AM Break

10:35 AM

Impact Behavior of Thermomechanically Fatigued Sn-Based Solder Joints: *K. Subramanian*[1]; Andre Lee[1]; T. Kobayashi[1]; [1]Michigan State University

Most of the accidental impact loading of electronic packages occurs after they have experienced different extents of thermal, electrical and mechanical field influences encountered during service. In order to evaluate the effects of thermal field influences on the impact behavior of Sn-based solders with and without Pb, studies were carried out after subjecting these joints to isothermal aging at different temperatures, and thermomechanical fatigue in different temperature regimes. Roles of the microstructural features and thickness/morphology of interface IMC layers resulting from imposed thermal field influences, and the type of loading, on failures resulting from impact were evaluated to understand the effects of such fields on the impact reliability of electronic components.

10:50 AM

The Effects of Nucleation and Solidification on the Fatigue Life of Pb-free Solders: *Babak Arfaei*[1]; Yan Xing[1]; Peter Borgesen[2]; Jim Woods[1]; Jeremy Wolcott[1]; Pushkraj Tumne[1]; Eric Cotts[1]; [1]Binghamton University; [2]Unovis Solutions

Variations in the nucleation behavior of near eutectic Sn-Ag-Cu alloys result in significant differences in both the Sn grain and precipitate morphologies of solidified solder joints. These microstructural factors profoundly affect the thermomechanical response of SnAgCu Pb free solder alloys. The present study utilized differential scanning calorimetry to examine the nucleation behavior of Pb-free solders of various compositions and sizes, on different metallizations and substrates. Individual solder balls were tested in a load controlled shear fatigue test. The microstructure of cross sectioned samples was characterized by means of electron backscattered diffraction in a scanning electron microscope. The effect of solidification temperature and number of reflows on the number of precipitates and grain orientation was studied. The variations of fatigue life of SnAgCu solder joints with both Sn grain size and orientation, and with secondary precipitate size and number, are reported.

11:05 AM
Influence of Thermal Cycling on Thermal Resistance of Solder Interfaces: J. Guo[1]; H. Guo[1]; *J. Shang*[2]; [1]Institute of Metal Research; [2]University of Illinois at Urbana-Champaign

Solder alloys are attractive thermal interface materials for thermal management in microprocessors because of their excellent thermal conductivity. However, solder interfaces with the device metallization or heat spreader are prone to damage by thermal cycling. In this study, the effect of thermal cycling on thermal resistance of solder interfaces was investigated by comparing interface thermal resistance at various stages of thermal cycling. The thermal resistance was found to increase with thermal cycling. The increase was related to formation of the interfacial defects.

11:20 AM
Improvement of Thermal Fatigue at Sn-Ag-Cu Sysytem Alloy by Addtion of Bi: *Minoru Ueshima*[1]; [1]Senju Metal Industry Co. Ltd.

The Sn-3Ag-0.5Cu alloy have been already put to practical use, and the developement of high reliability solder alloy is expected for automobile. Indeed the crack penetrate the Sn-3Ag-0.5Cu solder fillet of 6432 chip resister(6.4mm length, 3.2mm width) after the thermal fatigue of 2000 cycles between -55 and +125 degrees centigrade at 30 minutes holding. And then the fine needle Ag3Sn became coarse at the crack neighborhood. In this study the crack generation after the thermal fatigue at Sn-Ag-Cu system Alloy is restricted by addition of Bi and then the mechanical properties of Sn-Ag-Cu-Bi system alloys are researched at the room temperature and +125 degrees centigrade. At Sn-3Ag-1Cu-3Bi alloys, the crack doesn't penetrate the solder fillet after 3000 cycles and the weibull distribution of the shear strength is equivalent to that of Sn-3Ag-0.5Cu alloy after 1000 cycles. Sn-3Ag-1Cu-3Bi alloy is recomended for automobile and the power device.

11:35 AM
The Influence of Solder Composition on the Impact Strength of Lead-Free Solder BGA Joints: *Hideaki Tsukamoto*[1]; Kazuhiro Nogita[1]; Stuart McDonald[1]; Tetsuro Nishimura[2]; Shoichi Suenaga[2]; Keith Sweatman[2]; [1]University of Queensland; [2]Nihon Superior Co. Ltd.

At the interface between Sn-based solders and substrates, intermetallic compounds (IMCs) form and grow during soldering operations and subsequent use. Such IMCs provide a metallurgical bond, and their interface microstructure has a critical effect on solder joint mechanical reliability. In the present study, the impact strength of BGA solder joints between (i) Cu substrates and Sn-Cu solders and (ii) Cu substrates and Sn-Cu-Ni solders has been evaluated using shear and tensile ball tests and Finite Element Modelling. In both tests, connections made using Sn-Cu-Ni solders show consistently better properties than those made using Sn-Cu solders, particularly at high displacement rates. Microstructural analysis and fractography are used to interpret the results and develop the FEM.

11:50 AM
Mechanism Solder Joint Failure under Cyclic Bending Fatigue: *Woong Ho Bang*[1]; Liang-Shan Chen[1]; Choong-Un Kim[1]; Tae-Kyu Lee[2]; Kuo-Chuan Liu[2]; [1]University of Texas at Arlington; [2]Cisco Systems

The mechanical stability of a solder joint is one of the primary concerns in microelectronics as it determines the reliability of assembled devices and thus has been extensively investigated. Such studies have examined various factors affecting solder joint stability, including joint metallurgy, joint geometry and testing conditions. Recently, there is a growing research interest on new types of load conditions that impart significant impact to solder reliability. One of such is the fatigue incurred by cyclic loading on solder joints. We have investigated the failure mechanics of solder joint under cyclic bending fatigue, and part of our results will be presented in this presentation. Particular focus of this study is the dependence of fatigue failure on bending cycle frequency (at high speed regime) and the mechanics leading to such a dependency.

12:05 PM Invited
Mechanical Shock of Environmentally-Benign Pb-Free Solders: Kyle Yazzie[1]; Jason Williams[1]; *Nikhilesh Chawla*[1]; Hanqing Jiang[1]; [1]Arizona State Univ

Pb-free solder alloys are routinely subjected to mechanical shock and drop conditions in service. While these solder alloys are somewhat susceptible to dynamic loading, a fundamental understanding of mechanical shock and vibration fatigue is lacking. Existing methods developed to probe this strain-rate regime do not provide a direct and local measurement of strain in the solder. Quantifying the contributions of intermetallic thickness and solder microstructure to the mechanical shock behavior of the solder specimen is extremely important and needs to be studied. In this study the mechanical shock behavior of pure Sn and Sn-3.5Ag-0.7Cu solders was systematically quantified. Intermetallic thicknesses and solder microstructures of individual solder specimens were varied to elucidate their individual contributions to mechanical shock resistance. The strain distribution and evolution was measured using a novel high-speed camera. Multiscale modeling of the complex stress state experienced by the solder during mechanical shock was conducted and will be discussed.

12:25 PM
Reliability Assessment of Interconnects by an Accelerated Mechanical Fatigue Testing Technique: *Golta Khatibi*; Witold Wroczewski[1]; Agnieszka Betzwar Kotas[1]; Brigitte Weiss[1]; Thomas Licht[2]; [1]University of Vienna; [2]Infineon Technologies AG

A mechanical fatigue testing method is presented for rapid evaluation, lifetime determination and early failure detection of interconnects consisting of a special experimental set-up in combination with an ultrasonic fatigue testing system and laser doppler vibrometery. This technique allows testing various interconnect types under shear, tension-compression and combined loading modes. Validity of the mechanical fatigue testing as an alternative to time consuming thermal cycling is demonstrated. Using this technique lifetime curves up to 10^9 cycles for Al wire bonded interconnects in high power electronic devices were determined which showed a good correlation to thermal fatigue life of similar bonds obtained by power cycling tests. In conformity with the main failure mechanism of power cycling tests, wire bond lift-off could be reproduced. Application of the system in combination with vibrational analysis of the bonds for early failure detection is as well demonstrated and confirmed by the micro-sections of tested interconnects.

Peirce-Smith Converting Centennial Symposium: Short Course on Injection Phenomena in the Peirce-Smith Converter

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS: Pyrometallurgy Committee
Program Organizer: Joël Kapusta, Air Liquide

Wednesday AM — Room: Exhibit Hall
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Joël Kapusta, Air Liquide

1. Introduction to Fluid Dynamics
   a. Definitions
   b. Compressible Versus Incompressible Flows
   c. Continuity and Momentum Equations
   d. Sonic Flow

2. Single-Pipe Tuyere Injection
   a. Background - Bubbling Versus Jetting Regime
   b. Injection Under Bubbling Regime
   c. Injection Under Jetting Regime

3. Shrouded Sonic Injection
   a. Shrouded Injection Fundamentals
   b. Trials and Implementation in Cu and Ni Converters
   c. Shrouded Injector Design
   d. Shrouded Injector Modes of Operation

Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials VIII: Session V

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

Wednesday AM Room: 2022
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Jenn-Ming Song, National Dong Hwa University; Jae-Ho Lee, Hong Ik University

8:30 AM
Electropolishing of Overplated Copper for the Planarization of Copper through Via: Suk-Ei Lee[1]; *Jae-Ho Lee*[1]; [1]Hong Ik University

Planarization of copper thin films by electropolishing has great challenges due to the minimum amount copper to be removed. In this research the effects of current density, electrolytes and additives on the electropolishing of 50 and 20μm diameter copper via were investigated to eliminate thickness disparity during overplated copper removal in 3D SiP through via. The termination time was determined with analysis of applied potential on anode and cathode to avoid excess electropolishing. Acetic acid played a role of accelerator and glycerol played a role of inhibitor in phosphoric acid electrolytes. The overplated copper on the through via was effectively electropolished by applying proper current density in the phosphoric electrolytes with acetic acid and glycerol addition. The electropolishing was terminated at the point of abrupt change of applied potential to remove only overplated copper on the through via.

8:45 AM Invited
Fabrication of Copper and Tin Bump Using Electropolishing and Electroless Plating Methods in 3D SiP: Seong-Hun Kim[1]; Yun-Sung Moon[1]; Suk-Ei Lee[1]; Yeong-Kwon Ko[1]; *Jae-Ho Lee*[1]; [1]Hong Ik University

In conventional flip chip interconnection, CMP and lithographic processes were used in Cu/Sn bumps fabrication. During CMP process, copper via substrates were easily damaged and lithographic process is high-cost process. Series of electropolishing, electroless copper plating, electroless tin plating followed by reflow were suggested to replace CMP and lithographic process. The 50 and 20μm diameter copper via were fabricated by electroplating method. Overplated copper were successfully removed by electropolishing method in suppressor and accelerator added phosphoric acid. Copper bumps with 5μm height were formed on electropolished via by electroless plating method. Suppressor added electroless copper bath can promote selectivity of copper bump formation on via after proper acid cleaning. Tin bumps with 9μm height were formed on electroless copper bump. Finally uniform sized Cu/Sn bumps were fabricated after reflow process.

9:05 AM
15 nm Ru Diffusion Barrier on NiSi/Si for Sub-45-nm Cu Contact Plug: *Jia-Huei Lin*[1]; Chen-Sheng Hsu[1]; Jau-Shiung Fang[1]; [1]National Formosa University

This study aims at evaluating Ru barrier on NiSi/Si for Cu contact metallization. The films were deposited by magnetron sputtering using Ni, Ru and Cu targets. Low resistivity NiSi film was firstly obtained by carefully optimizing the conditions of rapid thermal annealed Ni/Si, then Ru and Cu films were sequentially deposited onto NiSi/Si substrate. The diffusion barrier properties of the studied films were elucidated using four-point probe (FPP), x-ray diffraction (XRD), scanning electron microscopy (SEM), Auger electron spectroscopy (AES) and transmission electron microscopy (TEM). The failure temperatures of 600°C (Cu/NiSi/Si) and 650°C (Cu/Ru/NiSi/Si) have been demonstrated. Structural analysis revealed that the failure mechanisms of the studied film involved the Cu penetrating through Ru/NiSi stack film, inducing the accelerated dissociation of the NiSi. Interposing a Ru layer remarkably improved thermal stability and barrier performance in the Cu/NiSi/Si stack films; which can be an effectively diffusion barrier for sub-45-nm Cu contact plug.

9:20 AM
Thermal Stability Properties of Various Vopper Alloy Films Used in Advanced Barrierless Cu Metallization: Jinn P. Chu[1]; *Chon-Hsin Lin*[2]; Wean-Kuan Leau[3]; Dung-Yuan Yu[1]; [1]Graduate Institute of Materials Science and Technology, National Taiwan University of Science and Technology; [2]Chin-Min Institute of Technology; [3]Institute of Materials Engineering, National Taiwan Ocean University

Owing to its low resistivity and high electromigration resistance, Cu is increasingly being used as an interconnect material in Si-based devices. Unfortunately, Cu diffuses rapidly in Si, thus deteriorating the device properties. To prevent device failure, a diffusion barrier must be placed between Cu and Si. However, with a decrease in the feature size, the barriers cause many problems. Therefore, barrier-free Cu metallization is proposed in this study. A Cu seed layer is prepared by alloying Cu with insoluble substances such as RuN to improve the thermal stability of the seed layer; the layer acts as a barrier and minimizes Cu/Si interdiffusion. The seed layer is characterized using X-ray diffraction, a focused ion beam, secondary ion mass spectroscopy, transmission electron microscopy, the film resistivity, and current-voltage measurements. The results indicate the enhancement of the thermal stability of the Cu film, with no apparent interaction between Cu and Si.

9:35 AM
Failure Behavior of Electroless CoWP Film as a Diffusion Barrier between Electroless Cu and Si: *Ting Tsai*[1]; Jiing Lee[1]; Sin Wu[2]; Jau Fang[1]; [1]National Formosa University; [2]National Taiwan University of Science and Technology

The electroless CoWP film with 89.4 at.% Co, 2.4 at.% W and 8.2 at.% P was deposited on silicon substrate as diffusion barrier for electroless Cu and Si. The Cu/CoWP/ Si stacked samples with 100 nm electroless CoWP films were prepared and annealed in a rapid thermal annealing (RTA) furnace. The failure behavior of electroless CoWP film in Cu/CoWP/ Si has been investigated by transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive X-ray spectrometer (EDS), X-ray diffraction (XRD) and sheet resistance measurement. The failure temperature of Cu/CoWP/Si is 650° and its failure is caused by the interdiffusion of Co and Cu. The cobalt penetrates through the copper film to form granular grains on the copper film and the copper penetrates through the electroless CoWP film to form the Cu3Si facet-like grains in the silicon. Finally, Co reacts with Cu3Si to form cobalt silicide on the top of Cu3Si.

9:50 AM Break

10:10 AM
Investigations of the Interfacial Properties in Ni-FUSI/Hf-Based/Si and Ni-FUSI/SiO2/Si Stacks by I-V and XPS Techniques: *S.Y. Tan*[1]; Ming-Yuan Wu[1]; Hsing-Hung Chen[1]; [1]Chinese Culture University

Metal gate and high-k gate dielectric are widely believed to be necessary for 45 nm CMOS node.Hf-based film is a promising candidate to replace SiO2 as gate dielectric,due to its much higher dielectric constant and stability.The combination of Ni-FUSI gate electrodes and high-k gate dielectrics is one of the most promising gate stacking structures. In this works, we established an effective way to identify the phase transformations by some nondestructive techniques.Furthermore, the aim of this work is to carry-out a comparative electrical and physical characterization of Ni-FUSI/Hf-based/Si and Ni-FUSI/SiO2/Si Stacks. The thermal stability, phase and interface uniformity of Ni-silicide are some key issues for Ni-FUSI technology. Lot of concerns on phase and interface uniformity of nickel silicide and electrical active defects caused by rapid Ni diffusion.We investigated the phase and interface properties of Ni-silicides formed by Ni–Si solid-state reaction and will be characterized by XRD, XPS and I–V techniques.

10:25 AM
Enhancement in Conductivity and Transmittance of Zinc Oxide Prepared by Chemical Bath Deposition: *Wei-Hsiang Luo*[1]; Ting-Kan Tsai[1]; Jau-Shiung Fang[1]; [1]National Formosa University

Low resistivity and high transparency ZnO thin films were prepared on cleaned Corning Eagle^{2000} glass substrate by chemical bath deposition (CBD) and the effect of deposition parameters on the structural, electrical and optical properties of the films were investigated. Using CBD to prepare ZnO film has the benefits of low cost and low temperature process, which make it potential to be used on a roll-to-roll process. The electrical properties were correlated with the structure of the film, and detail structural characterization was performed using

x-ray diffraction and field emission scanning electron microscopy. Experimental results indicated that the studied thin film had a low resistivity of 2.9×10^{-2} Ω cm and high transmittance above 80% in the visible range when the film was annealed under $Ar+H_2$ ambient. The results revealed that low resistivity and high transmittance ZnO film can be prepared using chemical bath deposition by carefully adjusting the deposition conditions.

10:40 AM

The Study on the Solubility of TiO-TiC System: Na Hou[1]; Chengjun Gao[1]; *Xiaohui Ning*[1]; Xiaotong Hu[1]; Hongmin Zhu[1]; [1]Beijing University of Science & Tech

The solubility of TiO-TiC system was studied by heating conversion and X-ray diffraction analysis(XRD). Pellets of the mixture of titanium monoxide(TiO) and titanium carbide(TiC) were heated under vacuum up to 1700°C. The results of XRD indicated that all the samples with various compositions had the single phase with NaCl-typed structure, which is the same of TiC and beta-TiO. The lattice parameter of the sample changes continuously with the change of the component. All the samples after the heat conversion showed high electronic conductivity.

10:55 AM

Dynamic Study on Formation Processes and Thermal Stability of Nickel Germanides by Using *In Situ* Transmission Electron Microscopy: *Jae-Wook Lee*[1]; Kwan-Woo Song[1]; Jee-Hwan Bae[1]; Min-Ho Park[1]; Han-Byul Kang[1]; Hyungsub Kim[1]; Cheol-Woong Yang[1]; [1]Sungkyunkwan University

Ge MOSFET has been considered as one of the promising devices for future high-speed CMOS technology due to higher carrier mobility than Si. However, the NiGe shows a poorer thermal stability than NiSi. It has been reported that the thermal stability of Ni-germanide improved through the addition of an alloying element, such as Ta or Zr. In this study, the formation and morphological evolution of metal (Ni, Ni-Ta, Ni-Zr) germanides as a function of temperature was investigated by *in situ* annealing of 15 nm-thick metal/Ge systems in the TEM with a specimen heating holder. Through the addition of alloying elements, Ni germanide grain growth was retarded and the surface morphology of the Ni germanide layer improved. Eventually, the agglomeration of Ni germanides was retarded and the thermal stability of the Ni germanides formed from the Ni-alloys became superior to that formed from the pure Ni.

WEDNESDAY AM

Recycling—General Session: Session I: 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 Organizer: Joseph Pomykala, Argonne National Laboratory

Wednesday AM Room: 2024
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Joseph Pomykala, Argonne National Laboratory

8:30 AM

Structure and Features of Slag from the Aluminum and Aluminum Alloys Melting and the Consequences: *Sergey Novichkov*[1]; Anatoliy Zholnin[1]; [1]Mosoblprommontazh

The influence of the flux structure upon the consistence of hot slag was investigated on basis of the earlier published model of aluminum slag structure. The aim of the investigation was to receive an experimentally proved conclusion which ensued from the described model. A practical consequence of the realized investigation was the detection of additives minimizing the flux consumption required to achieve the pre-determined consistence of hot slag. Two possible ways for decreasing of flux consumption ensue from the suggested model: a)use of additives which decrease the surface tension of melted flux; b)use of fluoride additives which reduce to fragments the oxide scales as principal thickener of melted flux. The investigations were realized with slag from salt-free remelting of aluminum scrap. As basic flux the equipoised mixture of sodium chloride and potassium chloride was used. The possibility of the substantial decreasing of flux consumption was obtained.

8:45 AM

Effective Recovery of Aluminium from Aluminum Dross by Selective Grinding and Air Classification: Euisup Shin[1]; Sujeong Lee[1]; SangBae Kim[1]; Wantae Kim[1]; Hosung Yoon[1]; *Sung-Baek Cho*[1]; [1]Korea Institute of Geoscience & Mineral Resources

Physical separation containing selective grinding and air classification were carried out to effectively recover aluminium from the aluminium dross. The aluminium dross sample used in this work contained about 12-18wt.% of metal aluminum and the metal content was varied in the fractions prepared by crushing and sieving. Aluminum metal is ductile and easily enlarged when a compressive force is engaged, whereas, oxide forms of aluminium can be easily broken and their resultant sizes are reduced after grinding. Since +5mm product is mainly aluminum metal, it was recovered by sieving and then -5mm product was ground by rod mill or steel ball mill for various periods and then air classified. The results showed that aluminium metals can be effectively recovered by air classification after milling using steel ball mill rather than rod mill. Thus recovered aluminum was leached out by various solvent to synthesize a alum, alumina and Al-alkoxide.

9:00 AM

The Energy and Environmental Implications of Recovering Salt Flux from Salt Slag Generated by the Aluminum Industry: *John Hryn*[1]; [1]Argonne National Laboratory

Recycling aluminum at secondary smelters usually involves the use of salt fluxes to improve aluminum recovery. Unfortunately, the use of salt fluxes results in the generation of a salt slag (or salt cake) waste stream. Attempts at developing a viable salt cake recycling technology to date have been unsuccessful, primarily due to the high energy cost of recovering a usable salt fraction from salt cake, and the lack of a suitable market for placing the residual non-metallic products, NMP. This presentation outlines the overall negative energy and environmental implications of attempting to recover salt flux from salt cake for reuse by the aluminum industry. From an energy and environmental perspective, the best practice today is to maximize aluminum recovery from salt cake and dispose the salt and NMP fractions in a controlled landfill.

9:20 AM

Recycling of Aluminum Metal Matrix Composite Reinforced with Zircon Sand: *Sanjeev Das*[1]; [1]Thapar University

In the present investigation, recycling of an aluminum alloy (Al-4.5 wt% Cu) from zircon sand reinforced aluminum metal matrix composite was done by simple melting and casting route. It is possible to separate molten matrix metal from zircon particles in the composite scrap by remelting, holding and casting. The scarp generated during casting of the composites was remelted at 800°C in a resistance furnace. It was observed that due to high difference in densities of the zircon sand and aluminum alloy, zircon readily settles down at the bottom of the crucible. About 50-70 wt%. of the matrix metals was separated from the composite reinforced with zircon particles of different amount and particle size. Minimum holding time of melt was evaluated for complete settling in which maximum amount of aluminum alloy was recovered.

9:35 AM

Selective Enrichment of Ti Components in Ti-Bearing Blast Furnace Slag and Coarsening of Perovskite Phase during Dynamic Oxidation: *Li Zhang*[1]; Yang Cao[1]; Tai Lou[1]; Zhi Sui[1]; [1]Northeastern University

The analysis of processing mineralogy on Ti-bearing blast furnace slag shows that most Ti components enrich in perovskite phase. Air was blown into the molten slag as oxygen resource during dynamic oxidation. It was found that the oxidation is not only in favour of Ti components into perovskite phase, but also the increase of slag temperature and decrease of slag viscosity during dynamic oxidation promote the precipitation and growth of perovskite phase, average crystal size of perovskite phase is 40 μm. The experimental result realizes the selective enrichment of Ti components and coarsening of perovskite phase, perovskite phase can be separated by mineral dressing method.

9:55 AM

The Recovery of Valuable Metals Containing in the Slag of Jamesonite Smelting in the Blast Furnace: *Xie Zhaofeng*[1]; Yang Tianzu[1]; Liu Wei[1]; Xia Wentang[2]; Liu Weifeng[1]; [1]Central South University; [2]Chongqing University of Science and Technology

A new recovery process of the valuable metals containing in the blast furnace slag of jamesonite was studied. In this process, the valuable metals such as Pb,

Sb, Zn and In etc. were fumed and volatilized and enriched in the dust firstly and then the dust was leached by sulfuric acid. In the leaching procedure, Zn and In in the dust entered into the leaching solution and were separated from Pb and Sb which remained in the residue.The volatilization rates of Pb, Sb, Zn and In are 95%, 92%, 86% and 75% respectively.The leaching rates of Zn and In are 98.5% and 95.2% respectively. The experimental results shows that the valuable metals in the dust can be recovered and separated effectively by this process and their recovery efficiencies are as follows: Pb94%,Sb91%, Zn84% and In69%.

10:15 AM Break

10:30 AM

Impact of Recycling on Materials Availability: A Case Study of Platinum: *Elisa Alonso*[1]; Frank Field[1]; Randolph Kirchain[1]; [1]MIT

Recycling is generally a more sustainable alternative to landfilling. However, recycling can be difficult to justify on the basis of simple cost recovery. As a consequence, significant amounts of even precious metals, such as platinum, can end up unrecovered. When one considers the risks of supply disruptions for firms that require scarce materials, recycling may serve to mitigate firm risk exposure, thus increasing its strategic value. This paper explores elements of this question, using a System Dynamics simulation model that strives to capture the dynamics of platinum market supply, demand and price. The detailed structures of supply and demand are key features of the model. Results show that since the secondary supply of platinum depends on a supply chain separate from primary extraction, recycling can diversify the risks of primary supply instabilities and reduce the impact of price fluctuations upon firms.

10:50 AM

Substance Flow Analysis of Lead in China: Xueyi Guo[1]; *Juya Zhong*[1]; Qinghua Tian[1]; Yu Song[1]; [1]Central South University

The method of Substance Flow Analysis (abbreviated as SFA) provides a helpful tool for the study of the industrial metabolism of a certain metal within a regional level. In this work, the flow of lead in China, 2006, was traced within the STAF model, and the situation of production, consumption and recycling of lead resources in China were introduced. The SFA is performed on lead flows during its one year life cycle in detail. As one of the biggest producer and consumer of lead in the world, China is facing a severe depletion of lead resources. How to increase the resource efficiency is a significant issue for lead industry of China. Based on the result, several advices were proposed in the paper, aiming to contribute important reference information for the industrial metabolism and resource management and recycling of lead industry in China.

11:05 AM

High Performance Recycling of Agricultural Wastes: Rice Husk Silica Used as Reinforcements of Magnesium Sintered Materials: *Katsuyoshi Kondoh*[1]; Junko Umeda[1]; [1]Osaka University

The utilization of silica particles originated in rice husks as reinforcements of magnesium composites is discussed. A reactivity of magnesium with silica particles in solid state to synthesize Mg2Si is investigated. Finer silica particles are more effective to be reacted with magnesium at low temperature due to the increase of their surface area contacted with magnesium. Amorphous silica is more useful for the reaction than crystalline one. The reactivity of rice husk silica is superior to the conventional mineral silica because of not only its amorphous structure but larger specific surface area by the porous structures. In the case of green compacts of the elemental mixture of silica particles and Mg powder, the silica particle size is not effective on the reactivity because the coarse particles are fractured into fine ones by cold compaction. The distribution of Mg2Si intermetallics of magnesium powder composites consolidated by SPS process is investigated.

11:25 AM

Treatment of Residues during Aluminum Recycling: *Bernd Prillhofer*[1]; Ramona Prillhofer[1]; Helmut Antrekowitsch[1]; [1]University of Leoben

Salt slag is a waste product, produced by the recycling of aluminum. In Europe the landfill disposal of this waste is forbidden, because the slag contains soluble salts and these represents a potential source of pollution to the surface and groundwater. Additionally, consisting metal and some compounds in the salt slag can react with water and resulting from this, noxious gases are generated. Recovery of useful materials from these wastes is according to this, both environmentally necessary and economically feasible. This study describes a complete recycling-process for the salt slag, which includes a leaching-crystallization-step to obtain the oxidic residue, a pyrometallurgical-step to treat the oxidic residue and final an extensive characterization of the achieved products. Due to the increasing price of raw materials, the utilization of the oxidic residue into commercial products was investigated and application possibilities are discussed.

11:45 AM

Recovery of Nickel and Cobalt by Pyrometallurgical Process of Waste from Producing Program of Nickel Metal Hydride Batteries: Hui Wang[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics & Astronautics

With the great increasing use of nickel metal hydride batteries recently, the waste of this kind batteries have been paid for close consideration. For the main reason that the waste of the batteries contained precious metals and lanthanide which cost great lose. This paper introduces the pyrometallurgical process of recovering nickel and cobalt from the waste of the nickel metal hydride batteries. Particularly, the waste for the process was from factory during the producing program. The use of designed solvent to divide nickel and cobalt in melted state from the waste is described. Also, the result of the nickel and cobalt and melted gangue that mainly consist of lanthanide oxide are included in details. The aim of the paper is to find out a simple and feasible way to reuse waste from nickel metal hydride batteries.

12:00 PM

Recovery of Ni, Co and Mn from Spent Battery Material: *Jinhui Li*[1]; Xinhai Li[1]; Yunhe Zhang[1]; Qiyang Hu[1]; Zhixing Wang[1]; Fangming Fu[1]; [1]Central South University

Recovery of Ni, Co and Mn from spent battery material not only protects the environment but also increases the utilization of resources and decreases the cost of battery material. With contrast of sulfate acid and hydrochloric acid, the results show that it has higher solubility and faster leaching rate in hydrochloric system than that in sulfate system, and hydrochloric acid can be recycled in the circuit. The experiments results show that the dissolution yields of Ni, Co and Mn can be 95% at least in hydrochloric system, the optimal conditions are that hydrochloric acid is 6mol/L and the particle size of waste is 120μm at least, temperature is about 60°, Liquid/Solid ratio=8:1, and the leaching time is 2 hour. The process seems to be able to claim economic recovery of base metals from waste, and which is reliable and feasible.

Shape Casting: Third International Symposium: Modeling

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Solidification Committee, TMS: Aluminum Processing Committee
Program Organizers: John Campbell, University of Birmingham; Paul Crepeau, General Motors Corp; Murat Tiryakioglu, Robert Morris University

Wednesday AM Room: 2011
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Christof Heisser, MAGMA Foundry Technologies, Inc.

8:30 AM Introductory Comments

8:35 AM

Prediction of Columnar to Equiaxed Transition in Alloy Castings with Convective Heat Transfer and Equiaxed Grain Transportation: *Wajira Mirihanage*[1]; Shaun McFadden[1]; David Browne[1]; [1]University College Dublin

A macroscopic, non-equilibrium model of the Columnar to Equiaxed Transition (CET) in alloy shape casting is presented. Convective heat transfer in the liquid metal and equiaxed grain transportation by fluid flow is included in the model. Nucleation from mould walls is used as the mechanism for columnar grain initiation. Nucleation from inoculants in undercooled liquid-ahead of the columnar front is considered for equiaxed grain formation. The front tracking model computes the advancement of the columnar front while the average growth of the equiaxed grain envelopes is simultaneously simulated. Latent heat release is incorporated in the model. The columnar mush and the coherent equiaxed dendrites are treated as porous media for convective flow. When equiaxed fraction is sufficient, no further advancement of the columnar front is permitted and the CET position is determined. CET is simulated for solidification of an aluminum-silicon alloy along with predictions of average equiaxed grain sizes.

9:00 AM
Prediction of Deformation and Hot Tear Formation Using a Viscoplastic Model with Damage: M. G. Pokorny[1]; Charles Monroe[1]; *Christoph Beckermann*[1]; [1]Department of Mechanical and Industrial Engineering, University of Iowa

A three-phase model is presented that predicts solid deformation as well as melt pressure, feeding flow and shrinkage porosity during casting. A viscoplastic constitutive theory with material damage is used to model the solid deformation. The damage created by mechanically induced voiding is proposed as a new hot tear criterion. The liquid feeding and shrinkage porosity model is coupled to the solid deformation model, which enables the final damage prediction to be more accurate than previous hot tear criteria. Novel steel casting experiments have been performed to measure the deformations and forces from solidification to shakeout. The measured and predicted deformations and forces show good agreement with the simulation results. Furthermore, the damage predictions show good correspondence with hot tear indications on radiographs of the test castings.

9:25 AM
A Diffusing Runner for Gravity Casting: *Fu-Yuan Hsu*[1]; Huey-Jiuan Lin[1]; [1]National United University

In gravity casting, the quality of an aluminium alloy casting relies on, among other things, the design of the runner system in which the gate velocity into the mould cavity should be controlled under a critical velocity close to 0.5 m/s. In this study a diffuser was proposed to reduce the velocity of liquid metal to below the critical value while the flow rate being almost unchanged. Flow separation and dead-zone in the diffuser design are avoided. A computational modeling package and real casting experiment (water analogy method) were employed for exploring and verifying the new design. The efficient of the diffuser is quantified by the measurement of coefficient of discharge, Cd.

9:50 AM
Process Modelling and Microstructure Prediction in Gravity Die Aluminium Castings with Sand Cores: Rosario Squatrito[1]; *Luca Tomesani*[1]; Ivan Todaro[1]; [1]University Bologna

The gravity die casting process for aluminium engine heads is one of the most complex of the casting industry, with high geometrical complexity, highest mechanical properties, lowest porosity levels. In the design of such a process by means of numerical methods, two kinds of problems arise: the first is the correctness of the boundary conditions of the thermal problem, the second is the availability of algorithms for predicting microstructural features, by which the final mechanical properties can be assessed. To assess this problems, a full numerical analysis has been carried out on an 8 cylinders A356 engine head which is in current production, by carefully replicating all the available process monitoring data: die temperatures and gradients, alloy composition and gas content, casting temperature, filling strategy and cooling times. An advanced microstructure module was then run to evaluate the porosity distribution throughout the casting; predicted and experimental values were finally compared.

10:15 AM Break

10:25 AM
Autonomous Optimization in Casting Process Simulation: *Christof Heisser*[1]; [1]MAGMA Foundry Technologies, Inc.

Computer processing speed has changed dramatically in the last 10 years. Traditional casting process modeling is based on "what-if" scenarios, requiring the user to make a decision, implement changes and start simulation. To gain the most advantage out of computer advances casting process modeling is now combined with genetic autonomous optimization tools, which produce a simulation based on a range of parameters rather than specific design points. Besides the optimization of designs, process parameters, as well as mechanical properties, can be optimized too. The production of high quality castings depends on many factors. As state-of-the-art simulation tools consider many of these factors, multi-objective autonomous optimization opens a whole new level of accuracy. Input parameters and thermophysical properties can be optimized to characterize the specific melt of a foundry or production line. This paper will show the back ground of genetic optimization tools and examples of optimized castings utilizing this technology.

10:50 AM
Modeling the Formation of Porosity during Low Pressure Die Casting (LPDC) of Aluminum Alloy A356: *Ehsan Khajeh*[1]; XinMei Shi[1]; Daan Maijer[1]; [1]The University of British Columbia

In this study, a coupled thermo-fluid flow model for predicting the position and the size of macro-shrinkage in low pressure die cast (LPDC) of A356 alloy has been developed using the Finite Volume Method (FVM). By solving the conservation equations, the interdendritic flow due to the change in density associated with phase transformation has been obtained. In order to satisfy continuity, the volume contraction must be accommodated by introduction of additional melt based on total volumetric strain rate. By studying the role of permeability, as the critical factor in determining pressure drop, feedability of mushy zone and the formation of macro-shrinkage in isolated zones have been determined. In order to validate the current method, the model has been applied to examine the evolution of shrinkage pores in a casting trial.

11:15 AM
Predicting Residual Stresses Caused by Heat Treating Cast Aluminum Alloy Components: *Chang-Kai Wu*[1]; Makhlouf Makhlouf[1]; [1]WPI/MPI

A mathematical model that enables the prediction of the effects of heat treatment on cast aluminum alloy components has been developed. The model uses the commercial software ABAQUS to predict residual stresses in heat treated components. An extensive database has been developed for A356 aluminum alloy and includes the mechanical, physical, and thermal properties of the alloy as functions of temperature. In addition, boundary conditions – in the form of heat transfer coefficients for each of the heat treatment steps – have been obtained from measurements performed with a specially designed quenching system. The database and boundary conditions were used to predict the response of a typical A356 cast component to a standard commercial heat treating cycle and the model predictions were found to be in good agreement with their measured counterparts.

11:40 AM
Internet-Based Casting CAE System: *Tao Jing*[1]; Baicheng Liu[1]; [1]Tsinghua University

Numerical simulation of solidification process has become a valuable tool for evaluating and optimizing casting pattern and rigging design to dramatically reduce the cost of reaching a satisfactory design. This paper presented the development of a Web-based Casting CAE System. The major competitive advantage of a Web-based casting CAE system, compared to a stand alone system, lies in its capability to share the limited resources through Internet within quite a lot of foundries, especially those medium and small scale foundries which may have difficulties to set up their own system alone. Java-based multi-tier architecture, CORBA-based distributed computing technology and Java Applet, Swing and Java Severlet technology are used for the implementation of the system.

Solar Cell Silicon: Production and Recycling: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizer: Anne Kvithyld, SINTEF

Wednesday AM — Room: 3004
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Anne Kvithyld, SINTEF

8:30 AM
Generation of Lustrous Carbon for Silicon Production: Valery Bogomolov[1]; Alexey Kozhan[1]; *Vladyslav Sokolov*[1]; Boris Bondarenko[1]; [1]Gas Institute NASU

The obvious way for increasing of efficiency of the solar grade Si production is lowering the amount of detrimental admixtures in its feedstock. The high purity raw materials should be applied in the charge to be smelted in reduction conditions. A unit for production of the correspondent semi-finished product has been designed, manufactured and put into operation. The applied novel technology is based on high temperature electro-thermal method of generation

of lustrous carbon on the surface of silica fines. . The silica reduction process should be facilitated in comparison with application of more traditional carbon black in terms of more intimate contact between the reacting matters in the charge. Very promising results were got earlier in production of silicon carbide by the technology. The current productivity of the unit is 35 kg silicon in semi-finished product per hour. The options for its increase are discussed.

8:55 AM

Settling of Particles in Molten Silicon before Directional Casting of a Solar Grade Silicon Ingot: *Arjan Ciftja*[1]; Eivind Ovrelid[2]; Merete Tangstad[1]; Thorvald Engh[1]; [1]Norwegian University of Science & Technology; [2]Sintef – Materials and Chemistry

Due to shortage of SoG-Si feedstock in the recent years, there has been a lot of effort to find alternative routes for the manufacturing of SoG-Si for solar cells. The development of cheaper processes for the production of SoG-Si has been subject of recent research worldwide. In this work, settling of inclusions in molten silicon is investigated. The material used is multi-crystalline silicon produced from a metallurgical route named Sunsilc. A 12kg directional-crystallisation furnace is used to cast metallurgical grade silicon with an addition settling step. The idea is to let SiC particles settle and then cast the ingot. Samples from the cast multi-crystalline silicon ingots are investigated by light microscope. The results show that the majority of particles remain at a depth of 2mm from the bottom of the cast ingot. The rest of inclusions are found along the whole height of the ingot but at much lower concentrations.

9:20 AM

Effects of Some Conditions on Si Growth from Si-Al Melts: *Yuuki Nishi*[1]; Kazuki Morita[1]; [1]University of Tokyo

In order to develop a new silicon refining process for solar cells, "solidification refining of silicon with Si-Al melt at low temperature" has been investigated in our research group. This process was considered to be more effective than conventional solidification refining from thermodynamic prediction. However, refined Si with needle-like shape was highly dispersed in a directional solidification refining and a bulk Si crystal could hardly be obtained. In this study, the solidification conditions to obtain bulk Si crystal were investigated. The shape variation of the interface between Si and Si-Al melt was observed with various temperature gradients and cooling rates. Bulk Si crystal was found to be obtained under the condition when the flat interface was observed. For better understanding of the Si growth condition, zone melting growth was also carried out and the condition to obtain facet Si growth was discussed.

9:45 AM

Refining of Silicon by Directional Solidification: *Eivind Øvrelid*[1]; Mari Juel[1]; Benjamin Tuffour[2]; Martin Bellman[2]; [1]Sintef; [2]NTNU

In all new processes for upgrading of metallurgical feedstock to solar grade, directional solidification is used as the last step to remove the last traces of impurities. In this work we have investigated the possibility to optimize the refining efficiency by modification of the fluid flow pattern by the Accelerating Crucible Rotation Technique (ACRT). We have made experiments in a furnace for directional solidification with and without ACRT, and characterised the material. Numerical modelling has been used to support the experiments. We have seen that the rotation gives a positive effect on the refining and we believe the results can be used to optimize industrial systems.

10:10 AM Break

10:20 AM

Simulation of Silicon Casting Process for Photovoltaic (PV) Application: Bei Wu[1]; *Sam Scott*[2]; Nathan Stoddard[1]; Roger Clark[1]; Adi Sholapurwalla[2]; [1]BP Solar; [2]ESI Group NA

Multicrystalline silicon ingot casting is widely used in Photovoltaic industry due to its feedstock tolerance, low cost, simple processes and equipment and high throughputs. To achieve high solar cell efficiency, crystal defects must be minimized and grain structure must be controlled. These crystal qualities strongly depend on temperature gradient and thermal history during casting processes. In order to better understand the casting process, numerical simulation is used to predict the temperature distributions and resulting crystal structure under different growth processes. Direct Solidification System (DSS), a common casting process in the solar industry, will be simulated using ProCAST, a commercial software primarily used for metal casting simulation. The software may be adapted to simulate the transient temperature field of the DSS station and the solidification process, including grain growth. The simulation results focus on the effects of insulation movement on crystallization initiation and grain structure with comparisons to cast silicon ingots.

10:45 AM

The Removal Rate of Phosphorus from Molten Silicon: *Takayuki Kemmotsu*[1]; Hisao Kimura[1]; Takashi Nagai[1]; Masafumi Maeda[1]; [1]The University of Tokyo

The production of solar cell is increasing rapidly and the shortage of silicon for solar cell becomes a serious problem. Developing a low-cost production process for solar grade silicon is required. An electron beam remelting technique under high vacuum is known to be effective for the removal of phosphorus from silicon. In this process, phosphorus can be removed preferentially from molten silicon because the vapor pressure of phosphorus is higher than that of silicon. However, the removal rate of phosphorus from silicon is not sufficient in this process. In this research, a more efficient removal process of phosphorus from silicon by an electron beam remelting technique was studied. Experiments showed that the removal rate of phosphorus from silicon increased with electron beam power. This indicated that raising temperature of molten silicon was effective for high-rate purification.

Structural Materials Division Symposium: Advanced Characterization and Modeling of Phase Transformations in Metals in Honor of David N. Seidman on his 70th Birthday: Kinetics of Phase Transformations II

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Chemistry and Physics of Materials Committee

Program Organizers: Robert Averback, University of Illinois, Urbana-Champaign; Mark Asta, University of California, Davis; David Dunand, Northwestern University; Ian Robertson, University of Illinois at Urbana-Champaign; Stephen Foiles, Sandia National Laboratories

Wednesday AM — Room: 3000
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Robert Averback, University of Illinois

8:30 AM Invited

Effect of Rare-Earth Elements Additions on Microstructure and Mechanical Properties of Al-Sc Alloys: *David Dunand*[1]; Matt Krug[1]; Alexandra Werber[2]; Marsha van Dalen[3]; Richard Karnesky[4]; David Seidman[1]; [1]Northwestern University; [2]Stuttgart University; [3]Momentive Performance Materials; [4]Sandia National Laboratories

Al-Sc alloys display a high number density of nanosize Al3Sc precipitates which are coherent with the Al matrix. Al-Sc-RE alloys (where RE is one of ten rare-earth elements: Y, Sm, Gd, Tb, Dy, Ho, Er, Tm, Yb or Lu) were studied by local-electrode atom-probe (LEAP) tomography. All RE segregate to the core of the Al3(Sc,RE) precipitates and some RE accelerate the kinetics of precipitation. The precipitate sizes and composition profiles are studied as a function of aging time at 300 °C and correlated with mechanical properties at ambient and elevated temperature. RE replacement of Sc, which reduces the cost of the alloys, does not increase their hardness, since the Orowan mechanism of precipitate bypass by dislocations is controlling strength at ambient temperature. RE replacement of Sc however improves creep resistance, which is explained by the increased matrix/precipitate lattice mismatch resulting in enhanced elastic interactions with matrix dislocations.

9:00 AM

Investigation of Alloying Effects on the Kinetics of Ni_2Cr Formation in Nickel-Chromium Alloys: George Young[1]; Reza Najafabadi[1]; James Vollmer[1]; Steven Attanasio[1]; *Julie Tucker*[1]; Mikael Christensen[2]; Walter Wolf[2]; Erich Wimmer[2]; [1]Lockheed Martin; [2]Materials Design

Isothermal aging experiments and first principles modeling were used to better understand the effects of alloying elements on the tendency of nickel-chromium alloys to undergo long range ordering (i.e., Ni_2Cr formation). Ordering is a concern in engineering alloys because it can increase residual stresses, lower the toughness, and degrade environmentally assisted cracking resistance. Long range order in both model and commercially produced alloys was assessed via

heat treatments at 475°C for times between 100 and 10,000 hours. The degree of ordering was quantified via lattice parameter contraction as determined by x-ray diffraction. Furthermore, the intrinsic effects of iron, niobium, and molybdenum on the stability of the Ni_2Cr structure were investigated via density functional theory. These results are used to assess the tendency for ordering in high chromium nickel-based alloys and to develop strategies to mitigate long range order.

9:15 AM

Atom-Probe Tomography in Materials Design: *Jason Sebastian*[1]; Gregory Olson[2]; [1]QuesTek Innovations, LLC; [2]Northwestern University

In honor of Prof. Seidman's birthday, we present a review of the role of atom probe tomography (APT) in materials design. Research efforts at Northwestern University and alloy development work at QuesTek Innovations have relied heavily on the ability of atom probe tomography to answer questions about the nanoscale structure and chemistry of engineering materials. Topics to be presented include the design of high strength intermetallic- and carbide-strengthened steels (including QuesTek's Ferrium®S53 corrosion resistant high strength landing gear steel), Ni superalloy design, aluminum alloy design, and high strength copper alloy design (QuesTek's Cuprium™ alloys). In all cases, APT allows for validation of important model predictions including phase compositions (as predicted by CALPHAD techniques), precipitate morphologies and size/spatial distributions, and segregation phenomena. Overall trajectories of nanoscale precipitate evolution under equilibrium and non-equilibrium conditions can be investigated experimentally via APT, and compared to model predictions.

9:35 AM

Phase Transformations in Pulsed Laser Deposited (PLD) FePd Thin Films: *Andreas Kulovits*[1]; John Leonard[1]; Jorg Wiezorek[1]; [1]University of Pittsburgh

We study the complex phase transformation behavior of off-stoichiometric Fe-rich and stoichiometric metastable FCC solid solution g-(Fe,Pd) and amorphous Fe-Pd thin films fabricated by pulsed laser deposition (PLD) on crystalline and amorphous substrates. We thoroughly characterized the microstructures of the as deposited films on the nanoscale using transmission electron microscopy (TEM) and X-ray diffraction (XRD). Upon equilibration the metastable FePd thin films undergo various different solid-state phase transformations, including crystallization, disorder–order transformation and phase separation reactions. We investigated solid–state transformation induced microstructural changes in grain size, morphology and local misorientation using HREM and TEM and changes in global texture using XRD after ex situ heat treatments. Furthermore we conducted in-situ heating TEM experiments to directly observe phase transformation sequences for the different metastable starting conditions.

9:50 AM Break

10:10 AM Invited

Dynamic Transmission Electron Microscope: Studying Nanoscale Material Processes with Nanosecond Time Resolution: *Thomas LaGrange*[1]; Geoffrey Campbell[1]; Bryan Reed[1]; Nigel Browning[1]; Judy Kim[1]; Mitra Taheri[2]; James Evans[1]; Wayne King[1]; [1]Lawrence Livermore National Laboratory; [2]Drexel University

There have been many efforts in the past decades to improve the spatial resolution of transmission electron microscopes but little in way of improving the temporal resolution of in situ transmission electron microscopy. Most materials dynamics occur at rates much faster than can be captured with standard video rate acquisition methods. Thus, there is a need to increase temporal resolution in order to capture and understand salient features of these rapid materials processes. To meet the need for studying fast dynamics in material processes, we have constructed a nanosecond dynamic transmission electron microscope (DTEM) at Lawrence Livermore National Laboratory to improve the temporal resolution of in-situ TEM observations. The DTEM consists of a modified JEOL 2000FX transmission electron microscope that provides access for two pulsed laser beams. One laser drives the photocathode (which replaces the standard thermionic cathode) to produce the brief electron pulse. The other strikes the sample, initiating the process to be studied. A series of pump-probe experiments with varying time delays enable, for example, the reconstruction of the typical sequence of events occurring during the rapid phase transformation. This presentation will discuss the core aspects of the DTEM instrument and how the DTEM has been used to study rapid solid-state phase transformations and chemical reactions. Work was performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under contract No. DE-AC52-07NA27344.

10:40 AM

Precipitation of Nano-Sized Nitrides in Nitrided Low-Alloy Steels: *Tadashi Furuhara*[1]; Yuusaku Tomio[1]; Keiichiro Oh-ishi[2]; Kazuhiro Hono[2]; Goro Miyamoto[1]; [1]Tohoku University; [2]National Institute for Materials Science

Nitriding becomes more important as a surface hardening heat treatment in wear resistant steels. High surface hardness is achieved by adding nitride-forming alloying elements because precipitation of fine alloy nitrides occurs. During nitriding, various kinds of metastable and stable nitrides precipitate depending upon alloying elements added. In this presentation, precipitation behavior of alloy nitrides during plasma nitriding of Fe-M(-C) alloys is described. Particularly, results of phase identification of nitrides and quantification of particle distribution made experimentally by means of advanced characterization methods, such as HRTEM, EFTEM and 3DAP, are shown.

10:55 AM

Segregation of W at γ' (L12) / γ (f.c.c.) Interfaces in a Ni-Based Superalloy: An Atom-Probe Tomographic and First-Principles Study: *Yaron Amouyal*[1]; Zugang Mao[1]; David Seidman[1]; [1]Northwestern University

Owing to their excellent mechanical behavior at high temperatures, Ni-based superalloys are utilized for turbine blades in aeronautical jet engines and land-based electricity generators, which are the most technologically-advanced energy conversion devices. Investigating the heterophase interfaces (HIs) of these alloys is essential for improving their high-temperature performances. γ' (L12) / γ (f.c.c.) HIs in a Ni-based superalloy are investigated using atom-probe tomography and first-principles calculations. {100} interfaces exhibit a Gibbsian interfacial excess of tungsten, $\Gamma_W = 1.2\pm0.2$ nm^{-2}, corresponding to a 5 mJ/m^2 decrease in interfacial energy. First-principles calculations for a W-alloyed Ni-Al system with a {100} HI have a similar *decrease* in interfacial energy when a W atom is placed as close as 1-3 atomic planes from it. Conversely, no measurable segregation of W is detected at general HIs. Indeed, similar calculations for {110} and {111} HIs predict an *increase* of 1 and 9 mJ/m^2 in their energies, respectively.

11:10 AM

Structure and Composition of γ/γ' Interfaces in the Ni-Base Superalloy Rene88DT: *Srinivasan Rajagopalan*[1]; Rajarshi Banerjee[2]; Gopal Viswanathan[1]; Junyeon Hwang[2]; Soumya Nag[2]; Jaimie Tiley[3]; Dennis Dimiduk[3]; Hamish Fraser[1]; [1]The Ohio State University; [2]University of North Texas; [3]Air Force Research Laboratory

The interfacial structure and composition in Ni-base superalloys can play a substantial role in determining several physical and mechanical properties. Of primary interest are the order-disorder transition, and the overall compositional transition from from the γ' to the γ phase. Using techniques such as aberration-corrected High Resolution Scanning Transmission Electron Microscopy (HRSTEM) and 3-Dimensional Atom Probe (3DAP) Tomography, this structural and compositional transition across the interface has been investigated at atomic resolution. Additionally, the effect of factors such as cooling rate (from a supersolvus annealing treatment) and aging time on the structural and compositional parameters affecting the interface are explored. Comparisons with atomistics-based modeling and implications for materials properties will be discussed.

11:25 AM

The Micromechanisms of Heterogeneous Phase Formation in Ti-Mo Alloys: *Robert Williams*[1]; Peter Collins[1]; Gopal Viswanathan[1]; Rajarshi Banerjee[2]; Srinivasan Rajagopalan[1]; Hamish Fraser[1]; [1]The Ohio State University; [2]University of North Texas

Ti-Mo alloys constitute an ideal model binary monotectoid system, providing the opportunity to gain insights into several microstructural processes that can be translated to the more complex β-Ti alloys. This study focuses on the observation and rationalization of key phase transformations in Ti-Mo alloys of varying compositions (8-18 wt% Mo). Of particular interest are the formation and growth of the ω phase, and the compositional dependence of the mechanics of α-phase nucleation. Employing techniques such as HRTEM, HRSTEM and EELS in an aberration corrected microscope, an attempt is made to capture the initial and later stages of phase formation and growth. Finally, an attempt is made

to rationalize the nucleation of α-phase in Ti-Mo alloys, and extend applicability of the rationale to other β-Ti alloy systems.

11:40 AM

The Temporal Evolution of the Nanostructures of a Low-Supersaturation Ni-Al-Cr Superalloy: *Christopher Booth-Morrison*[1]; Yang Zhou[1]; Zugang Mao[1]; Ronald Noebe[2]; David Seidman[1]; [1]Northwestern University; [2]NASA Glenn Research Center

The early stages of phase decomposition of a low-supersaturation Ni-6.5 Al-9.5 Cr at.% alloy, via the formation of coherent γ'($L1_2$)-precipitates from the γ-matrix, are studied by atom-probe tomography. The γ'-precipitate morphologies, radii, volume fractions, number densities, and interprecipitate spacing are measured, providing a complete description of the nanostructure during aging at 873 K. Additionally, the composition profiles across the heterophase γ-matrix/γ'-precipitate interfaces are quantified, providing information about elemental partitioning and the interfacial width. Experimental results are compared with the predictions of classical nucleation, growth and coarsening models, and are found to differ from these mean-field descriptions. The temporal evolution of the γ'-precipitate average radii and the γ-matrix supersaturations, however, follow the predictions of classical coarsening models. APT results are complemented by computational modeling employing Grand Canonical and Lattice Kinetic Monte Carlo simulation, and Thermo-Calc, Dictra and PrecipiCalc, to elucidate the kinetic pathways that lead to phase decomposition in this alloy.

11:55 AM

Dependence of Deformation Induced Martensitic Transformation on Crystallographic Orientation in a Metastable Austenitic Stainless Steel: *Jongbae Jeon*[1]; Young Won Chang[1]; [1]Pohang University of Science & Technology

Deformation induced martensitic transformation (DIMT) has been reported to depend on the amount of plastic strain, strain rate, chemical composition, and loading temperature. The strain distribution during plastic deformation is, however, inherently inhomogeneous due to different crystallographic orientation of each grain, so that each grain should exhibit different transformation behavior. It is therefore attempted in the present study to investigate the effects of crystallographic orientation on the DIMT behavior in a meta-stable austenitic stainless steel by means of an in-situ electron backscattered diffraction (EBSD) technique. The early stage of nucleation and growth of martensite phase induced by plastic deformation can be observed semi-directly from this in-situ EBSD method. Metastable austenite grains having the preferable crystallographic orientations for DIMT is more likely to nucleate first and grow into martensite phase.The nucleation sites are observed to be the intersections of thin shear bands e.g. localized dislocation bands, twins, or epsilon (HCP) bands.

Surface Structures at Multiple Length Scales: Bio Coatings and Nanoscale Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University; Sudipta Seal, University of Central Florida; Yang-Tse Cheng, University of Kentucky; Narendra Dahotre, University of Tennessee; Graham McCartney, University of Nottingham

Wednesday AM — Room: 3011
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

8:30 AM Invited

Nanoindentation of Biomaterials - The Evolution of Soft Material Testing: *Michelle Dickinson*[1]; [1]Hysitron Inc.

Nanoindentation is an established method for obtaining the nanomechanical properties such as hardness and modulus of traditional, elastic-plastic homogenous materials. Recently, the technique has moved into the biological field, which has created new challenges due to their inherent viscoelastic behavior and stringent environmental requirements. As biomaterials research progresses into smaller scale structures, new challenges such as time dependent analysis, heterogeneity and hydration become important considerations. Following a brief introduction on elastic-plastic indentation, the current paper reviews new techniques available for indentation analysis of viscoelastic and other non-standard materials. Recent works from nanoindentation of highly diverse biomaterials ranging from thin protein films, individual cells and insect exoskeletons to teeth, bones and replacement devices will be given with a discussion on the challenges and models used for each.

9:00 AM

Bioactivity and Biocompatibility of Laser Textured Ca-P Coatings for Hard Tissue Replacement: *Sameer Paital*[1]; Narendra Dahotre[1]; [1]University of Tennessee

As the interaction between the cells and tissues with biomaterials at the tissue implant interface is a surface phenomenon, surface engineered biomaterials aimed at modifying the surface properties while still maintaining the bulk properties of the implant are the design of current interest .In the present work a novel laser based direct writing technique is being explored to synthesize Ca-P coatings on Ti-6Al-4V substrate. Four samples with distinct patterned surfaces were obtained from two varying laser power conditions. Bioactivity of the coated samples was proved by the formation of an apatite like layer following immersion in SBF. Adhesion and proliferation of fibroblast and osteoblast like cells cultured on the coated samples further proved its biocompatibility. Elemental and phase analysis of the coated samples were studied by using both XRD and EDS. SEM and CLSM microscopy was used to study the morphology and focal contacts of the cell cultured samples.

9:20 AM

Interaction of Proteins with Primary Packaging Containers: *Rajendra Redkar*[1]; [1]SCHOTT North America, Inc.

Advances in the biotechnology industry have resulted in increased development of therapeutic protein drugs. Although Type I glass is the standard primary packaging container, the role played by the primary packaging material in destabilizing drug is not clearly understood. SCHOTT is interested in understanding the contribution of glass in inducing physical instabilities. A series of experiments involving treatment of glass vials to different buffers and other pharmaceutically relevant treatments were employed to evaluate how structural modifications can affect protein adsorption and aggregation over a period of time. In addition, the role of charge on the protein and surface charge on protein adsorption/aggregation will be presented. For syringes, the role of silicone oil in inducing protein aggregation and alternatives to the silicone oil, i.e. silicone-free lubricant system will be discussed. Finally, SCHOTT's approaches in mitigating other critical problems faced by the pharmaceutical industry through surface modifications will be presented.

9:40 AM Break

9:50 AM Invited

Laser Processing of Microstructured and Nanostructured Biomaterials: *Roger Narayan*[1]; [1]University of North Carolina - and - North Carolina State University

Lasers may serve to create novel medical devices with unique biological functionalities. For example, lasers offer a unique opportunity for microscale and nanoscale processing of biomaterials. We have recently developed microstructured and nanostructured biomaterials with unique biological functionalities using pulsed laser deposition, laser direct writing, and stereolithography processes. Laser processing of nanocomposite thin films, tissue substitutes, microstructured medical devices, and nanostructured medical devices will be discussed.

10:20 AM

Corrosion Control by Plant-Derived Agents in Silicone Coatings on Mild Steel Exposed to Seawater: *Sandy Tran*[1]; James Earthman[1]; [1]University of California, Irvine

Protective coatings are typically applied to improve corrosive and fouling resistance but often do not satisfactorily prevent localized corrosion. Immersion testing with nicotine and caffeine in simulated seawater solution was performed to evaluate the potential effectiveness of common plant-derived alkaloids for controlling corrosion. Relative to a control sample of mild steel, the most favorable results indicate that mild steel coated with silicone and exposed to seawater containing nicotine exhibit corrosion rates that are roughly 10-fold smaller than that for the same silicone coating in unaltered seawater. Coatings containing 4% and 10% wt. nicotine resulted in lower reductions in corrosion rate by comparison which was attributed to the effect of nicotine on the ability of the coating to adhere to the steel substrate. Similarly, seawater containing 1%

wt. caffeine in solution resulted in a reduction in corrosion rate by a factor of approximately 14 for samples coated with silicone.

10:40 AM Invited
Quantitative Deformation Tests inside TEM: *Z. W. Shan*[1]; A. Minor[2]; S. A. Syed Asif[1]; O. Warren[1]; [1]Hysitron Inc.; [2]Lawrence Berkeley National Laboratory and University of California, Berkeley

We report the current progress in the application of a unique TEM apparatus that enables to build a one-to-one relationship between the mechanical data and the evolving microstructure of the probed materials with a time resolution same as that of the recorded movies. For the indentation tests on submicron-sized Al grains, we found that even noise-level-force can nucleate dislocations from prefect Al grains and theoretical-level-stress can be achieved in grains with high density defects (Minor et al, Nat. Mat., 2006). Most recently, this device was used to perform compression tests on Ni pillars containing large amount initial defects. Surprisingly, the defects density was reduced dramatically upon the applied stress and in some cases, even resulted in a defect-free crystal. This phenomenon, termed as "mechanical annealing", can find its general application in interpreting the unusual mechanical behaviors that accompany the micro- and nano- structured crystals (Shan et al, Nat. Mat., 2008).

Synergies of Computational and Experimental Materials Science: Synergies in Integrated Computational Materials Engineering

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Katsuyo Thornton, University of Michigan; Henning Poulsen, Risoe National Laboratory; Mei Li, Ford Motor Co

Wednesday AM Room: 3003
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Mei Li, Ford Motor Co; Baicheng Liu, Tsinghua University

8:30 AM Invited
The Importance of Experiments in Integrated Computational Materials Engineering (ICME): *John Allison*[1]; [1]Ford Motor Company

Integrated Computational Materials Engineering (ICME) is the integration of materials information, captured in computational tools, with engineering product performance analysis and manufacturing-process simulation. While computational models provide the basic means of capturing and delivering quantitative processing-structure-property relationships to the engineering community, much of this knowledge relies critically on comprehensive experimental results. Despite important theoretical advances, processing-structure-property relationships are sufficiently complex and phenomenological that experiments are central to the development and calibration of these models and for filing information gaps in situations where theoretical models are not available. Experiments are also important for model validation. Virtual Aluminum Castings is an ICME method developed at Ford to improve the development process in the design of cast aluminum powertrain components. This talk will provide examples of the importance of a strong synergy between experiments and modeling in the development of Virtual Aluminum Castings.

9:00 AM Invited
An Integrated Multiscale Model for the Prediction of the Fatigue Life of Cast A356 Components as a Function of Solidification Microstructures: *Peter Lee*[1]; Pavel Ramirez lopez[1]; Trevor Lindley[1]; Junsheng Wang[1]; [1]Imperial College

A multiscale, through process model is developed to predict the impact of using high Fe-content secondary A356 alloy on the fatigue life of safety critical automotive components. The formation of Fe-rich intermetallics and microporosity is determined as a function of casting conditions and alloy composition at microstructural level in Al-Si-Mg alloys due to increased recycled scrap content. The final fatigue life is predicted by integrating the microstructural prediction into a macroscopic model of the casting, machining and final service of an automotive component. A second microstructural model was used to relate the maximum pore and intermetallic size, as well as stress state, to final fatigue life. In situ x-ray radiography experiments using synchrotron and laboratory sources were performed to quantify the nucleation and growth kinetics of the different microstructural features to inform and validate the models. The resulting lifing predictions showed good correlation to experimental measurements.

9:30 AM
Developing Local Mechanical Property Model for A319-Type Aluminum Alloys through Integrated Computational Materials Engineering (ICME) Approach: *Mei Li*[1]; Ruijie Zhang[1]; John Allison[1]; [1]Ford Motor Co.

In recent years, aluminum alloys have been increasingly used in automotive industry to replace cast iron for powertrain applications to reduce weight and emission. To provide high-quality blocks and heads at the lowest possible cost, products and manufacturing methods need to be developed and implemented in record time. The use of robust computational models in up-front analysis has proven to play an increasingly important role. This talk describes an ICME approach to develop local yield strength model for A319-type alloys which captures the influence of the manufacturing process history. The local yield strength model links a micro-model for solidification microstructures, a 3D phase field model for precipitation evolution morphology and kinetics and a micromechanical model for precipitation strengthening. The developed model was applied to a v6 engine block and the predicted local yield strength shows good agreement with the experimental measurement.

9:50 AM
Optimizing Aluminum Alloys for Multiple Objectives by Combining an Optimization Algorithm and FactSage Software: *George Dulikravich*[1]; Marcelo Colaco[2]; Carlos Velez[1]; [1]Florida International University; [2]Military Institute of Engineering (IME)

A novel concept of simultaneous optimization of several properties of aluminum alloys was developed and demonstrated. The method utilizes a commercially available software FactSage capable of predicting multiple alloy properties when provided with concentrations of each of the alloying elements. This alloy design method also utilizes a multi-objective evolutionary optimization algorithm that simultaneously extremizes several properties of the alloy. In order to accelerate the alloy design optimization process, an accurate and robust multi-dimensional response surface algorithm based on polynomial radial basis functions was utilized. The entire concept was successfully proven by the authors on H-type steels, Ni-based superalloys, Ti-based alloys and Hf-based bulk metalic glasses during the past six years. This paper presents an application of this design optimization methodology to Al-based alloys.Dulikravich and Egorov-Yegorov: Chemical Composition Design of Superalloys for Maximum Stress, Temperature and Time-to-Rupture Using Self-Adapting Response Surface Optimization. Materials and Manufacturing Processes. 20 (3) 2005, 569-590.

10:10 AM Break

10:30 AM Invited
Developing Micromechanism-Based Models to Assist in Alloy Design through an Integrated Experimental and Simulation Approach: Ning Zhou[1]; Chen Shen[2]; Ju Li[3]; Michael Mills[1]; *Yunzhi Wang*[1]; [1]Ohio State University; [2]GE Global Research; [3]University of Pennsylvania

To develop detailed understanding of dislocation-precipitate interactions in multi-phase alloys and to incorporate the most relevant deformation mechanisms in materials models, we have adopted an approach that integrates advanced experimental characterization with multi-scale computer simulations. Using Ni-base superalloys as examples we demonstrate (a) how computer simulations have helped in resolving a long-standing issue concerning the mechanisms of rafting in blade alloys and (b) how experimental characterizations have motivated and focused simulation studies of deformation mechanisms in disk alloys. In the former, defect-level simulations assisted in identifying the dominant mechanisms, upon which a micrometer-scale model was developed that makes quantitative predictions of rafting kinetics and the corresponding creep deformation at experimentally relevant length and time scales. In the latter, the activation energies associated with each individual deformation mechanisms suggested by experiment were calculated using a combination of ab initio informed microscopic phase field model and NEB method.

11:00 AM Invited
Solder Joint Lifetime Prediction via a Combined Experimental and Computational Approach: Michael Neilsen[1]; Paul Vianco[1]; *Elizabeth Holm*[1]; [1]Sandia National Laboratories

Because solders operate at high homologous temperatures, microstructural evolution, cracking, and failure can occur during service. Since circuit boards must function reliably for thousands of thermal cycles in applications such as aircraft and satellites, lifetime prediction is critical for product design and maintenance. We have developed a combined experimental and computational approach to predict both aging (crack initiation) and failure (open circuit) during thermomechanical cycling of lead-tin solder joints. A unified creep plasticity model, which includes contributions from microstructural parameters, predicts failure initiation during thermomechanical fatigue for a variety of solder joint geometries; in a blind study, model results are in excellent agreement with experimental observations of cycles to crack initiation. By adding element death to the model, crack propagation was captured over several thousand fatigue cycles; both crack location and morphology are in agreement with experiments. At each step, experiments inform and benefit from model predictions.

11:30 AM Invited
Macro and Micro Modeling of Conventional and Unidirectionally Solidified Investment Castings: Dong Pan[1]; Qingyan Xu[1]; Jing Yu[1]; *Baicheng Liu*[1]; Akihiko Kimatsuka[2]; Yasunoi Kuroki[2]; Yuriko Saito[2]; [1]Tsinghua University; [2]Ishikawajima-Harima Heavy Industry Co, Ltd

An integrated model for the three dimensional simulation of the solidification process both for conventional and unidirectionally solidified investment castings was developed. It was applied to industries to investigate the temperature distribution and shrinkage porosity, and to predict the grain growth and grain structure of the shaped castings. A ray tracing method was used to deal with the complex heat radiation transfer, and the micro structure evolution was simulated based on MCA method. In addition, a layer-by-layer calculation method was developed to couple the macro and micro model together and to save large computation memory as well. Experiments for titanium alloy turbine wheel casting produced by conventional investment process and nickel-based superalloy turbine blade casting by unidirectional solidification process were carried out, and the cooling curves, shrinkage porosity and microstructure during solidification were compared with the simulated results. Finally, some further recommendations were proposed.

12:00 PM
Genetic Computational Design of Novel High Strength Stainless Steels: Model Description and and First Experimental Validation: *Wei Xu*[1]; P.E.J. Rivera Diaz del Castillo[2]; Sybrand van der Zwaag[2]; [1]Materials Innovation Institute and Delft University of Technology, the Netherland; [2]Faculty of Aerospace Engineering, Delft University of Technology

A computational alloy design approach for precipitation hardened stainless steels coupling thermodynamic and kinetic calculations with a genetic optimization algorithm is presented. The alloys are designed so as to display desired microstructures, which include controlled densities of fine precipitates resulting from optimized composition and heat treatment aimed at maximizing the precipitation strengthening contribution. Alloy systems leading to MX carbide and/or Cu, NiAl and Ni3Ti intermetallic precipitates while keeping undesirable phases and Cr depletion at a minimum level were favored. Four novel alloys were fabricated and subjected to several heat treatment scenarios. The microstructures and resulting mechanical properties were evaluated and compared to model predictions as well as those of existing alloys.

Transformations under Extreme Conditions: A New Frontier in Materials: Solid-Solid Transformations and In Situ Diagnostics I

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Wednesday AM — Room: 3001
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Vijay Vasudevan, University of Cincinnati; James Belak, Lawrence Livermore National Laboratory

8:30 AM Invited
The Shock Properties of Mild and Bainitic Steels - Phase Transformation and Strength: *W.G. Proud*[1]; R.I. Hammond[2]; S.M. Walley[1]; D.J. Chapman[1]; [1]Fracture and Shock Physics Group, Cavendish Laboratory; [2]Fracture and Shock Physics Group, Cavendish Laboratory - and - currently at TWI Ltd.

Bainitic steels are ferrous alloys produced through the careful control of the cooling regime and are of potential interest in ballistic applications. An upper and a lower bainitic steel were studied. Whilst, superficially, the Hugoniots were found to be similar, the materials display important differences. The upper bainitic steel was recovered whole or in a few fragments, whilst the lower bainitic steel exhibited substantial brittle fracture. A further and interesting difference was that while the ferrite in the lower temperature Bainite underwent a pressure induced phase transition, the upper Bainite showed no evidence of such a transition. To study the transformation in detail, the lateral shock strength of the materials was measured. The gauges used required extensive protection in the region of the phase transition, a factor which affects the fidelity of the measurements.The properties of these alloys are compared with that of a well-characterised mild steel.

9:05 AM Invited
Atomistic Calculations of Shock Induced Phase Transformations and the Mechanical Properties of the Shocked Material: *Michael Baskes*[1]; [1]Los Alamos National Laboratory and University of California, San Diego

Shock waves in materials produce a number of interesting phenomena including phase transformations. This presentation will discuss calculations of this phenomenon at the atomic level using an embedded atom method potential for fcc Ni. Using standard flyer-plate geometry, molecular dynamics (MD) calculations at room temperature were performed. In summary: (1) the fcc crystal transforms to a bcc-like crystal structure just behind the shock front; (2) after a period of time, the bcc structure transforms to a layered hcp/fcc material; and (3) when the reflected shock wave traverses this material, the system mostly transforms back to fcc leaving twins and stacking faults (SFs) as the prominent defects. A small sample of he highly defective material was then tested using MD under a variety of loading conditions at high strain rate. The strength of the shocked material depends sensitively upon loading condition. The strengthening and weakening mechanisms will be discussed.

9:40 AM Invited
Kinetics of the Shock-Induced α-ε Phase Transition in Solid Iron: *Timothy Germann*[1]; Kai Kadau[1]; Brad Holian[1]; [1]Los Alamos National Laboratory

Previously we have demonstrated that molecular dynamics (MD) simulations can provide unique insight into the atomic-scale mechanisms of shock-induced solid-solid phase transformations. In situ dynamic x-ray diffraction studies have shown excellent agreement with these simulations, for thin foils of single crystal iron subjected to laser shock. However, the rapid kinetics observed in these studies is orders of magnitude faster than that observed in traditional macroscopic shock experiments (e.g. gas gun), including recent experiments with single crystal targets as thin as 100 μm. Moreover, these latter experiments indicate a three-wave structure, with bcc plasticity evident ahead of the phase transformation wave, unlike the smaller-scale MD simulations and laser-shock experiments. After reviewing this earlier work, we will present the results of a series of very-large-scale MD simulations of single crystal and polycrystal iron

samples (as long as 10 μm), subject to both shock and ramp wave loading, to explore these discrepancies.

10:15 AM Break

10:30 AM Invited
Studying Dynamic Material Response under Dynamic Compression: The Intersection of Experiment and Simulation: *Hector Lorenzana*[1]; Eduardo Bringa[1]; Bassem El-Dasher[1]; James Hawreliak[1]; Daniel Kalantar[1]; Giles Kimminau[2]; James McNaney[1]; Despina Milathianaki[1]; Warren MoberlyChan[1]; Babak Sadigh[1]; Michael Surh[1]; Damian Swift[1]; Anthony Van Buuren[1]; Justin Wark[2]; [1]Lawrence Livermore National Laboratory; [2]Oxford University

Understanding the dynamic lattice response of solids under extreme conditions of pressure, temperature and strain rate is a topic of broad scientific and technological interest. Critical to developing this understanding is the ability to probe and simulate the spatial and temporal evolution of phase transformations, material microstructure, and related properties at the characteristic timescale of the physical phenomena driving these processes—down to picoseconds. We will present studies exploiting new generation of bright and fast x-ray sources to investigate the real-time physical evolution of dynamically compressed materials. Specifically, we will discuss laser-based in situ x-ray diffraction measurements of phase transformations and lattice relaxation in single crystal and polycrystalline systems and results of post-processed large-scale molecular dynamics simulations that calculate physical observables and couple to experiment at similar temporal scale. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

11:05 AM
Phase Transformation under Pressure: Studies Combined Synchrotron High Energy X-Ray Diffraction and 3D Microtomography Techniques: *Luhong Wang*[1]; Haozhe Liu[1]; Xianghui Xiao[2]; Peter Lee[2]; [1]Harbin Institute of Technology; [2]Argonne National Laboratory

Application of new high-pressure synchrotron diamond anvil cell microtomography and high energy x-ray diffraction techniques has uncovered unexpected dynamics and volume expansion associated with pressure-induced crystallization of amorphous Se. The unusual volume expansion phenomenon under pressure associated with the recrystallization of 'over-pressurization' of a metastable phase may be more common than previously believed. Moreover, tuning relative densities and energetics of phases in this way may provide a new route for creating new structures from highly metastable states. Finally, the new microtomographic technique developed here could find widespread use in accurate determinations of the equation of state of glasses and melts up to megabar pressures, which is crucial to many problems in earth and materials sciences. The results demonstrate the importance of using new time- and spatially- resolved high-pressure x-ray diffraction and imaging techniques to understand the kinetics of structural transformations in materials under extreme conditions far from equilibrium.

11:25 AM
Measuring Plastic Response of Materials during Shock Compression Using In-Situ X-Ray Diffraction: *James Hawreliak*[1]; Bassem El-Dasher[1]; Andrew Higginbotham[2]; Giles Kimminau[2]; James McNaney[1]; Despina Milathianaki[3]; William Murphy[2]; Damian Swift[1]; Justin Wark[2]; Hector Lorenzana[1]; [1]LLNL; [2]University of Oxford; [3]University of Texas

The relaxation of a material from a uniaxially compressed state to a more hydrostatic state during shock compression through the generation and propagation of defects is a scientifically rich but relatively unexplored area of material science. Using in-situ x-ray diffraction to probe during the shock compression gives the ability to look at the lattice relaxation during shock compression. We discuss in-situ probing techniques and recent results looking at the plastic response of single and poly crystalline iron shock at pressure around the alpha to epsilon phase transition. This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

11:45 AM Invited
Prospects for Validation of Crystal Level Phase Transformation Models: *Nathan Barton*[1]; Joel Bernier[1]; [1]Lawrence Livermore National Laboratory

We explore prospects for validation of a crystal level model of phase transformation and twinning. The model treats the formation of distinctly orientated crystalline domains during polymorphic phase transformation and twinning, and allows for plastic deformation by crystallographic slip in each variant. In cases involving concurrent transformation and crystallographic slip, complexity of both the experimental material response and the model details have limited validation efforts. Work on carefully chosen materials and loading conditions has enabled validation of certain aspects of the model, and this work will be reviewed. New work focuses on the prospects for more extensive validation using recently developed in situ diffraction based techniques. Preliminary results will be presented for twinning in magnesium, and we will discuss the outlook for validation using in situ measurements under elevated pressure conditions. This work performed under the auspices of the US DOE by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 (LLNL-ABS-405264).

12:20 PM
Transforming Graphite to Diamond: An Ab Initio Molecular Dynamics Study of Graphite under Shock Compression: *Nir Goldman*[1]; I.-F. W. Kuo[1]; Evan J. Reed[1]; Laurence E. Fried[1]; C. J. Mundy[2]; Alessandro Curioni[3]; [1]Lawrence Livermore National Laboratory; [2]Pacific Northwest National Laboratory; [3]IBM Research, Zurich Research Laboratory

We present an extremely large scale ab initio calculation of the transformation of graphite to diamond under shock compression utilizing Car-Parrinello Molecular Dynamics (CPMD) in conjunction with the Multi-Scale Shock Method (MSSM). Our results indicate that the transition from graphite to diamond is Martensitic, in agreement with experimental observations. We find that a shock of 12 km/s forms a short-lived layered diamond phase which eventually relaxes to a cubic diamond state. Moreover, access to the electronic structure allows the computation the x-ray absorption spectra (XAS) to characterize the final states. The XAS spectra and wide angle x-ray scattering spectra (WAXS) confirm the presence of a cubic diamond final state.

2009 Functional and Structural Nanomaterials: Fabrication, Properties, and Applications: Nanoscale Phenomena: Mechanics, Phase Stability and Properties

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS: Nanomaterials Committee, TMS: Nanomechanical Materials Behavior Committee

Program Organizers: Gregory Thompson, University of Alabama; Amit Misra, Los Alamos National Laboratory; David Stollberg, Georgia Tech Research Institute; Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington; Wonbong Choi, Florida International University; Alexander Howard, Air Force Research Laboratory

Wednesday PM — Room: 3018
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Jiyoung Kim, University of Texas at Dallas; Seong Jin Koh, University of Texas at Arlington

2:00 PM Invited
3-D Compositional Imaging at the Nano-Scale: *David Larson*[1]; [1]Imago Scienctific Instruments

Atom-probe tomography can provide 3-D atomic-scale structural and compositional analysis of materials which is difficult to duplicate using other high-performance techniques such as transmission electron microscopy and secondary ion mass spectroscopy. Recent specimen fabrication techniques using focused-ion beam instruments with in-situ manipulation now allows a variety of nanoscale materials with site-specific features to be prepared and analyzed in the atom probe. This talk will address the fabrication and atom probe analysis, as well as comparison to other characterization techniques, of dopant profiling and defect ring structures in semiconductor transistors materials. Additionally, microstructural analysis of nanoscale laminates in magnetic and quantum-well layered features will be described.

2:30 PM

Advanced Production of Nanobased Photoctalytic Surfaces: *Christian Wögerer*[1]; Robert Katzensteiner[1]; Thomas Placzek[1]; Georg Waldner[2]; Norica Godja[3]; [1]Profactor Research and Solutions GmbH; [2]Austrian Research Center; [3]CEST GmbH

The paper describes a continuous wet-chemical production of photocatalytic nanoparticles technology with different direct connected coating processes including necessary pre-and aftertreatment for Photocatalytic-surfaces on different substrates. A newly developed nozzle-technique based on a simple precipitation process will be used to establish a continuous particle production capable for Up-Scaling without having to deal with the typical problems of "conventional" precipitation. The most important facts in this matter are: continuous operation, controllable particle characteristics, easy Up-Scaling, pcnp´s in colloidal solution, cheap precursors combined with simple plant design resulting in a cost effective production method. For an advanced coating procedure new developed coating techniques will be implemented by establishing a "tandem plant" which will be able to produce pcnp´s and deposit them on substrates within one single device or depositing the pcnp´s in a subsequent device apart from the production ("ambulant coating")Both methods will lead to prototype plants/devices capable to be commercialised.

2:45 PM

Characterization and Hydrogen Cycling Behavior of Nanoporous Palladium Alloy Thin Films: *Wen Chung Li*[1]; T. John Balk[1]; [1]University of Kentucky

Nanoporous palladium (np-Pd) films were prepared by dealloying of co-sputtered Pd alloy precursors on Si substrates. Film stress, microstructure and composition were tracked during dealloying using wafer curvature, electron microscopy, x-ray diffraction and energy-dispersive spectroscopy. Np-Pd films exhibit a uniform porous structure with pores and ligaments as small as 5 nm, yielding a high amount of surface area for possible hydrogen sensing applications. To test this capability, hydrogen cycling experiments were performed in the wafer curvature system with a hydrogen-containing atmosphere (partial pressure of hydrogen gas varied from 0 to 760 torr) over a range of temperatures (-50 to 200 degrees Celsius). Various np-Pd alloy films were compared and found to exhibit quicker hydrogen sensing than dense Pd films of comparable thickness. Finally, the elastic and plastic deformation components of np-Pd alloy film behavior were analyzed in relation to film microstructure and stability.

3:00 PM

Metastable Phases in Sputtered Fe-C Thin Films: *Arnaud Weck*[1]; Chad Sinclair[1]; Colin Scott[2]; Christian Maunders[3]; [1]The University of British Columbia; [2]Arcelormittal; [3]McMaster University

Vapor deposition as a continuous technique for modifying the surface of sheet metals provides rich opportunities for the development of novel functional and mechanical properties in metallurgical systems. In this work, we focus on the wealth of metastable structures that can be obtained in the iron-carbon system when thin films are deposited by sputtering with and without an applied substrate bias (ion plating). Various characterization techniques (e.g. EELS, HRTEM, APFIM) have been employed to characterize the as-deposited structures which range from amorphous to highly faulted nanocrystalline carbides (e.g. Fe_7C_3). The results suggest the possibility of colossal carbon supersaturation in bcc Fe under particular conditions of deposition, not inconsistent with previous observations of carbon supersaturation resulting from other far from equilibrium processes.

3:15 PM

Phase Diagram of the Ag-Pd Bimetallic Nano Cluster: Hyun You Kim[1]; Da Hye Kim[1]; *Hyuck Mo Lee*[1]; [1]KAIST

We report on the complete phase diagram of 135Ag-16Pd bimetallic nano cluster that is widely used as a selective hydrogenation catalyst. The solid to liquid transition region was investigated using molecular dynamics simulations with an improved collision method, and the solid state region of the phase diagram was explored with the combination of molecular dynamics, modified basin hopping Monte Carlo, and density functional theory. The solid state structure of the 135Ag-16Pd cluster is discussed in a viewpoint of over-stability of the meta-stable structure. Because the properties of the clusters are a kind of manifestation of their structures, our phase diagram will serve as a brief guidance for practical application of the Ag-Pd catalysts.

3:30 PM Break

3:45 PM

A Mimetic NanoPorous Carbon Model by Quench Molecular Dynamics Simulation: *Yunfeng Shi*[1]; [1]Rensselaer Polytechnic Institute

Despite extensive applications in many fields and even greater prospects in the future, the structure of nanoporous carbon is poorly understood at the atomic level. Due to the difficulty in its structural determination experimentally, many virtual carbon models have been put forth with various physical and chemical details. In this talk, a mimetic porous carbon model is generated using quench molecular dynamics simulations that reproduces experimental radial distribution functions of activated carbon. The quench conditions are systematically varied and the final porous structure is scrutinized in terms of its pore size distribution, pore connectivity and fractal dimension. It is found that the initial carbon density affects the fractal dimension but only causes a minor shift in the pore size distribution. On the other hand, the quench rate affects the pore size distribution but only causes a minor shift in the fractal dimension.

4:00 PM

Scale-Dependent Performance of Piezo-Composites: *Nelson Pinilla*[1]; Kishore Pochiraju[1]; [1]Stevens Institute of Technology

Composites reinforced with piezoelectric fibers are use in smart structures and high performance sensors. Piezo-composites act as both sensing and actuation elements in smart material applications with embedded closed loop control. However, response time delay between strain development and electrical signal detection may be significant; the response time delay lowers the control loop frequency and limits the performance of the composite when subjected to high frequency impact or shock loading. This paper investigates the fiber size dependence on the response time delay of piezo-composites. Nanoscale fibers have the potential to reduce the time delay and increase the control loop frequencies. This paper presents a characterization of model piezo-composites reinforced with fibers with varying diameters. Processing and electrical interconnections of the piezo composites are quite challenging; a unique experimental setup has used to determine the response delays of the composites. Model-based, performance, and experimental results would be presented in this paper.

4:15 PM

Plastic Flow Stability of Nanotwinned Cu Films: *Osman Anderoglu*[1]; Amit Misra[2]; Xinghang Zhang[1]; [1]Texas A&M University; [2]Los Alamos National Laboratory

We have room temperature rolled sputtered deposited, (111) textured, nanotwinned Cu foils up to 50% thickness reduction. X-ray pole figure measurements indicate no significant out-of-plane rotation of the grains. No significant change in the average twin lamellar thickness is seen although the height of the columnar grains was reduced by a factor of two after rolling. Transmission electron microscopy was used to elucidate the stored dislocation content at twin boundaries. A dislocation mechanism is developed to explain the plastic flow stability of nanotwins to large strains.

4:30 PM

Influence of Crystallization on Sheet Resistivity Associated with the Size Effect of AgInSbTe Chalcogenide Films: *Chung-Wei Yang*[1]; Truan-Sheng Lui[1]; Chien-Chih Chou[1]; [1]National Cheng Kung University

The as-deposited AgInSbTe (AIST) chalcogenide films with different thickness were prepared with using a series of power, pressure and sputtering durations by RF-sputtering process. The quasi-crystallized structure of as-deposited films can be recognized according to evidences of grazing-incidence x-ray diffraction analysis and transmission electron microscopy. Through the estimation of crystal size, a higher sputtering power and a longer sputtering duration can result in a larger extent of atomic rearrangements. The sheet resistivity measurement of amorphous films reveals that the relationship between the sheet resistivity and thickness of amorphous films is against the common known size effect. The low carrier mobility in the amorphous films is due to the effect of its disorder microstructure from the measurement of Hall coefficients to the as-deposited and crystallized films.

4:45 PM

Electrical Transport Behavior of Nanoscale Ga-Doped ZnO Film in Low Temperature Regime: *Young Gun Ko*[1]; Byung Du Ahn[2]; Hyun Jae Kim[2]; Dong Hyuk Shin[3]; [1]Massachusetts Institute of Technology; [2]Yonsei University; [3]Hanyang University

This paper deals with the electrical response of nanoscale Ga:ZnO thin films fabricated through pulsed laser deposition method and the transporting mechanisms associated with electrical conduction and scattering behavior with respect to temperature tested within the range of 300 to 10 K. All films that contain the degenerate band due to heavy doping process itself exhibit the metallic characteristics except those observed below 100 and 60 K, respectively when deposited at 298 and 773 K, respectively. This metal-semiconductor transition here is explained based on the role of weakly localized electrons found in a disorder conductor. As for scattering behavior of the present films, governing mechanism is thought to be ionized-impurity scattering below metal-semiconductor transition temperature. Depending on grain size, however, matrix and grain boundary will act as a barrier to inhibit the motion of electrons in a regime above metal-semiconductor transition temperature.

5:00 PM

Interfacial Effects in the Relaxation Dynamics of Silver Nanometal-Glass Composites Probed by Transient Grating Spectroscopy: *José Jiménez*[1]; Sergiy Lysenko[1]; Valentin Vikhnin[1]; Huimin Liu[1]; [1]University of Puerto Rico at Mayagüez

The relaxation dynamics of silver nanoparticles (NPs) in phosphate glass have been studied by picosecond time-resolved transient grating spectroscopy. Glasses were prepared by melting and heat treatment (HT) processes where two different metal-glass interfaces were presumably produced. The first is a glass system containing silver and tin in which Ag NPs are embedded in the matrix upon HT. The second is a heat-treated silver-doped glass with spectroscopic indications of Ag^{+}-Ag° pairs located at/or near the metal-glass interface. The time evolution of the light-induced transient diffraction grating for the Ag/Sn-doped glass shows an uncommon relaxation in the nanosecond time scale. Such behaviour is explained in terms of energy transfer processes between polaronic and/or excitonic states in the near-interface region of the glass matrix and NPs. In contrast, a faster monotonic relaxation is observed for the Ag-doped nanocomposite. This result is attributed to Ag NP —> Ag^{+}-Ag° plasmon resonance energy transfer.

WEDNESDAY PM

Alumina and Bauxite: Alumina Precipitation

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Everett Phillips, Nalco Co; Sringeri Chandrashekar, Dubai Aluminum Co

Wednesday PM — Room: 2002
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Jorge Aldi Lima, Alunorte

2:00 PM Introductory Comments

2:05 PM

Kinetics of Super-Fine Aluminum Hydroxide Precipitation from Sodium Aluminate Solutions with Gel-Seed: *Jianli Wang*[1]; Qiyuan Chen[2]; Wangxing Li[1]; Zhoulan Yin[2]; [1]Zhengzhou Research Institute of Chalco; [2]Central South University

Effect of precipitation temperature, sodium hydroxide concentration and seed ratio on super-fine aluminum hydroxide precipitation from sodium aluminate solutions with gel-seed was studied and the kinetics model was proposed. According to this model, super-fine aluminum hydroxide precipitation follows second order reaction kinetics, however, its rate constant is larger than the rate constant of metallurgical grade aluminum hydroxide precipitation. Both the precipitation temperature and the gel seed ratio affected the precipitation rate and the particle size. Compared with metallurgical gibbsite precipitation, the reaction rate was faster, the precipitation rate could reach about 40% when precipitation time was 8 hours. The gel seeds were dissolved partly first and then new aluminum hydroxide was precipitated and grew on the surface of the seeds. Dynamic light scattering was used to characterize super-fine gibbsite particle growth and it was found that the dominant mechanism of the crystal of gibbsite growth was single particle growth.

2:30 PM

Effect of α-Alanine on the Seeded Precipitation of Sodium Aluminate Solution: Baolin Lv[1]; *Qiyuan Chen*[1]; Zhoulan Yin[1]; Huiping Hu[1]; Xing Chen[1]; [1]Central South University

The effect of α-alanine on the seeded precipitation of sodium aluminate solution was investigated. The relative concentrations of $Al_2O(OH)_6^{2-}$ with the absorption band at about 530cm^4 or polynuclear complex with the absorption bands at about 885cm^4 and 635cm^4 and the stability of sodium aluminate solution were determined using semi-quantitative method of FT-IR spectrum and 27Al NMR spectra, respectively. All the results show that compared to the blank, α-alanine at proper dosages reduces the precipitation ratios of sodium aluminate solution during the initial reaction time, then accelerates precipitation process, which may result from the break of dynamic balance among aluminate specieses present in sodium aluminate solution or from the variation of the stability of sodium aluminate solution due to the transformation of some new aluminate specieses which could be beneficial for the formation of growth unit in sodium aluminate solution.

2:55 PM

Effects of Four Aromatic Carboxylic Acids as Inhibitors on the Seeded Precipitation Ratios of Sodium Aluminate Solutions and the Agglomeration Efficiency of Gibbsite: Baolin Lv[1]; *Qiyuan Chen*[1]; Zhoulan Yin[1]; Huiping Hu[1]; [1]Central South University

The effects of four aromatic carboxylic acids as inhibitors on precipitation ratios of sodium aluminate solutions, particle size distribution (PSD) of gibbsite were investigated using titration method, particle size analyzer, respectively. The net charges of oxygen atoms in the four aromatic carboxylic anions and the dipole moments of the four aromatic carboxylic anions were calculated by GGA-PW91 in Module-Dmol3 of Materials Studio and B3LYP/6-31G of Gaussian, respectively. Results show that the inhibitory effects on precipitation ratio decrease in the order of p-toluic acid>benzoic acid>m-toluic acid>o-toluic acid. The negative effects on agglomeration efficiency of gibbsite decrease in the order of m-toluic acid>benzoic acid>p-toluic acid˜o-toluic acid. All the phenomena correlate with the net charges of oxygen atoms in the four aromatic carboxylic anions and the dipole moments of the four aromatic carboxylic anions. Therefore, a possible electrostatic adsorption-polarization mechanism about the interaction between these inhibitors and gibbsite surfaces is proposed.

3:20 PM

Effect of Ultrasound on Particle Size Distribution of Al(OH)3 in Seeded Precipitation of Sodium Alumina Solution: *Yusheng Wu*[1]; Li Mingchun[1]; Bi Shiwen[2]; Yang Yihong[2]; [1]Shenyang University of Technology; [2]Northeastern University

To solve the problem of periodically explosive attenuation of $Al(OH)_3$ particles in seeded precipitation process of sodium aluminate solution, a systematic study was made of the effect and mechanism of ultrasound on particle size distribution of $Al(OH)_3$ crystals in seeded precipitation. The results indicate that ultrasound significantly enhance growth of $Al(OH)_3$ crystals, and optimize the particle size distribution. For instance, with ultrasound, the mean particle size of $Al(OH)_3$ increases by 4.37μm. The crystal morphologies show that the ultrasounds enhance the agglomeration process and second nucleation process.

3:45 PM Break

4:05 PM

Effect of Carbonization Seeds on Seeded Precipitation of Sodium Aluminate Solution: *Yusheng Wu*[1]; Shiwen Bi[2]; Yihong Yang[2]; [1]Shenyang University of Technology; [2]Northeastern University

To solve the problem of periodically explosive attenuation of $Al(OH)_3$ particles in seeded precipitation process of sodium aluminate solution, the effect of carbonization seeds on seeded precipitation were investigated under industrial conditions modeled in laboratory. The results show that adding carbonization seeds to Bayer process can enhance precipitation rate, reduce fluctuation of particle size and improve the intensity of $Al(OH)_3$. Analysed the physical chemistry characteristic of $Al(OH)_3$ crystals, high quality $Al(OH)_3$ product can been obtained with 200g/L carbonization seeds. SEM photomicrographs shows that carbonization seeds transformed inlay structure during seed precipitation of sodium aluminate solution.

4:30 PM
Effect of Cationic Polyacrylamide on the Seeded Agglomeration Process of Sodium Aluminate Liquors: *Jianguo Yin*[1]; Qiyuan Chen[2]; Zhoulan Yin[2]; Wangxing Li[1]; Zhonglin Yin[1]; [1]Zhengzhou Research Institute of Chalco; [2]School of Chemistry and Chemical Engineering of Central South University

Effect of cationic polyacrylamide (PAM) on the seeded agglomeration of sodium aluminate liquors was investigated, conclusions were drawn as follows. Cationic PAM can increase precipitation ratio of sodium aluminate liquors and it is 2.5% higher than the blank at the dosage of 2.5 ppm. It can also improve particle size distribution (PSD) of gibbsite products. Average size of the products is 4.6 μm larger and particles less than 45μm is 21% less than the blank at the dosage of 10 ppm. Gibbsite agglomerates with the addition of cationic PAM are made up of median size crystallines which are filled with many crystallines of 5 μm or so, and there fills with even less ones among 5 μm crystallines. That is to say, cationic PAM is beneficial to get products of higher intensity. Cationic PAM is expected to be a crystal growth modifier (CGM) or be one component of CGM.

4:55 PM
Model on Batch Seeded Gibbsite Precipitation from Bayer Liquor: *Jibo Liu*[1]; Wangxing Li[1]; Yadong Wang[1]; Zhiming Liu[2]; [1]Zhengzhou Research Institute; [2]Zhengzhou Research Institute - and - Central South University

A mathematical model incorporating concepts of various parameters evolution has been developed to predict the alumina hydrate productivity of batch seeded gibbsite precipitation process. The model is based on the theoretical concepts rather than on empirical basis, and considers the evolution of liquor composition, solid content and crystal surface area. The relationship of kinetic constant and other important parameters versus reaction conditions were determined by the laboratory experiments, these parameters can also be used in the practical process model. The value estimated by the model is well fitted with the data that detected in practical precipitation system, and the ratio of data that the error lower than 5% is exceed 95%.

5:20 PM Concluding Comments

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

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Processing Committee
Program Organizers: Weimin Yin, Williams Advanced Materials; Subodh Das, Phinix LLC; Zhengdong Long, Kaiser Aluminum Company

Wednesday PM Room: 2004
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Yansheng Liu, SECAT Inc

2:00 PM
Modelling Homogenization Heat Treatment of AA3003 Alloy: *Qiang Du*[1]; Warren Poole[1]; Mary Wells[2]; [1]University of British Columbia; [2]University of Waterloo

The homogenization treatment of AA3003 involves the growth and dissolution of inter-granular constituent particles and intra-granular dispersoids. It is a multi-scale problem involving the long-range diffusions (~10 μm) and short-range diffusions (~0.1 to 1 μm) of all of the alloying elements (Fe, Mn and Si). In this paper a comprehensive model is proposed to simulate these phenomena. It consists of 1D pseudo front tracking method for inter-granular constituent particles and a multi-precipitate growth model for dispersoids. The multi-precipitate model is developed based on a variational approach and it could capture the influence of precipitate size distribution on the overall precipitate kinetics. The abilities of the multi-precipitate growth model will be demonstrated by comparing with existing models in the literature. The comparison of the simulation results with the experimental measurement for an industrial practice of this homogenization treatment will be also be conducted.

2:20 PM
Evaluation of AA5083 Constitutive Models for Elevated-Temperature Bulge Forming Simulations under QPF Conditions: *Eric Taleff*[1]; Louis Hector[2]; Paul Krajewski[2]; [1]University of Texas; [2]General Motors R&D Center

Finite-element (FE) predictions of dome pole height and pole thickness in gas-pressure bulge forming of fine-grained AA5083 sheet under quick plastic forming (QPF) conditions are compared with experimental bulge data. We examine four material constitutive models constructed from tensile tests. These include a single-mechanism model without temperature dependence commonly used to simulate SPF processes and a single-mechanism model that accounts for temperature. A pair of two-mechanism models that account for the independent creep mechanisms observed in QPF are also investigated. One includes the effect of threshold stress and the other does not. Based upon our results, a recommendation is made as to which of the four models should be used in FE simulations of QPF processes. The applicability of material models based upon uniaxial tensile data to predictions for forming under balanced-biaxial tension is addressed in detail, and suggestions for future improvements to the exiting models are offered.

2:40 PM
A Monte Carlo Simulation of Grain Refinement during Thermomechanical Processing of an Al-Mg-Si-Cu Alloy: *Panthea Sepehrband*[1]; Shahrzad Esmaeili[1]; Haiou Jin[2]; [1]University of Waterloo; [2]Novelis Global Technology Centre

The grain refinement of a 6000 series aluminum alloy during a newly developed thermomechanical processing route has been simulated using the Monte Carlo technique. Based on the TEM studies on the as-deformed state of the alloy, the initial microstructure of the simulation is generated considering deformation inhomogeneities around large particles as well as within the heavily deformed grains. Subsequently, the simulation is formulated based on a concurrent recovery-recrystallization process and the pinning effect of precipitates. The technique provides simulated microstructures and predictions for the recrystallized fraction, grain size and grain size distribution at different stages of annealing. EBSD tests on thermomechanically processed samples are used to validate the simulation results.

3:00 PM
Microstructure and Stress Corrosion Cracking of Al-5083: *Ramasis Goswami*[1]; Peter Pao[2]; George Spanos[2]; Ronald Holtz[2]; [1]SAIC ; [2]Naval Research Laboratory

Commercial Al 5XXX alloys have been used for marine applications because they exhibit excellent resistance to corrosion. However, these alloys become susceptible to stress corrosion cracking (SCC) when exposed to temperatures in the range of 50-200°C for many days. The present investigation focuses on the effect of corrosive environments on crack propagation in Al-5083 exposed to 175°C for 10 days. Pre-cracked double cantilever beam specimens, treated with a dropwise exposure to 3.5% NaCl, were bolt loaded with a constant crack opening displacement. The microstructure, chemistry and dislocation structures at the grain boundary and inside the grains have been investigated using transmission electron microscopy (TEM) just below the crack surface. TEM reveals a large number of precipitates in the matrix, while most of the grain boundaries are covered with Al3Mg2. The correlation between the microstructure, dislocations and the stress corrosion cracking behavior will be discussed.

3:20 PM
Welding Techniques and Corrosion Behavior of 5xxx Alloy for Marine Structural Application: *Zhengdong Long*[1]; Subodh Das[2]; John Kaufman[3]; Shridas Ningileri[3]; Yufu Wang[3]; [1]Center for Aluminum Technology, University of Kentucky; [2]Phinix LLC; [3]Secat Inc

Al-Mg 5xxx aluminum alloys are broadly used in naval ship structures due to their superior strength-to-weight ratio and generally excellent salt-water corrosion resistance. However, the strength and corrosion properties are significantly affected by joining with the traditional gas metal arc welding (GMAW) process. The large heat affected zone resulting from the GMAW has relatively low strength and is susceptible to intergranular corrosion. The Navy has experienced both intergranular corrosion (IGC) and exfoliation corrosion of Al-Mg alloys used in long-term exposure in equatorial environments. The friction stir welding (FSW) technique provides less heat input, potentially providing improvement in this condition, and therefore was investigated in this study. The welding efficiency, microstructure, microhardness and corrosion resistance of FS and GMAW joints in a representative 5456-H116 plate were

compared. The FSW provides higher weld strength efficiency and may also have improved corrosion resistance.

3:40 PM
Hydrogen Generation by Aluminum-Water Reactions: *Paul Rozenak*[1]; Ester Shani[1]; [1]Hydrogen Energy Batteries LTD

The aluminum-water surface reaction in the alkaline dissolution of aluminum was studied by secondary ion mass spectroscopy (SIMS) and transmission electron microscopy (TEM). In our experiments, hydrogen (deuterium) absorption in the aluminum surface after reaction with an alkaline solution was characterized. We suggest the interpretation that anodic oxidation during the first anodic scan irreversibly converts the active hydride-covered Al surface to a passive oxide-covered surface. The result suggests that, at least in alkaline solutions, the Al dissolution pathway proceeds through a hydride oxidation step. Hydride may be formed by the etching of Al and hydrogen gas generated cathodically on the surface. Various densities and distributions of hemispherical bubbles, ranging in size from large (some micrometers in diameter) to very small (a few nanometers in diameter) were obtained in the surface hydroxide layers of aluminum.The principles of the production of gaseous hydrogen are described.

4:00 PM Break

4:15 PM
Effect of Scandium Addition on Microstructure and Corrsosion Properties of Al-Cu-Mg-Ag Alloys: *D. H. Xiao*[1]; K.H. Chen[1]; [1]Central South University

The Al-Cu-Mg-Ag alloys with different scandium contents were prepared by ingot metallurgy technology. Effect of scandium additon on microstructure and corrosion prperties of an Al-5.3Cu-0.8Mg-0.6Ag-0.2Zr alloy were investigated using optical microscope, scanning electron microscopy (SEM), transmission electron microscopy (TEM), intercrystalline corrosion and exfoliation corrosion. It has been shown that 0.3~0.5%Sc addition refines the grains of the casting alloys and the average grain size decrease from over 300 μm to 60 μm. Increasing Sc content from 0.1 to 0.3 wt% increased corrosion-resistance properties. However, the coarsenening Al3(Sc,Zr) compounds in alloys with 0.5%Sc decrease corrosion-resistant properties seriously.

4:35 PM
Splitting Water with Al Rich Alloys: Structure and Reaction Kinetics: Go Choi[1]; *Jerry Woodall*[1]; Jeffrey Ziebarth[1]; Charles Allen[1]; J-H Jeon[1]; Deborah Sherman[1]; Robert Kramer[1]; [1]Purdue University

Solid alloys of aluminum, gallium, indium and tin are capable of reacting with water at room temperature to form hydrogen, alumina, and heat. The alloys are shown to contain a phase of solid aluminum-rich grains with small amounts of gallium. In and Sn have nearly zero solid solubility in Al, and energy dispersive x-ray (EDX) analysis found In and Sn to be in the grain boundaries together with Al and Ga. It is believed that the grain boundary phase becomes liquid at or near room temperature and as a result enables reaction with water. When these alloys react with water or oxidize in air, EDX results show that the reaction occurs at or near the grain boundary. Current research efforts focus on studying the reaction mechanisms for alloy compositions containing 50 wt% and 95 wt% Al, with 95 wt% Al alloys being the more interesting of the two from an economics standpoint.

Aluminum Reduction Technology: Fundamentals

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Wednesday PM — Room: 2012
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Alton Tabereaux, Consultant

2:00 PM
Alumina Dissolution in Aluminum Smelting Electrolyte: *Xiangwen Wang*[1]; [1]Alcoa Inc

Alumina dissolution rate in cryolitic electrolytes has been a subject of intensive studies, and its relation to the alumina physical properties had been attempted through various studies over past decades as reported in the open literature. The findings from these reported lab studies have been proved to be difficult to relate alumina solubility in industrial operating cells due to lack of similarities between the two settings. Alcoa has been using a different experimental approach to measure alumina dissolution rate to monitor ore quality changes for providing necessary information to our smelting operations. This paper briefly describes the background for alumina dissolution studies, experimental setup and procedures for measuring dissolution rate. Some comparison results of typical good and bad alumina ores in term of dissolution rate are presented.

2:20 PM
Alumina Dissolution and Current Efficiency in Hall-Heroult Cells: *Bjørn Lillebuen*[1]; Marvin Bugge[1]; Helge Høie[1]; [1]Hydro Aluminium

The dissolution and distribution of alumina can be described as a coupled heat- and mass transport process, with intermediate formation of solid cryolite. Current Efficiency(CE) can be evaluated by means of the rate equations for the back reaction between dissolved metal and carbon dioxide gas. Solid cryolite may be formed close to the metal pad, under certain conditions in the cell. The bath superheat, and the mass transfer coefficient at the bath/metal interface, will be important parameters for the maximizition of CE. In some cells, there is a clear correlation seen between CE and the sodium content in the metal , indicating that mass transfer is the dominant factor. In other cells ,sodium levels can be quite low even at high CE, which can indicate that cryolite formation plays a significant role, making superheat the dominant factor.

2:40 PM
The Behaviour of Moisture in Cryolite Melts: *Karen Osen*[1]; Christian Rosenkilde[2]; Asbjørn Solheim[1]; Egil Skybakmoen[1]; [1]SINTEF Materials and Chemistry; [2]Hydro Aluminium AS

HF emissions are still an environmental challenge for the aluminium industry. Hydrogen fluoride is generated when fluorides present in the bath and in the vapour phase react with moisture. It has been established that the main sources of water are structural hydroxyl contained in the primary alumina and humidity in the air. The objective of the present work was to study the behaviour of moisture in cryolite melts. Argon saturated with water vapour was bubbled through the melt, while gas analysis with respect to HF and H_2O as well as electrochemical measurements on a gold electrode were performed. The results from the gas analysis and the electrochemical measurements demonstrate that one or several hydrogen-containing species are soluble in the melt. The results also showed that these species exhibit long residence times, i.e. they remained in the melt long after the water sypply was terminated.

3:00 PM
Physical-Chemical Properties of the KF-NaF-AlF3 Molten System with Low Cryolite Ratio: *Olga Tkatcheva*[1]; Alexei Apisarov[1]; Alexander Dedyukhin[1]; Alexander Redkin[1]; Elena Nikolaeva[1]; Yurii Zaikov[1]; Pavel Tinghaev[1]; [1]IHTE

The development of low temperature aluminum electrolysis makes possible to reduce the consumption of energy and raw materials per unit of final product and to increase the electrochemical cell lifetime. The supposed operating temperature rang of this technology is 750-850oC that corresponds to the KF-NaF-AlF3 electrolyte compositions at cryolite ratio 1,3-1,7. The liquidus temperature, electrical conductivity, density and alumina solubility in the KF-NaF-AlF3 molten mixtures at the [KF]/([KF]+[NaF]) ratio from 0 to 1 and ([KF]+[NaF])/[AlF3] ratio equal to 1,3; 1,5 and 1,7 have been measured. There is a maximum on the liquidus curves in KF-rich region and a minimum in NaF-rich region at constant CR. The electrical conductivity increases but alumina solubility decreases with potassium fluoride substitution by sodium fluoride.

3:20 PM
Aspects of Crust Formation from Today's Anode Cover Material: *Tatiana Groutso*[1]; Mark Taylor[1]; Anthony Hudson[2]; [1]Light Metal Research Centre, The University of Auckland; [2]Kempe Engineering

The structure and composition of different anode cover materials were analyzed by XRD and examined by SEM. SEM images have shown that slow sintering of alumina was a cause to conversion of transition alumina phases to alpha plates and XRD showed corundum content reached 40-45%. However, the morphology of the alpha alumina has not previously been observed and gave both the anode cover and partially crushed bath samples particular properties of hardness and resistance to crushing. Specifically, crushed bath agglomerates have "keyed" into the corundum platelets. For many agglomerates, the fused chiolite formed a casing surrounding the interlocked alpha plates. The resulting

agglomerates are prevented from breaking in normal brittle fracture through the multiple interlocked corundum "gluing" phases. The amount of chiolite increases from the bottom of the crust to the top which also increased the propensity for formation of these hard agglomerates towards the top of the crust material.

3:40 PM Break

4:00 PM
Sideledge in Aluminium Cells: The Trench at the Metal-Bath Boundary: *Asbjørn Solheim*[1]; Henrik Gudbrandsen[1]; Sverre Rolseth[1]; [1]SINTEF

The paper sums up some observations concerning sideledge in industrial aluminium cells. Some laboratory experiments using a gas-cooled cylinder (coldfinger) immersed into bath and metal are also reported. The freeze formed on the coldfinger when it was located in the bath did not melt away when it was lowered deep into the metal; this is in accordance with a recently suggested hypothesis concerning sideledge facing the metal. It appeared, however, that the freeze melted away rapidly at a zone near the metal-bath interface, even though stirring or wave motion was not applied. This observation gave rise to a new interpretation of the conditions at the ledge-metal-bath boundary. It is suggested that the trench formed at that zone is caused by a high heat transmission coefficient between metal and ledge at the meniscus formed at the ledge-metal-bath boundary.

4:20 PM
Inert Anode: Challenges from Fundamental Research to Industrial Application: Vittorio de Nora[1]; *Thinh Nguyen*[1]; [1]Moltech Invent SA

To overcome the thermodynamic penalties in cell voltage and heat generation, higher current density should be operated with oxygen evolving inert anodes retrofitted in conventional Aluminum reduction cells. Being semiconductor metallic oxides the electrochemical characteristics of inert anode active layer may be modified by diffusion interactions, oxygen activity and composition balance. Unstable cell voltage regimes have been observed with metallic inert anodes operating at high current densities; the formation of n-p semiconductor junctions may be a possible hypothesis. Several questions are still open; further fundamental research should be investigated for better understanding prior to the industrial application of oxygen evolving inert anodes.

4:40 PM
Studies on the Possible Presence of an Aluminum Carbide Layer or Bath Film at the Bottom of Aluminum Electrolysis Cells: Sverre Rolseth[1]; *Egil Skybakmoen*[1]; Henrik Gudbrandsen[1]; Jomar Thonstad[2]; [1]Sintef Materials & Chemistry; [2]Norwegian University of Science and Technology

The background of this work is the hypothesis that an aluminium carbide layer will be formed at the interface between the liquid aluminium and the carbon cathode at the bottom of aluminium electrolysis cells. It is assumed that the formation and dissolution of aluminium carbide is one of the crucial steps of the wear mechanism of carbon cathode blocks in industrial electrolysis cells. The electrical potential between liquid aluminium and the carbon cathode was measured during current interruption using specially designed probes, both in laboratory and on industrial scale. A potential difference corresponding to the theoretical aluminium carbide formation cell was found in small laboratory cells, but not in industrial cells. It is believed that when the contact area is so large as in an industrial cell, it is unlikely that a permanent coherent aluminium carbide containing layer can be established that covering the entire contact area.

5:00 PM
Solid State Carbothermal Reduction of Alumina: Dongsheng Liu[1]; *Guangqing Zhang*[1]; Jiuqiang Li[1]; Oleg Ostrovski[1]; [1]The University of New South Wales

The Hall-Heroult process, the only commercial technology for aluminium production requires high energy and is a major origin of perfluorocarbons and green house gases. A promising alternative process, carbothermal reduction of alumina to metallic aluminium has advantages of lower capital cost, less energy consumption, and lower emission of green house gases. Carbothermal reduction processes under development are based on formation of aluminium carbide-alumina melts at high temperatures. Solid state carbothermal reduction of alumina is possible at reduced CO partial pressure. This paper presents results of experimental study of carbothermal reduction of alumina into aluminium carbide in Ar, He and H_2 atmospheres at 1500-1700\\176C. The reduction rate of alumina increases with increasing temperature, and is significantly faster in He and H_2 than in Ar. Increasing gas flow rate and decreasing pressure favours the reduction.

5:20 PM
***In-Situ* Analysis Methods for Electrowinning in Chloride and Fluoride Baths**: *Kathie McGregor*[1]; Graeme Snook[1]; Andrew Urban[1]; Marshall Lanyon[1]; Nicola Scarlett[1]; Ian Madsen[1]; [1]CSIRO

The in-situ analysis of electrode and cell materials in their functional states, i.e., during electrolysis, is highly desirable. Such methods eliminate the possibility of experimental artifacts brought about by changes during sample preparation. This is particularly challenging, however, for electrochemical processes conducted at high temperatures in molten salts. In this paper, several new in-situ techniques, and their challenges and limitations, will be described: (1) Dynamic measurements of resistance and capacitance obtained simultaneously during electrolysis; (2) An innovative Fast Fourier Transform Current Pulse technique to investigate anode bubble resistance; (3) Synchrotron X-ray diffraction phase-mapping for a model inert anode material via Tomographic Energy Dispersive Diffraction Imaging (TEDDI). Application of these techniques to aluminium and titanium reduction cells will be discussed. This work was conducted as part of the CSIRO Light Metals Flagship and CSIRO Emerging Science Initiative (Synchrotron Science).

Aluminum Reduction Technology: Operational Improvements

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Gilles Dufour, Alcoa Canada, Primary Metals; Martin Iffert, Trimet Aluminium AG; Geoffrey Bearne, Rio Tinto Alcan; Jayson Tessier, Alcoa Deschambault

Wednesday PM — Room: 2001
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Mohd Mahmood, Aluminium Bahrain; Jean Crépeau, Rio Tinto Alcan

2:00 PM
AP30 toward 400 kA: *Oliver Martin*[1]; Laurent Fiot[1]; Claude Ritter[1]; Renaud Santerre[2]; Herman Vermette[1]; [1]Rio Tinto Alcan; [2]Alcan Primary Metal

After an intensive development program, two new versions of the AP30 have been developed. The AP37 cell, today available in its industrial version, is able to cope with more than 370 kA and to guarantee reliable technical and environmental performance. Industrial test has been carried out on the Alma (Canada) boosted section in order to validate the AP37 technology. The technical results of the Alma trial are presented in detail. The advantages of the new AP37 compared with the previous version of the AP30 on a greenfield project are presented. The AP39 will soon be able to cope with 400 kA without degradation of the specific energy consumption. The Saint Jean de Maurienne (France) industrial test has demonstrated reliable operation above 390 kA and gives confidence to industrialize the AP39 in high performance industrial package for future greenfield projects.

2:20 PM
Evolution of CD20 Reduction Cell Technology towards Higher Amperage Plan at Dubal: *Maryam Al-Jallaf*[1]; Ali Hussain Ahmed Mohamed Al Zarouni[1]; Arvind Kumar[1]; Mohammad Shabbir Ali[1]; [1]Dubai Aluminium Company Limited

Dubai Aluminium and Comalco (now part of Rio Tinto Alcan) jointly developed the CD20 reduction cell technology, with the first cells commissioned in 1996 at 190 kA and now currently running at 233 kA. DUBAL has since pursued an ambitious programme to optimise the cell design and its successor, the D20 cell technology, for higher amperage and production. The D20 has been continually developed over the past five years to enable this increase in amperage and production. The main changes were in lining design, anode design and operational practices i.e. anode cover thickness, bath/metal height, control of excess AlF3, AEF detection and termination and improved HF gas recovery. This paper summarises the evolution of the CD20 cell technology through to the development of the D20 cell technology, which is currently operating at 248 kA with current efficiency over 96%.

2:40 PM
Development of D18 Cell Technology at Dubal: *Daniel Whitfield*[1]; Abdulmunim Al-Moniem Said[1]; Maryam Mohamed Al-Jallaf[1]; Ali Mohamed Al Zarouni[1]; [1]Dubai Aluminium Company Limited

Despite the development and construction of new pot technologies at Dubai Aluminium, development and improvement of the original D18 cell technology has been sustained, and continues to play a significant part of the growth and expansion of the company. This paper summarises the progress of the original D18 cell technology at Dubal over the past few years, and its contribution towards the goal of 1 million tonnes plant annual (hot metal) production. Amperage has increased from an original design target of 155kA up to 196kA in 2008. To ensure adequate pot performance is maintained with this increase in production, there has been significant development of the cell alumina, bath chemistry and heat balance control. Other changes such as anode size increase, modifications to the cathode and measures to ensure busbar integrity have allowed for further planned amperage and production increase over the next five years.

3:00 PM
ACD Measurement and Theory: *Marianne Jensen*[1]; Kjell Kalgraf[1]; Tarjei Nordbø[2]; Tor Bjarne Pedersen[1]; [1]Elkem Aluminium ANS; [2]Elkem Research

People generally assume that the ACD is constant nearly all the time in prebake pots. The argument is that the anode consumption will soon bring any deviations in the ACD to zero because the current will increase where the ACD is lower and vice versa. Measurements, however, show that there are great variations in the ACD between the different anodes in the same pot. Differences of 2 cm are not uncommon, and there are also more regular variations along the pot side depending on the position in the anode set cycle. This is because anodes are being replaced, and the ACD is therefore never in equilibrium, but always in a transient state. The current distribution and the magnetic field are constantly changing and affecting the ACD distance. Findings agree with modeled results. A method was developed to measure the ACD.

3:20 PM Break

3:40 PM
Improved Cell Operation by Redistribution of the Alumina Feeding: *Bjorn Moxnes*[1]; Asbjorn Solheim[2]; Morten Liane[3]; Anveig Halkjelsvik[1]; Ellen Svinsås[1]; [1]Hydro Aluminium Sunndal; [2]SINTEF Materials Technology; [3]Hydro Aluminium Technology Centre Årdal

The local anode-cathode distance in aluminium cells, as well as the local superheat,depend strongly on the local concentration of alumina in the bath. Based on the idea that it is beneficial to have a uniform alumina concentration, a measurement campaign aiming at a redistribution of the alumina feeding was undertaken at the SU4 potline, Hydro Aluminium Sunndal. By using AlF3 as a tracer, the path of each alumina dosis could be followed by measuring the current pick-up at each individual anode. This enabled mapping of the connection between each of the alumina feeders and the anodes. Based on the measurements, the rate of each individual alumina feeder was optimised in test cells. The change turned out to be higly successful with respect to improved operational results, such as higher current efficiency, reduced anode effect frequency, and less anode problems. Optimised alumina feeding has now been implemented in the entire potline.

4:00 PM
Improving Anode Cover Material Quality at Nordural – Quality Tools and Measures: *Halldor Gudmundsson*[1]; [1]Nordural

Anode cover material (ACM) composition and granulometry determines the properties of the anode cover. This paper describes the experience Nordural has had with a new ACM mixing station employing autogeneous milling and dense phase to convey the material to the potrooms. The pros and cons of this system for delivering the required granulometry is discussed by showing the evolution of the ACM granulometry before and after the commissioning of the new mixing station. To evaluate the effect of the conveying system the granulometry of the material at the output of the mixing station is compared with the granulometry of the material on the anodes. The automated XRD/XRF method for bath analysis has been used to evaluate the alumina- and chiolite composition of the ACM which can impact the bath mass balance and cover structural stability. Finally, heat flux data is shown as one measure of quality.

4:20 PM
Comparison of Bubble Noise of Søderberg Pots and Prebake Pots: *Kjell Kalgraf*[1]; Marianne Jensen[1]; Tor Pedersen[1]; Tarjei Nordbø[1]; [1]Elkem Aluminium Research

In previous work we analyzed the relationship between bubble noise, bath height, and anode quality with data mostly from Søderberg pots. We have now acquired additional data for both Søderberg pots and for prebake pots. From an accidental current increase from 127 to 145 kA over 2 days at a Søderberg plant, we can see both the immediate increase of bubble noise proportional to the square of the current, and the gradual decline of bubble noise due to increasing crack area in the anode. For prebakes, however, the response is different. In the short run the bubble noise is increasing when bath height goes up, because of increased adhesion when pressure goes up, but after 1-2 hours the bubble noise has fallen to a value lower than before the bath height went up. This happens because less current flows in the central regions when there is more gas there, and more current flows further away from the central region, giving a steeper slope of the anode closer to the boundary. In fact, the long term decline of bubble noise for prebake pots is very similar to the decline found for Søderberg pots when bath height goes up. Because of the opposite short term and long term response, the correlation between bath height and bubble noise of prebake pots can be positive for rapid height changes and negative for height changes with a bigger time spacing.

Applicable Computing Technologies in Heat Treating: Numerical Modeling and Simulation for Heat Treatment

Sponsored by: TMS Materials Processing and Manufacturing Division, TMS/ASM: Computational Materials Science and Engineering Committee
Program Organizers: Lei Zhang, Scientific Forming Technologies Corporation (SFTC) ; Yiming Rong, Worcester Polytechnic Institute

Wednesday PM — Room: 3000
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Yiming Rong, Worcester Polytechnic Institute; Lei Zhang, Scientific Forming Technologies Corporation

2:00 PM Introductory Comments

2:05 PM
Modeling of Carbon Behaviors during Hot and Cold Rolling in Low Carbon Steels: *Kyung Jong Lee*[1]; J.M. Choi[1]; J.Y. Lee[1]; K.S. Lee[1]; K.J. Lee[1]; [1]Hanyang University

It is very important to understand interstitial carbon behaviors in cold rolled steel to get the good formability as well as the high strength. In low carbon steel, most of carbons are consumed by the formation of grain boundary cementite during cooling. During heating and holding between Ae1 and Ae3, cementite is dissolved and consequently carbon enriched austenite is formed. In this study, the effect of heating rate and holding temperature on carbon by the formation and dissolution of cementite and austenite are modeled by nucleation and growth, diffusion and dissolution. Partitioning of substitutional elements is also considered.

2:25 PM Question and Answer Period

2:30 PM
Microstructure-Based Models for the Austenitization of Steels during Industrial Heat-Treatment Processes: *Ramanathan Krishnamurthy*[1]; Narendra Singh[1]; Amy Clarke[1]; [1]Caterpillar Inc

Reliable predictions of property changes in steels during industrial heat treatment processes require accurate descriptions of the kinetics of associated solid state phase changes, and accompanying changes in microstructure, over a specimen of macroscopic dimensions. Here, we describe such a model for the kinetics of austenitization of a ferritic-pearlitic steel. Nucleation of austenite grains, cementite platelet decomposition, and carbon-diffusion driven ferrite-austenite transition are all included in the model. The effect of the initial ferritic-pearlitic microstructure on austenitization is included through the effect of the various length scales characterizing it. Temperature gradient effects and temperature and carbon concentration dependent carbon diffusivities

are also included. Model predictions compare well with continuous heating transformation curves generated from dilatation experiments. We show model predictions for spatial and temporal variations in the progress of austenitization obtained by integrating the current model within a full-scale simulation of an induction heat treatment process, to demonstrate its efficacy.

2:50 PM Question and Answer Period

2:55 PM

Multiphase Flow Model of Porosity Formation for Casting Process of Aluminum Alloy: Gang Wang[1]; *Yiming Rong*[1]; Shoumei Xiong[2]; [1]Worcester Polytechnic Institute; [2]Tsinghua University

An integrated model for the casting process of binary aluminum alloy has been implemented on CFD prediction in this paper. The model, which uses much of the multicomponent multiphase fluid architecture as a comprehensive system, involves solid-liquid change, latent heat term, Darcy-based flow in mushy zone, and entrapped air shift. It can predict the formation derived from air entrapment, and the morphology and distribution of porosity. The model is demonstrated on a representative 2-D aluminum shape casting example, in which it produces reasonable results and describes generation and distribution of porosities with detailed flow structure.

3:15 PM Question and Answer Period

3:20 PM Break

3:35 PM

Problems in Assessing Thermal Diffusivity of Steel Constituents for Quench Simulation: *Donato Firrao*[1]; Paolo Matteis[1]; Chiara Pozzi[1]; Elena Campagnoli[1]; Giuseppe Ruscica[1]; Ion Vasile[1]; Marian Miculescu[2]; [1]Politecnico Di Torino; [2] Universitatea Politehnica Bucuresti

In developing a finite-elements thermo-metallurgical model of the quench of carbon and low-alloy steels, a significant improvement can be obtained from the knowledge of the thermal diffusivity of each metallographic constituent possibly involved in the process (martensite, bainite, pearlite, stable and metastable austenite), as a function of temperature. Two medium-carbon steels designed for quenching and tempering and one low-carbon precipitation hardening steel, all employed to fabricate large plastic molds, were examined.Standard flash measurements were performed at increasing temperatures on each steel constituent and on as-received mixed-microstructure material. Non-standard measurements were performed on metastable austenite during continuous cooling, to avoid the phase transformation. The thermal diffusivity was determined by fitting the whole thermal transient data with analytical models. Two separate testing apparatuses, equipped with different vacuum furnaces, pulse sources (flash lamp, pulse laser) and temperature detectors (infrared pyrometer, thermocouple), yielded significantly different results, which are thoroughly compared and discussed.

3:55 PM Question and Answer Period

4:00 PM

Modelling of Precipitation Hardening in Aluminium Alloys with the KWN Model: *W. George Ferguson*[1]; Linda Wu[1]; [1]University of Auckland

Phase separation in supersaturated metastable solid solution is often assumed to occur in three distinct steps: nucleation, growth and coarsening. However, recent studies have shown that these processes significantly overlap which leads to the formation of a particle population that can be described by the particle size distribution (PSD). The Kampmann and Wagner Numerical (KWN) model is a powerful method for dealing with concomitant nucleation, growth and coarsening and for predicting the evolution of the size distribution. In the present work, the precipitation kinetics are modeled by the KWN method, and a strength model is used to evaluate the resulting change in strength at room temperature by taking into account contributions from the lattice resistance, solid solution hardening and precipitation hardening. The modeling is applied to isothermal and non-isothermal heat treatments and is validated by comparison with experimental results.

4:20 PM Question and Answer Period

4:25 PM

A Thermal-microstructure Model to Predict the Grain Growth of a Dual-phase Steel DP980 in Laser Heat-treatment: *Fanrong Kong*[1]; Soundarapandian Santhanakrishnan[1]; Dechao Lin[1]; Radovan Kovacevic[1]; [1]Research Center for Advanced Manufacturing

A coupled model combining an experiment-based finite element analysis with Monte Carlo method was developed to study the grain evolution in the heat-affected zone (HAZ) of heat treated dual phase steel DP 980 by direct diode laser. In this study, an energy distribution model matching the peculiarity of direct diode laser beam was considered to obtain the temperature field. In addition, the Monte Carlo method was applied to simulate the grain growth in the HAZ based on the calculated temperature history of heating and cooling process. The martensite decomposition in the HAZ of DP 980 has also been involved into consideration. The results show that temperature gradient in the HAZ decreases with an increased scanning speed for the fixed laser power. Thereby, the mean grain size of HAZ becomes finer and the percentage of martensite decomposition in the HAZ will be smaller with increase in the laser scanning speed.

4:45 PM Question and Answer Period

4:50 PM

Surface Modification of Tool Steel AISI S7 by Using High-Power Direct Diode Laser: *Soundarapandian Santhanakrishnan*[1]; Fanrong Kong[1]; Dechao Lin[1]; Radovan Kovacevic[1]; [1]Southern Methodist University

Key components used in die making industries require high quality molds with superior surface quality and mechanical properties. Tool steel AISI S7 has found an application in this area since it is characterized with shock and impact-resistance. To retain the core property and to produce a hardened surface, laser surface hardening technique can be used. The objective of this work is to determine the surface hardening conditions by using a high power direct diode laser. Compared to other lasers, direct diode laser has several unique properties for localized heat treatment such as a rectangular (12x1 mm) beam footprint and shorter wavelength (808 nm). Results show that the treated area includes a melted zone and a heat affected zone (HAZ), and their geometry depends on the process parameters. A machine vision system including a high speed camera and an optical filter was developed to monitor the treated area in real-time.

5:10 PM Question and Answer Period

5:15 PM

Study of Natural Convection in a Closed Square Cavity: *Xiang-mei Li*[1]; Jie-yu Zhang[1]; Wei-hua Qi[1]; Bo Wang[1]; [1]Shanghai University

Natural convection flow analysis in closed cavities has many thermal engineering applications, such as cooling of electronic devices, energy storage systems and the process of solidification. In particular, natural convection in the thermally driven closed cavity is one of the classical problems. In this paper, the feasibility of the investigation of heat and momentum transfer by using CALCOSOFT software was testified in comparison with some reported results. Simultaneously, numerical solutions were presented by studying the influences of Rayleigh number on the streamlines, isotherms, and the variation of velocity, temperature and Nusselt numbers. With the increase of Rayleigh number, the heat transfer mechanism was changed from heat conductivity to natural convection, and boundary layer began to form near the sidewalls. Dimensionless velocity in the midpoint of the cavity was equivalent to zero while dimensionless temperature was equal to 0.5. And Nusselt and Rayleigh numbers possiblely abides by a certain rule.

5:35 PM Question and Answer Period

5:40 PM

Solution on the Heat Transfer Coefficients during the Heat Treatment Process of a Turbine Disk: Jiafeng Zhang[1]; *Jinwu Kang*[1]; Baicheng Liu[1]; Jinwen Zou[2]; Shunquan Liu[2]; [1]Tsinghua University; [2]Institute of Aeronautical Materials

Heat treatment, as one of the most important processes of manufacturing P/M superalloy turbine disk, determines its final microstructure and properties. It is necessary to understand the cooling potential of the quenching media and the actual cooling rate of the disk to ensure fast and even cooling and avoid defects such as deformation and cracks. In this paper, a series of quenching experiments of a sample turbine disk were carried out. Thermal couples were placed at center of the disk with different depth. And only the measured surface was exposed

while the other surfaces were insulated to ensure one dimensional heat transfer. Based on the cooling curves of the measured points, the heat transfer coefficient was calculated by the inverse heat transfer method. The heat transfer coefficients of the top and bottom surfaces were obtained during forced air cooling and oil quenching.

Biological Materials Science: Implant Biomaterials II - Scaffolds

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Biomaterials Committee, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Ryan Roeder, University of Notre Dame; John Nychka, University of Alberta; Paul Calvert, University of Massachusetts Dartmouth; Marc Meyers, University of California

Wednesday PM — Room: 3014
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Ryan Roeder, University of Notre Dame; Paul Calvert, University of Massachusetts

2:00 PM Keynote
PEEK Biomaterials: From Isoelastic Hip Stems to Bone Scaffolds: *Steven Kurtz*[1]; [1]Drexel University and Exponent

Developed in the 1980s, Polyetheretherketone (PEEK) is a relatively new structural biomaterial that is gaining increased acceptance for use in orthopedic and spine implants. With over a decade of clinical experience, PEEK composites can be tailored with elastic moduli ranging from titanium alloy, at the high end, to cortical and cancellous bone, at the low end of the spectrum. Used initially in spinal cages due to its MRI-compatibility, recent PEEK research has been focused on isoelastic hip stems, thin orthopedic bearings, and bioactive composites. This talk will provide researchers with an overview of current orthopedic and spinal applications for PEEK biomaterials, a primer on the biocompatibility and biotribology of PEEK, as well as and recent advances in bioactive PEEK composites.

2:40 PM
In-Vitro Bioactivity and Mechanical Properties of a Novel Implantable Biomaterial: Nano-Tricalcium Phosphate-Silicone Rubber Nanostructured Composite: Jinesh Shah[1]; Wah Wah Thein-Han[1]; *Qiang Yuan*[1]; Devesh Misra[1]; [1]University of Louisiana

An excellent vehicle to achieve the objective of good cell attachment and proliferation of fibroblast and osteoblast in conjunction with the desired mechanical properties in an implant is to consider compounding a bioactive material with the superior mechanical properties of a scaffold. The approach to accomplish this objective involves the synthesis of tricalcium phosphate (TCP) nanoparticles using the concept of reverse micelle, which are dispersed via shear mixing and ultra-sonication, followed by cryo-compounding with silicone rubber (SR) and pressure-induced solidification. Experiments using the approach have confirmed that high strength-at-break and undiminished intrinsic ductility of silicone rubber and high cytocompatibility are achieved by uniquely combining the high-extensibility of silicone rubber with bioactive and bone-bonding properties of nano-TCP. Such composites represent a new class of biomaterials for biomedical implants and scaffolds, where ultra-fine surface features are used to modulate cell-substrate interactions and to ensure the long term stability of the implant.

3:00 PM
Mechanical Behavior of Hydroxyapatite Whisker Reinforced Collagen Scaffolds: *Robert Kane*[1]; Ryan Roeder[1]; [1]University of Notre Dame

Hydroxyapatite-collagen composite scaffolds have been investigated as a tissue engineering scaffold that mimics the extra-cellular matrix of bone tissue. While numerous hydroxyapatite-collagen scaffolds have been fabricated, the effects of hydroxyapatite addition on the mechanical properties of the scaffolds have not been systematically investigated. Freeze-dried collagen scaffolds were fabricated with equiaxed or unidirectional pore structures, three hydroxyapatite whisker reinforcement levels (1:1, 2:1, and 4:1 mass ratio of hydroxyapatite to collagen), and cross-linked by heating to 105°C for 24 h under vacuum. HA whiskers were observed to be aligned within each collagen strut. Unconfined uniaxial compression tests showed that increased levels of hydroxyapatite resulted in an increased apparent elastic modulus and strength for scaffolds with either an requiaxed and unidirectional pore structure.

3:20 PM
Pre-Osteoblast Response of Biomimetic Chitosan/Nano-Hydroxyapatite Composite Scaffolds for Bone Tissue Engineering: *Wah Wah Thein-Han*[1]; Devesh Misra[1]; [1]University of Louisiana

We describe here three dimensional biodegradable chitosan-nanohydroxyapatite (nHA) composite scaffold with improved mechanical, physico-chemical, and biological properties compared to pure chitosan scaffolds for bone tissue engineering. High and medium molecular weight chitosan scaffolds with 0.5, 1, and 2 wt.% fraction of nHA were fabricated by freezing and lyophilization. The nanocomposite scaffolds were characterized by a highly porous structure with interconnected pores and the pore size was similar for the scaffolds with varying content of nHA. The nanocomposite scaffolds exhibited greater compression modulus, slower biodegradation rate and reduced water uptake, but the water retention ability was similar to pure chitosan scaffolds. Favorable biological response of pre-osteoblast (MC 3T3-E1) on nanocomposite scaffolds includes improved cell adhesion, higher proliferation, and well spreading morphology in relation to pure chitosan scaffold. The study underscores chitosan-nHA composite as a potential scaffold material for bone regeneration.

3:40 PM Break

3:50 PM
Genetically Engineered Inorganic-Binding Peptides for Medical Applications: *Candan Tamerler*[1]; Mehmet Sarikaya[2]; [1]Istanbul Technical University; [2]University of Washington

We utilize peptides and protein constructs as molecular building blocks in synthesizing, assembling, and fabricating materials systems. The major components in this approach are inorganic binding polypeptides which are selected through combinatorial biology methods and tailored for their functionality through post selection engineering approaches. Here, we will explain how genetic engineering tools can be employed for tailoring functionality, and then present examples from different medical application areas by describing their use: i. On calcium phosphate materialization with controlled morphology. ii. As molecular films and scaffolds in developing biocompatible materials, including testing engineered peptide effects on cell proliferation, adhesion and toxicity. Here, we will summarize our work on the induced mineralization on scaffolds prepared by hydroxyapatite-binding peptides conjugated with peptide hydrogels. The results show that combinatorially selected peptides may be used to tailor morphogenesis of calcium phosphate in restoration or regeneration of hard tissues such as those in teeth and bone.

4:10 PM
Creation of Ovalbumin Based Porous Scaffolds for Bone Regeneration: Gabrielle Farrar[1]; Justin Barone[2]; *Abby Morgan*[1]; [1]Materials Science and Engineering, Virginia Tech; [2]Biological Systems Engineering, Virginia Tech

Despite recent discoveries in tissue engineering, there is still a need for bio-based materials due to synthetic polymer failure. 3D porous bio-based scaffolds have been made in the past; however ovalbumin has not been researched. Ovalbumin is a natural protein, therefore, ovalbumin cross-linked with glutaraldehyde was the focus in this research. Salt leaching and freeze drying were used to create interconnected porous structures needed for tissue formation. Mechanical properties were determined using compression tests and DMA. Thermal properties were investigated using DSC and beta sheet formation using FTIR spectroscopy. Scaffolds were sterilized with ethylene oxide prior to seeding. WST-1, von Kossa and live/dead assays were used to examine proliferation, calcium deposits and gluteraldehyde toxicity on cells. OPN and ALP levels were also tested to determine cell differentiation and mineralization. Biodegradability was investigated to determine mass loss. This work demonstrated the use of ovalbumin scaffolds for bone tissue engineering applications.

4:30 PM
Templated Precipitation and Growth of Calcium Phosphate Nanocrystals on Self-Assembling Ionic Block Copolymers: *Yusuf Yusufoglu*[1]; Mathumai Kanapathipillai[1]; Aditya Rawal[2]; Yanyan Hu[1]; Yunus Kalay[1]; Klaus Schmidt-Rohr[1]; Surya Mallapragada[1]; Mufit Akinc[1]; [1]Iowa State University; [2]University of California, Santa Barbara

In an effort to imitate the growth of natural bone, polysulfobetaine-based zwitterionic and poly acrylic acid-based self-assembling ionic pentablock

copolymers were employed as templates for growth of calcium phosphate nanocrystals from aqueous solutions. Calcium and phosphate ions were dissolved in block-copolymer micellar dispersion at low temperatures and hierarchically assembled nanocomposite calcium phosphate-copolymer gels were prepared at pH ~5. As the ions were driven into the interstitial cavities of polymer micelle structures by aging at ambient temperature, inorganic nanoparticles were formed at the polymer-inorganic interface, presumably nucleated by ionic interactions. XRD experiments revealed that calcium phosphate in the zwitterionic copolymer gel was natural brushite, while the one in PAA-based pentablock gel was synthetic brushite. TEM, solid-state NMR and SAXS studies showed that calcium phosphate precipitated on and interacted with the polymer micelles forming approximately 15 nm diameter nanospheres. Further, inorganic fraction of the nanocomposite was around 30 wt% of dried hydrogel.

Bulk Metallic Glasses VI: Joint Session of Mechanical Behavior of Nanostructured Materials and Bulk Metallic Glasses VI: Mechanical Behavior of Nano and Amorphous Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee; Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Wednesday PM — Room: 3012
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Nathan Mara, Los Alamos National Laboratory; Julian Raphael, Columbus McKinnon

2:00 PM Keynote
Micromechanisms for Plastic Flow in Nanocrystalline and Amorphous Metals: *Frans Spaepen*[1]; [1]Harvard University

Amorphous or glassy metals can be considered the ultimate nanostructure, in that their microstructural length scale is the atomic one. Correspondingly, their strength approaches the theoretical strength of the material. In the absence of lattice periodicity, plastic deformation of glassy metals can not occur by the motion of dislocation. Instead, the deformation occurs by sequential shear of equiaxed pockets of atoms. It has recently become possible to observe this process directly by confocal tracking of the particles in a colloidal glass. Nanocrystalline materials, even with very small grain size, still have a structure that is fundamentally different from that of a glass. It is interesting, therefore, to compare the mechanical properties and flow mechanisms in the two types of materials.

2:30 PM Invited
Systematic Studies of the Hall-Petch Breakdown: *Christopher Schuh*[1]; Jason Trelewicz[1]; [1]MIT

The breakdown of Hall-Petch scaling in the finest nanocrystalline metals has been the subject of speculation and controversy for some time. With recent advances in techniques to synthesize nanocrystalline alloys and control their grain size, new possibilities have emerged for systematic experimental exploration of the Hall-Petch breakdown. In this talk, our work on binary nanocrystalline alloys with grain sizes ranging from 2-200 nm is discussed. The scaling of strength, as well as its rate-, pressure-, and temperature-dependence are systematically revealed across the entire range of the Hall-Petch breakdown. Activation energies, activation volumes, and pressure coefficients are revealed through the regime of mechanistic changes. Additionally, the effects of annealing, relaxation, and alloy composition are addressed. Some points of confusion in the literature are clarified through these data, such as the presence vs. absence of "inverse Hall-Petch" scaling, the role of alloy composition, and the tendency for shear localization.

2:50 PM
Atomic Scale Study of Plastic-Yield Criterion in Nanocrystalline Metals Using Molecular Dynamics Simulations: *Avinash Dongare*[1]; A. Rajendran[2]; B. LaMattina[2]; M. Zikry[1]; Donald Brenner[1]; [1]North Carolina State University; [2]Army Research Office

The plastic deformation mechanisms of nanocrystalline materials depend on the interplay between dislocation and grain boundary processes. A reduction in grain size results in an increase in yield strength of materials, a relation known as the Hall-Petch effect. Recent studies indicate that the increase in strength with decreasing grain size reaches a maximum after which further a decrease in the grain size (less than ~ 15 nm) results in the weakening of the metal due to the shift in the dominating mechanism of plastic deformation from dislocation induced plasticity in the case of coarse grained materials to grain boundary sliding in the case of ultra-small grain sizes. The commonly used yield criteria for polycrystalline metals and alloys are the Tresca and the von Mises criteria. These criteria are based on the maximum shear stress during loading, and the fact that the deformation of the metals is primarily due to the motion of dislocations. As a result, it can be expected that the yield criterion needs to be modified to account for the change in deformation mechanisms at the ultrafine grain size (= 10 nm) of nanocrystalline metals. The plastic-yield surface (three-dimensional) for these ultra-fine grain sized nanocrystalline Cu during multi-axial loading at room temperature will be presented. I addition we will discuss the inclusion of a normal stress dependence in addition to the maximum shear stress in the criterion to predict the yield surface for nanocrystalline metals.

3:05 PM
Microstructure and Wear Resistance of Vacuum Hot Pressed Ti-Based Bulk Metallic Glass Composites: *Pee-Yew Lee*[1]; Chih-Feng Hsu[1]; Hong-Ming Lin[2]; [1]National Taiwan Ocean University; [2]Tatung University

In the present study, Ti50Cu28Ni15Sn7 metallic glass composite powders were successfully synthesized by mechanical alloying of powder mixtures of pure Ti, Cu, Ni, Sn, and carbon after 8 h of milling. The metallic glass composite powders were found to exhibit a large supercooled liquid region before crystallization. The thermal stability of the glassy matrix is affected by the presence of the carbon particles. Bulk metallic glass composite compact discs were obtained by consolidating the 8 h as-milled composite powders by a vacuum hot pressing process. Although the hardness of carbon/Ti50Cu28Ni15Sn7 bulk metallic glass composites is increased with carbon addition, the wear resistance of the composites is not commensurate with their high hardness. The residual porosity and in situ-formed hard TiC particles inside the matrix of composites may be related to the increase in wear rate of Ti50Cu28Ni15Sn7 bulk metallic glass composites with high carbon content.

3:20 PM Break

3:30 PM
Electron Irradiation Induced Amorphization and Crystallization in Metallic Materials: *Takeshi Nagase*[1]; [1]Osaka University, Research Center for Ultra-High Voltage Electron Microscopy

Atom-displacement mediated phase transitions between a glass phase and a crystal phase, namely, solid-state amorphization and crystallization not by thermal process but by mechanical process, can be induced by various techniques such as electron-irradiation (E-IR), severe plastic deformation (SPD), mechanical milling (MM), shot peening and so on. Among these processes, E-IR is an attractive technique because in-situ observations of the transition can be achieved without heavy contamination, or change in chemical composition, and with negligible temperature rise. Recently a unique disordering-ordering phase transition in metallic glass alloys driven by mechanical atom-displacement was found to take place during the MM and E-IR processes: a cyclic crystalline-amorphous (Cyclic-CA) transformation during MM, and a crystal-to-amorphous-to-crystal (C-A-C) transition during E-IR. In the present study, the electron irradiation induced C-A-C transition in metallic glasses will be reviewed. Materials discussed here include metallic glasses such as binary Zr-based, ternary Fe-Nd-B and Fe-Zr-B alloys.

3:45 PM
Co-Deformed Metallic Glass/Light Alloy (MEGA) Sandwiches: Jennifer Ragani[1]; Antoine Volland[1]; Sebastien Gravier[1]; *Jean-Jacques Blandin*[1]; Michel Suery[1]; [1]Grenoble Institute of Technology

MEtallic Glass / light Alloys (MEGA) multilayered materials were elaborated by high temperature co-deformation of the glass and the light alloy. The produced

multimaterials associate a zirconium based bulk metallic glass to light alloys (i.e. aluminium or magnesium alloys). Co-deformation was performed above the onset glass transition of the glass and the process conditions were selected on the one hand from the rheologies of both the metallic glass and the light alloys and on the other hand from information related to the thermal stability of the glass. After elaboration, structural and mechanical characterisations of the sandwiches were carried out. The effect of the ratio of the strains undergone by the glass and the light alloy during the process has been investigated. It was concluded that the quality of the bonding depends strongly upon the strain undergone by the glass during the process.

4:00 PM Invited
Strain Softening and Sample Size Effects in Bulk Metallic Glasses: *Hongbin Bei*[1]; S Xie[2]; S Shim[1]; Easo George[1]; [1]Oak Ridge National Laboratory; [2]The University of Tennessee

Plastic deformation in crystalline metals occurs by the motion and multiplication of dislocations. In contrast, BMGs deform by highly localized shear bands. Therefore, there is much interest in understanding the relationships between shear band formation and mechanical properties. In this talk, first we will discuss shear banding induced softening in a Zr-based BMG. Systematic strain-induced softening was observed in the BMG, which contrasts sharply with the hardening typically observed in crystalline metals. Second, we will discuss how sample size affects mechanical behavior in both compression and indentation tests. In compression, even at the millimeter scale, stable shear band propagation and extensive plastic deformation can be achieved in small specimens in contrast to large specimens which fail catastrophically after limited plastic deformation. In indentation, it is found that the maximum shear stress to initiate plasticity in a Zr-based BMG is almost constant when the indenter radius is smaller than 70 μm.

4:20 PM
Fatigue and Fracture Behavior of a Ca-Based Bulk Metallic Glass: *Julian Raphael*[1]; Gongyao Wang[2]; Peter Liaw[2]; Oleg Senkov[3]; Daniel Miracle[4]; [1]Columbus McKinnon Corporation; [2]University of Tennessee; [3]UES, Inc.; [4]Air Force Research Laboratory

The compression and fatigue behavior of a $Ca_{65}Mg_{15}Zn_{20}$ bulk-metallic glass (BMG) was studied in air at room temperature. During the preparation of cubical samples of the $Ca_{65}Mg_{15}Zn_{20}$ for compression and fatigue investigations, small spherical cavities were found. Under both monotonic and cyclic compression loadings of the samples, fractures initiated in these cavities and propagated in a direction parallel to the loading axis. Finite element analysis (FEA) was used to model the fracture behavior. The FEA of a centrally located spherical void showed that under compression loading large tensile stresses evolved in the cavities. The orientation of the maximum principal stress was normal to the direction of crack propagation. FEA of a void located near the loaded surfaces was also performed and the influence of void location in the cubical sample on the fracture behavior was quantitatively discussed.GYW and PKL are very grateful for the support of NSF IMI Program.

4:35 PM Invited
Fracture and Strength of Bulk Metallic Glasses: *Z. F. Zhang*[1]; R. T Qu[1]; [1]Institute of Metal Research

Tension and compression tests were systematically applied to various bulk metallic glassy (BMG) materials at room temperature for comprehensive understanding of their fracture and strength behavior. It is found that the fracture behavior of metallic glasses is strongly affected by the loading mode. Based on the experimental results, we propose an ellipse criterion as a new failure criterion to unify the four classical criteria above and apply it to exemplarily describe the tensile fracture behavior of BMGs as well as a variety of other materials. It is suggested that each of the classical failure criteria can be unified by the present Ellipse criterion depending on the difference of the ratio $\alpha = \tau_0/\sigma_0$. In addition, we designed some new tests to prove that the Ellipse criterion is better than the Mohr-Coulomb criterion. Furthermore, we developed the Ellipse criterion into a more general case through introducing a new parameter β.

4:55 PM
The Investigation of the Correlation between the Structure Evolution in the Elastic Region and the Plasticity of CuZrAl Bulk-Metallic Glasses with In-Situ Synchrotron X-Ray Measurement: *Feng Jiang*[1]; Yandong Wang[2]; Yang Ren[3]; Lu Huang[4]; Yan Li[4]; Tao Zhang[4]; Taleshi Egami[1]; Peter Liaw[1]; Hahn Choo[1]; [1]University of Tennessee; [2]Northeastern University; [3]Argonne National Laboratory; [4]Beijing University Aeronautic and Astronautic

In the (Cu50Zr50)100-xAlx (x = 4, 5, 6, and 8) glass-forming alloys, it has been found that minor deviations in composition can drastically change the plasticity of the alloys. The anisotropy in the structure function, S(q), and the atomic pair density function, g(r), was measured at ambient temperature with an in-situ high-energy synchrotron x-ray diffraction facility equipped with a loading device. The changes in the peak width of S(q) of (Cu50Zr50)100-xAlx (x = 4, 5, 6, and 8) alloys indicate that the more homogeneous the atomic-level behavior, the better the plasticity.

5:10 PM Invited
New ZrCuNiAl Bulk Metallic Glasses with Superhigh Glass-Forming Ability: *Jun Shen*[1]; [1]Harbin Institute of Technology

Three new Zr-Cu-Ni-Al bulk metallic glasses were developed through appropriate mixing of three binary eutectics Zr44Cu56, Zr51Al49 and Zr64Ni36. By suppressing solidification of competing crystalline phases, a new glass forming alloy Zr50.7Cu28Ni9Al12.3 with the critical diameter of up to 14 mm is obtained. Structural analysis identified by Synchrotron radiation high energy X-ray diffraction (HEXRD) shows the first peaks for the three new alloys in the atomic pair distribution function curves all split into two sub-peaks. Based on D.B. Miracle's theoretical model, we consider substitution of smaller Cu atoms for Zr in the three investigated alloys is a very comfortable path to stabilize the ECP structure, which lead to an increased GFA.

5:30 PM Invited
Glass-Forming Ability and the Competitive Crystalline Phases for the Light-Weighted Ti-Be Based Alloys: *Yong Zhang*[1]; WeiGui Zhang[1]; JunPin Lin[1]; GuoJian Hao[1]; GuoLiang Chen[1]; [1]University of Science and Technology Beijing

The glass forming ability (GFA) for the Ti-Be based alloys in the Ti-Be-Zr ternary alloy system was systematically studied. It is found that the best GFA obtained at a composition of Ti47Be34Zr19 in the Ti-Be-Zr ternary alloy system, and the bulk metallic glass (BMG) rod samples with diameter of 5 mm were fabricated by conventional Cu-mold casting. The competitive crystalline phases around the composition of the best GFA were determined by scanning electron microscopy (SEM) and X-ray diffractometer (XRD). The GFA of the ternary alloys were further improved by addition of a small amount of Vanadium. The largest supercooled liquid region DeltaTx (DeltaTx=Tx-Tg, Tg is the glass transition temperature and Tx the crystallization temperature) in the ternary alloy system reaches 110 K for the Ti35Be32Zr33 alloy.

Bulk Metallic Glasses VI: Structures and Modeling

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

Wednesday PM — Room: 3007
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Mo Li, Georgia Institute of Technology; Wendelin Wright, Santa Clara University

2:00 PM Invited
Mechanical Response and Atomic Structure Characterization of Model Metallic Glasses: *Mo Li*[1]; [1]Georgia Institute of Technology

Mechanical responses of several model metallic glasses under various external loadings are investigated using extensive molecular dynamics simulations, including shear, tension, compression, hydrostatic pressure, and bending. The structure changes associated with the mechanical deformation are characterized. In particular, the volume dilatation is seen to occur almost universally in all samples with the metallic bonding under these deformation modes, which is

closely related to the atomic packing and neighbor change. Of particular interest is the behavior of the model metallic glasses under compression. The systematic structure evolution is presented with an emphasis on the thermodynamic mechanism of stability and first-principle calculations.

2:15 PM
Experimental Characterization of Shear Transformation Zones for Plastic Flow of Bulk Metallic Glasses: *Mingwei Chen*[1]; [1]Tohoku University

The basic units of plastic flow of BMGs, in a form of a small cluster of randomly close-packed atoms known as shear transformation zones (STZs), are the key to establish a fundamental model of deformation of BMGs at low temperatures. However, despite of extensive theoretical predictions and MD simulations, a direct experimental portrayal of STZ volumes in BMGs is still missing due to their small length scales and diminutive time scales. Here we report an experimental characterization of STZ sizes by proposing an experimental approach based on a newly-developed cooperative shearing theory and traditional deformation thermodynamics. By determining the strength and its rate sensitivity, we measured STZ volumes of a variety of BMGs, which offers compelling evidence that the plastic flow of BMGs occurs through cooperative shearing of unstable clusters of atoms activated by shear stresses. This study offers a new way to gain a quantitative insight into the atomic-scale mechanisms of BMG mechanical behaviour and has implications for characterizing the physical processes in the dynamics and rheology of noncrystalline solids.

2:25 PM Invited
Structure of Metallic Glasses: Beyond Pair Correlation Functions: *Todd Hufnagel*[1]; [1]Johns Hopkins University

Traditional scattering techniques can reveal some aspects of the atomic short-range order of metallic glasses (through pair correlation functions) but cannot provide information about higher-order (three- and four-body) correlation functions or about structure over longer length scales (so-called "medium range order"). Here, we discuss the the application of fluctuation electron microscopy to structural characterization of metallic glasses. We show that models of atomic-scale structure that are consistent with highly constrained pair correlation data from Pd-Ni-P metallic glasses are not improved by adding variable coherence fluctuation electron microscopy data as an additional constraint. This implies that the fluctuation signal is largely determined by the pair correlations, and is not the result of additional medium-range order. We discuss the implications of this finding for our understanding of metallic glass structure, as well as prospects for future progress.

2:40 PM
Elementary Shear Banding in Model Metallic Glasses: *Craig Maloney*[1]; Anael Lemaitre[2]; [1]Carnegie Mellon University; [2]Institut Navier

We present results on an extensive set of computer simulations on simple model atomistic amorphous solids in 2D. The deformation which results is shown to consist of avalanches of plastic activity which localize onto elementary shear bands, similar to those observed in analog macroscopic laboratory models such as bubble rafts and foams. These elementary shear bands have widths on the order of atomic dimensions and their role in the formation of the much larger-scale shear bands which are observed experimentally is currently a crucial open question. Key results are the emergence of a quantum of slip analogous to a Burgers vector and a universal value of the yield strain in good agreement with experiment.

2:50 PM Invited
Molecular Dynamics Simulations of Poisson Ratio Effects in Metallic Glasses: *James Morris*[1]; Rachel Aga[1]; Takeshi Egami[2]; Valentin Levashov[2]; [1]Oak Ridge National Laboratory; [2]University of Tennessee

Recent studies have indicated correlations between the Poisson ratio of a metallic glass and its properties. We provide direct evidence for this, using a new atomistic model that allows us to tune the Poisson ratio without changing the cohesive energy, lattice parameter, or bulk modulus of the crystalline phase. An increase in the Poisson ratio (from ~0.25 to ~0.3) dramatically stabilizes the disordered phase. The melting temperature drops nearly 30%, due in large part to a lower enthalpy of the liquid phase. The diffusion barrier drops by a comparable amount. We demonstrate the importance of shear fluctuations in the liquid properties, and that changes in this are correlated with changes in the liquid's structure and viscosity. Experimentally observable effects will be discussed. 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 DE-AC05-00OR-22725 with UT-Battelle.

3:05 PM Invited
Bond Deficiency Defects Assisted Flow in Amorphous Metals: *Aiwu Zhu*[1]; Gary Shiflet[1]; Joseph Poon[1]; [1]University of Virginia

Atomic bond deficiency (BD), as characteristic defects, is considered to assist atomic rearrangement for mass transports in amorphous metals. Plastic strain (flow) response to external shear stress is attributed to local cooperative movements of multiple adjacent atoms that are facilitated by the aggregated BD defects. This complements the STZ model and can be formulated to reproduce observed features of steady-state flows. Additionally, it explains one of the puzzles concerning the density of expected defects for flow which is only the square of that for diffusion.

3:20 PM Break

3:30 PM
Nanoindentation of Amorphous Alloys with Various Indenter Tips: *Jae-il Jang*[1]; Young-Wook Park[1]; So-Jung Kwon[1]; Byoung-Wook Choi[1]; Byung-Gil Yoo[1]; [1]Hanyang University

In this study, we critically evaluated the influence of indenter geometry on the nanoindentation-induced elastic/plastic deformation of amorphous alloys which are known to exhibit an elastic-perfectly-plastic deformation and (arguably) no indentation size effect and thus are good for the analysis on the basis of classical contact mechanics theories. After performing nanoindentation experiments with a series of triangular pyramidal indenters having different centerline-to-face angles as well as various spherical indenters having different radii, we carefully compared the mechanical responses from spherical indentations to those from geometrically self-similar sharp indentations. Results are discussed in terms of the relationship betwwn the constraint factor and the plasticity index. * This work was sponsored by Hanyang Fusion Materials Program funded by Ministry of Education, Science and Technology, Korea.

3:40 PM Invited
Molecular Dynamics Simulation of Structure and Liquid-Glass Transition in Cu-Zr Alloys: *Mikhail Mendelev*[1]; Ryan Ott[1]; Matthew Kramer[1]; Daniel Sordelet[1]; [1]Ames Laboratory

The diffraction experiments provide only information about pair correlations in non-crystalline materials and even these data are averaged over different types of atoms. Therefore, atomistic computer simulation is required for detailed analysis of structure. In this talk, we will discuss how semi-empirical interatomic potentials can be developed to provide an excellent agreement between molecular dynamics (MD) simulation and diffraction data for Cu-Zr alloys. Next we present the results of MD simulation of the structure and thermodynamics properties of these alloys. A special attention will be paid to the concentration dependences of liquid diffusivities and their relation to the best glass forming composition in this system. Finally, we will show how these properties depend on the cooling rate which varies in our simulations from 1e14 K/s to 5e9 K/s.Work at the Ames Laboratory was supported by the Department of Energy, Office of Basic Energy Sciences, under Contract No. DE-AC02-07CH11358.

3:55 PM Invited
Experimental Studies of the Amorphous and Liquid Structures in the $Cu_{1-x}Zr_x$ Binary System: *Matthew Kramer*[1]; [1]Iowa State University

The short-range order of the bulk metallic glasses has been postulated to be dominated by icosahedral order. High energy X-ray scattering studies have been performed on compositions in the $Cu_{1-x}Zr_x$ binary system to explore the relationship between the topological and chemical order in the liquid state and the amorphous state. In all compositions studied, the first diffuse scattering peak sharpens and shifts to a higher Q with undercooling. However, the most notable change in the S(Q) in the deeply undercooled state is a sharpening in the low Q side of the 2nd diffuse scattering peak which shifts to a lower Q with increasing undercooling. While the amorphous alloys show a clear bifurcation in the second diffuse peak, this is not observed in the undercooled liquid. The changes in the short range order will be discussed in light of molecular dynamics simulations using ab initio and embedded atom methods.

4:10 PM Invited
Molecular-Dynamics Study of Shear Band Formation and Propagation in Zr-Based Metallic Glass under Indentation: *Yun-Che Wang*[1]; Hong-Chang Lin[1]; Chun-Yi Wu[1]; Fengxiao Liu[2]; Chi-Chung Hwang[1]; Jinn Chu[3]; Yanfei Gao[2]; Peter Laiw[2]; [1]National Cheng Kung University; [2]The University of Tennessee; [3]National Taiwan University of Science and Technology

Formation and propagation of shear bands in metallic glasses dominate their mechanical properties. In this paper, the molecular dynamics models of the Zr-based metallic glass are first deposited with simulated sputtering processes. Then, the as-deposited films are used as initial structures for subsequent nano-indentation simulations. For the deposition simulations, a many-body, tight-binding potential is adopted for interatomic interactions among the multiple species of atoms. Interactions between metallic atoms and working gas (Ar+) are modelled with the pair-wise Moliere potential. As for indentation simulations, a right-angle conical indenter tip is adopted, and homogeneous flow occurs to form pile-ups on the surface of the metallic glass; a signature for amorphous materials under indentation. Both three-dimensional stress and strain calculations reveal the formation and propagation of shear bands under the indenter tip and near the film-substrate interfaces. In addition, effects of loading rate are investigated.

4:25 PM Invited
The Effects of Crystalline Phases on the Deformation and Fracture Behaviors of Fe-Based Bulk Metallic Glassy Alloys: *Ke-Fu Yao*[1]; Chang-Qing Zhang[1]; Feng-Juan Liu[1]; [1]Tsinghua University

Different from the traditional glassy alloy which possesses limited whole plasticity, recently it has been found that some bulk metallic glasses exhibit high plasticity despite that the reason has not well been understood. Then investigating the factors influencing the deformation behavior of metallic glasses is meaningful and important, both for the understanding the deformation mechanism and for improving the mechanical properties of the glassy alloys. Here, we report that the in-situ formed nanocrystalline phases possess significant influence on the deformation ability and fracture behavior of bulk metallic glasses. For an Fe-based alloy, the full glassy alloy exhibits good plasticity, while with the increase of the crystalline phases, the whole plasticity of the alloys decreases greatly and the fractographical morphology changes significantly. The effects of the crystalline phases on the deformation and fracture behaviors of the glassy alloys have been discussed.

4:40 PM
Structural Changes during Deformation of Zr-Based Metallic Glasses: *Ashwini Bharathula*[1]; Weiqi Luo[1]; Wolfgang Windl[1]; Katharine Flores[1]; [1]Ohio State University

Flow defects in metallic glasses are commonly associated with locally increased free volume. Indeed, positron annihilation measurements reveal a trimodal distribution of open volume in several metallic glasses, suggesting that some regions are more open than others. This distribution shifts with deformation. However, a detailed description of the flow defect structure and operation is lacking. In the present study, the mechanical responses of simulated Zr-Cu and Zr-Cu-Al glasses under tension, compression and shear are investigated using large-scale molecular dynamics simulations. Fluctuations in the electron density distribution are examined to characterize the evolution of low atomic density regions with deformation. The computational results are compared with experimental results for a Zr-based glass. TEM investigations reveal that homogeneous flow in tension results in nanocrystallization, which is not observed during annealing at the same temperature. Evolution of the experimental glass structure with deformation is characterized using DSC, TEM, EELS, and positron annihilation spectroscopy.

4:50 PM Invited
The Oxygen-Induced Degradation of Cu- and Zr-Based Bulk Glassy Alloys: *Wu Kai*[1]; P. C. Kao[1]; P. K. Liaw[2]; [1]National Taiwan Ocean University; [2]Department of Materials Science and Engineering, The University of Tennessee

Cu- and Zr-based bulk metallic glasses (BMGs) have been extensively developed during the past two decades. These BMGs generally possess excellent mechanical properties and good corrosion resistance in various aqueous solutions. However, one important challenge to use them for certain applications is to retain their amorphous structure involved in thermal-activated processes at ambient atmospheres. In this study, the effect of oxygen on the degradation of several BMG systems near the glass transition temperature (Tg) was discussed. In general, the degradation can be catalogued at three different cases, consisting of the first case of pre-oxidation, and then, followed by the substrate crystallization, while a reverse situation was occurred for the second case. In addition, the third case was composed of the simultaneous oxidation and crystallization.

Cast Shop for Aluminum Production: Casting Structure vs. Process

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Pierre Le Brun, Alcan CRV; Hussain Alali, Aluminium Bahrain

Wednesday PM — Room: 2005
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Michel Rappaz, Swiss Federal Institute of Technology

2:00 PM Introductory Comments

2:05 PM Keynote
Recovery vs Structure Driven DCCasting Process Optimisation: *Philippe Jarry*[1]; [1]ALCAN Centre de Recherches de Voreppe

DCC has to be optimised along internal criteria as well as external to the casthouse. Oftentimes parameters have an influence both on the casting recovery and on the metallurgical structure of the slabs or billets, therefore certain heredity in the downstream fabrication schedule. Trade-offs are thus necessary. Distribution systems, casting recipes, grain refinement strategies, etc. will be successively reviewed at the light of both recent academic findings and industrial experience under this dual perspective. Most relevant issues will be pointed out and research avenues for the future will be suggested.

2:35 PM
3D Modeling of the Flow and Heat Transfer during DC Casting with a Combo Bag: Florin Ilinca[1]; Jean-François Hétu[1]; *André Arsenault*[2]; Daniel Larouche[2]; Sylvain Tremblay[3]; [1]National Research Council, Industrial Materials Institute; [2]Laval University; [3]Pyrotek Inc

The goal of this study is to determine the influence of combo bag on the velocity and temperature fields in the liquid metal pool during the DC casting of aluminum ingots. For this, a 3D finite element solution algorithm is used to compute the flow and heat transfer phenomena. The solution approach is able to deal with high Reynolds number turbulent flows, buoyancy effects and flow through combo bag meshed openings. An isothermal study with turbulence modeling quantifies the effect of the combo bag on the flow and an effective viscosity is determined for the respective flow conditions. The coupled flow and heat transfer during ingot formation are solved for forced convection conditions (no buoyancy) and by including the natural convection terms. It will be shown that the flow is driven by the inlet flow rate in the vicinity of the combo bag and by natural convection outside this region.

2:55 PM
Advanced CFD Modeling of DC Casting of Aluminum Alloys: *Mainul Hasan*[1]; Kamal Ramadan-Ragel[1]; [1]McGill University

A 3-D CFD model for the simulation of vertical direct chill (DC) slab casting of aluminum alloys is developed. The basis of the model is the 3D time-averaged turbulent transport equations. An in-house developed CFD code is used to solve the modeled equations. The model is qualitatively and quantitatively verified by comparing the computed results with a physical water model and a real casting experiment of independent researchers. Each of the comparisons showed a good agreement. A parametric study has been carried out for a DC slab caster of aspect ratio 2.0 fitted with a combo bag for melt distribution from the nozzle. The primary and the secondary cooling zones are simulated by changing the heat transfer coefficient on the slab surface. An in-depth understanding is gained of some behaviors of the melt flow and solidification profile in the steady state operational phase of the commercial DC casting process.

3:15 PM
Mathematical Modeling of DC Cast Sheet Ingots Using a Semi-Solid Tensile Constitutive Behaviour for Hot Tearing Prediction: *Daniel Larouche*[1]; Dung-Hanh Nguyen[1]; Steven Cockcroft[2]; André Larouche[3]; [1]Laval University; [2]University of British Columbia; [3]Rio Tinto Alcan

Hot tearing occurs generally during the last stages of solidification, especially near the solidus where the fraction solid is very high and the metal is under

tensile loading. When the deformation is too high for the strength of the semi-solid microstructure, rupture occurs and a defect appears if the liquid metal cannot feed the void created by the rupture. A stress-strain based theory has been developed previously and was applied based on tensile curves obtained with a direct chill surface simulator. The constitutive model built from the results represents the tensile behaviour of the alloy in the mushy zone. This constitutive behaviour was implemented in a 3D thermal-stress model of the direct chill casting process created within ABAQUS. Stresses calculated in zones where hot tearing occurs in DC casting were found to be close to the failure stresses found when the fraction solid lies in the interval 0.9 - 0.95.

3:35 PM

A Comparison of Hot Tear Testing and Hot Tensile Testing of Al – Cu Alloys: *David Viano*[1]; Mary Wells[2]; David StJohn[3]; [1]CSIRO; [2]University of Waterloo; [3]CAST

The determination of mechanical property data in the semi-solid region is vital for developing hot tearing numerical models. The mechanical properties are measured either during cooling from the fully liquid state or during reheating from the fully solid state. An experimental program was conducted comparing the two techniques on AA196, Al – 0.5wt%Cu and Al – 2wt%Cu alloys. Hot tear experiments were conducted using the CAST Hot Tear rig which measures load development during solidification. The same alloys were tested in an apparatus developed to test reheated alloys at temperatures above the solidus. A model was used to estimate the effect of solid state diffusion during the reheating test. Load development from the hot tear test and maximum stress from hot tensile tests were compared as a function of fraction solid. The results of this comparison and challenges faced in conducting these types of tests are discussed in this paper.

3:55 PM Break

4:15 PM

Influence of Iron and Manganese on Structure and Microporosity of the DC Cast AA5083 Alloy: *Carmen Stanica*[1]; Petru Moldovan[2]; Gheorghe Dobra[1]; Cristian Stanescu[1]; Dionezie Bojin[2]; [1]ALRO; [2]Politechnic University Bucharest

The AA5083 alloy is one of the most common alloys in the AA5XXX series wrought aluminum alloys. The effect of iron and manganese content intermetallics on the microstructure and microporosity has been studied by optical microscopy (OM), scanning electron microscopy (SEM) and energy – dispersive X-ray analysis (EDS). It was clear underlined that micropores formation is well connected with iron and manganese intermetallics, which cause blockage in the interdendritic channels which can hinder feeding and hence promote porosity.

4:35 PM

Influence of Ultrasonic Melt Treatment on Structure Formation in Aluminum Alloys with High Amount of Transition Metals: Tetyana Atamanenko[1]; Dmitry Eskin[2]; *Laurens Katgerman*[1]; [1]TU Delft; [2]Materials Innovation Institute

In the casting of aluminum, ultrasonics can be used to promote the formation of a fine, uniform, non-dendritic grain structure. From previous investigations it is known that main condition for obtaining fine equiaxed grain structure is a combined action of grain refiners and an intense ultrasound inducing developed cavitation on a solidifying melt. Additions of transition metals (Zr, Ti etc.) can significantly increase the number of nucleation sites in the melt without using an Al-Ti-B master alloy. Many commercial wrought aluminum alloys contain these elements because they also prevent recrystallization. The paper describes the results on the influence of zirconium and titanium in hypo- and hyperperitectic concentrations on structure formation during ultrasonic melt treatment (UST) in liquid state. In separate experiments, model binary Al-Zr and Al-Ti alloys are solidified with and without UST. The final microstructure is analyzed in terms of grain size and formation of intermetallics.

4:55 PM

Ultrasonic Treatment of a Solidifying Al-Cu Melt in the Presence of Micron-Sized Hydrogen Bubbles: *Mainul Hasan*[1]; Ali-Reza Naji-Meidani[1]; [1]McGill University

The possibility of dynamic grain refinement of aluminum alloys using high-powered ultrasonic waves in the presence of small hydrogen bubbles in the melt is explored. In this regard, a mathematical model is developed to simulate the dynamic behavior of a hydrogen bubble present in the mushy region of a solidifying aluminium-3.4 wt pct copper alloy melt under various applied ultrasonic pressure fields. Due to violent collapse of a small gas bubble, the melt pressure surrounding the bubble increases very rapidly. If the pressure in the vicinity of the dendrites exceeds a threshold value, dendrite fracturing can take place. Dendrite fragments then can act as nuclei during metal crystallization process. This can lead to refined crystalline structure of the metal. This study demonstrates that even far from the bubble's surface, the melt pressure can be sufficiently high to fracture the dendrite arms and produce nuclei for equiaxed crystal growth.

5:15 PM

Silicon Crystal Formation during DC Casting of Aluminium-Silicon Alloys: *Torbjorn Carlberg*[1]; [1]Mid Sweden University

During brazing of automotive heat exchangers, aluminium alloys containing 7-12% silicon is used as filling material. Although these alloys are hypoeutectic, polyhedral silicon crystals, of a type similar to primary precipitation in hypereutectic alloys, can form before to the growth of the eutectic silicon. This occurs during casting of the alloys, which is done by the DC casting process. If these crystals are too large they can cause problems during the brazing. The polyhedral silicon crystal formation has been studied both in industrial ingots and in simulation experiments in a Bridgman furnace. It was found that the nucleation temperature and the cooling rate were important factors influencing the amount and size of the polyhedral crystals. Modelling of diffusion controlled growth of the silicon phase in this type of aluminium-silicon alloys shows how the nucleation temperature influences the morphology of the precipitated silicon, and how large crystals can be avoided.

5:35 PM

Effect of Application of out-Phase Electromagnetic Field on Horizontal Direct Chill Casting of 7075 Aluminum Alloy: *Qingfeng Zhu*[1]; Zhihao Zhao[1]; Jianzhong Cui[1]; Yubo Zuo[1]; [1]Key Laboratory of Electromagnetic Processing of Materials, Ministry of Education, Northeastern University

The effect of application of the out-phase electromagnetic field in HDC on the process and the metallurgical quality of ingots of 7075 alloy was investigated in detail. The results show that when out-phase electromagnetic field was applied, the effect of gravity on the HDC casting process was eliminated effectively, the temperature distribution in the pool become more uniform, cooling difference between upper surface and bottom surface and depth of sump was reduced, the sump shape was changed to be more symmetric about geometrical center of the mold, the thickness of segregation layer decreased and the surface quantity and the microstructures of the ingots were improved, the area of feathery grains decreased and the area of equiaxed grains increased, the equiaxed grains were refined and the floating grains eliminated so that the quality of the ingots was improved.

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

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

Program Organizers: Toru Okabe, University of Tokyo; Ann Hagni, Geoscience Consultant; Sergio Monteiro, State University of the Northern Rio de Janeiro - UENF

Wednesday PM — Room: 3009
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Dafei Kang, Michigan State University; Jeongguk Kim, Korea Railroad Research Institute

2:00 PM

Structural Investigations of the Orientation Patterning in Plastically Deformed Single Crystals: *Olga Dmitrieva*[1]; Dierk Raabe[1]; [1]Max-Planck-Institute for Iron Research

In this contribution we present the investigations of the orientation patterning on single crystals after a plastic shear deformation. The digital image correlation applied during the shear experiments allowed determining both the macroscopic deformation and the local strain distribution during the plastic deformation of the sample. The microstructure of the deformed crystals was investigated by high resolution electron backscattered diffraction (EBSD). By comparing the strain data to microstructure, orientation patterning effects such as the formation

of microbands can be correlated to the local rotation and shearing of the sample. The orientation pattern observed in high resolution EBSD experiments revealed local lattice rotations up to 3° within the microbands. The spatial distribution of the microbands was investigated using the combination of a focused ion beam with a high resolution EBSD analysis. This work was supported by the Deutsche Forschungsgemeinschaft.

2:15 PM

Quantification of the Mineral Phases in Sintered Ores from Gray Historgram of Micrograph: *Xuewei Lv*[1]; Lifeng Zhang[1]; Shaojiang Deng[2]; Chenguang Bai[2]; [1]Missouri University of Science and Technology; [2]Chongqing University

An intelligent recognition and quantification system for the micrographs sintered ores was developed in the current paper. The average distribution parameters in a gray histogram of minerals were obtained using the Gaussian gray distribution model. The feature curves of the gray histogram of two minerals were achieved by combining the two density functions. The feature indexes, such as the number and position of the peaks and the valleys of two minerals in different ratios, were quantified by differentiating the distribution functions. This algorithm is able to extract the feature of the minerals adequately and accurately for recognizing the composition and phases of the minerals intelligently.

2:30 PM

Green Rust: Its Electrochemical Generation, Characterization, and Implications: *Jewel Gomes*[1]; David Cocke[1]; Hector Moreno[2]; Doanh Tran[1]; Appel Mahmud[1]; Kamol Das[1]; Mallikarjuna Guttula[1]; [1]Lamar University; [2]Instituto Tecnologico de la Laguna

Green rust is an important intermediate in oxidative transformation of Fe(II) phase. This unstable compound contains a mixture of ferrous and ferric hydroxides that belong to a family of minerals known as layered double hydroxides (LDH). This brucite-type LDH contains anions such as chloride, carbonate and sulfate, and also water molecules filling the interlayers. In addition, either the bivalent or the trivalent iron can be replaced by other trivalent or bivalent metal ions. It was first identified as corrosion product, later in soils as a product of interactions between microbes and metals in soils. Due to its high reactivity, it is used in the reduction of organic and inorganic compounds, As removal, and the treatment of acid mine drainage. In this paper, we described the method of electrochemical generation of green rust and its characterization by XRD, SEM and FT-IR. We also illustrated its implications to electrocoagulation.

2:45 PM

Antimicrobial Property of Copper Stamp Sand: *Jiann-Yang Hwang*[1]; Domenic Popko[2]; Bowen Li[1]; Jaroslaw Drelich[1]; Susan Bagley[1]; [1]Michigan Technological University; [2]Lesktech Ltd.

Mining of native copper was active in the Upper Peninsula of Michigan in the last century. Stamp sand is the mining waste left behind after separating copper from the crushed rock fragments. It is estimated that there are about 500 million tons of stamp sands left in the area. Since the separation of copper was not complete in the early days, there are copper left in the stamp sand. Copper is an element that has the antimicrobial property. Therefore, a study to determine if the stamp sand has the antimicrobial property was carried out and the results are reported.

3:00 PM

Numerical Modeling of Liquid Flow Permeability on 3D Microtomographic Geometry of Al-Cu Alloys: *Ehsan Khajeh*[1]; Daan Maijer[1]; [1]The University of British Columbia

Modeling the formation of defects related to the flow of interdendritic liquid, requires a good knowledge of the way in which permeability changes with local geometry of interdendritic channels. In this study, the permeability of Al-15.5wt%Cu and Al-19.5wt%Cu has been modeled by solving the full Navier-Stokes equations on real 3D geometries of interdendritic channels obtained by X-ray microtomography (XMT). The samples for XMT were obtained from different positions in directionally solidified cylinders in order to produce different microstructure for each composition. The flow has been solved using a 2nd-order accurate Finite Volume Method (FVM) approach. Marching-cube triangulation method was necessary to produce an accurate surface and unstructured volume meshes. Calculated values of permeability for this range of solid fraction show partial agreement with previous experiments and the Carman-Kozeny expression for flow through granular beds. Observed deviations are analyzed and attributed to the experimental difficulties of permeability measurements.

3:15 PM Break

3:35 PM

Mechanical Behavior of Polyester Composites Reinforced with Alkali Treated Coir Fibers: *Sergio Monteiro*[1]; Hélvio Santafé Jr.[1]; Lucas da Costa[1]; [1]State University of the Northern Rio de Janeiro - UENF

Polymeric matrix composites reinforced with coir fibers are being used in many industrial applications such as automobile interior panels and cushions. These composites have relatively low mechanical strength due to the weak fiber/matrix interface. The objective of the present work was to investigate the effect of a surface alkali treatment of the curaua fiber on the mechanical behavior of polyester composites with different amounts of these fibers. The treatment, also known as mercerization, was performed with distinct concentrations, 0.1 and 10% of NaOH at 1 min and 1 hour, before incorporation of the coir fibers into the polyester composite. The results showed no significant change in comparison to similar untreated coir fiber composites. A microstructure analysis revealed that, in addition to reducing the hydrophilic characteristic of the fiber,the treatment also affects the surface morphology and impairs the fiber/matrix interfacial strength.

3:50 PM

Characterization of the Mechanical Behavior of Epoxy Matrix Composites Reinforced with Ramie Fibers: *Sergio Monteiro*[1]; Frederico Margem[1]; Luiz Fernando dos Santos Jr.[1]; [1]State University of the Northern Rio de Janeiro - UENF

Fibers extracted from the ramie plant have been traditionally used inn textile and are now being considered as polymeric composite reinforcement owing to their superior strength. Recently the mechanical behavior of ramie fiber reinforced polyester composites was investigated but no significant improvement was found. Therefore, the objective of this work was to carry out a similar investigation by changing the type of polymeric matrix. Specimens with up to 30% in volume of ramie fibers incorporated into epoxy were bend tested until fracture. The results showed a moderate improvement with a tendency of decreasing stress for higher amounts of ramie fibers due to the weak interface developed with the epoxy matrix.

4:05 PM

Characterization of Clays from Campos Dos Goytacazes, State of Rio De Janeiro, Brazil: *Carlos Maurício Vieira*[1]; Sergio Monteiro[1]; [1]State University of the Northern Fluminense

The main characteristics and physical and mechanical properties of clays from the county of Campos dos Goytacazes, located at the northern part of the State of Rio de Janeiro, Brazil, are presented. The characterization included mineralogical composition, chemical composition and particle size distribution. The properties related to water absorption, linear shrinkage and flexural rupture strength were obtained in samples that were prepared by 20 MPa uniaxial pressing and fired at 950, 1100 and 1250°C. The results showed that the clays are predominantly kaolinitic with high percentage of clay minerals. The kaolinitic structure associated with a small amount of fluxes confers a refractory behavior which makes difficult the sinterization and impairs the ceramic consolidation.

4:20 PM

Effect of the Particle Size of Incorporated Grog on the Properties and Microstructure of Clayey Bricks: *Carlos Maurício Vieira*[1]; Sergio Monteiro[1]; [1]State University of the Northern Fluminense

The brick industry generates a significant amount of residues composed of broken pieces that can be recycled, after crushing, as a particulate type of waste called grog. In the present work a grog screened at two different particle size, 840 and 420 μm, was reintroduced in the process in mixtures with clays to make bricks. The effect of the grog addition up to 20 wt.% on the properties and microstructure of bricks fired at 700°C was evaluated. The results indicated that both the particle size and the amount of grog addition changed the fired properties of the clayey body. Additions above 5 wt.% of grog, with the coarser particle size, decreased the mechanical strength of both the dry body and the fired ceramic pieces. By contrast, the finer particle size grog may be used up to 10% wt. without impairing the properties and corresponding microstructure of the clayey body.

4:35 PM
Characterization of the Critical Length of Sisal Fibers for Polyester Composite Reinforcement: *Sergio Monteiro*[1]; Wellington Inácio[1]; Felipe Perissé Lopes[1]; Lucas da Costa[1]; Luiz Fernando dos Santos Jr.[1]; [1]State University of the Northern Rio de Janeiro - UENF

From the leaves of the sisal plant (Agave sisilana) a relatively strong fiber can be extracted and is nowadays being investigated as a possible reinforcement for polymeric matrix composites. For this purpose, it is important to determine the critical length of the fiber with respect to the matrix to be reinforced. This has never been properly done for sisal fibers in polyester matrix. Therefore, this work characterized the critical length of sisal fibers, with different sizes, embedded in polyester capsules by means of pullout tests. The critical length obtained allowed the sisal fiber/polyester matrix interface shear stress to be calculated. The relevance of these values for polyester composites reinforcement with sisal fibers is discussed.

Computational Thermodynamics and Kinetics: Thermodynamics

Sponsored by: The Minerals, Metals and Materials Society, ASM International, 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
Program Organizers: Long Qing Chen, Pennsylvania State University; Yunzhi Wang, Ohio State University; Pascal Bellon, University of Illinois at Urbana-Champaign; Yongmei Jin, Texas A&M

Wednesday PM Room: 3002
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Raymundo Arroyave, Texas A & M University

2:00 PM Invited
Massively Parallel Architectures and Alloy Theory: *Axel Van De Walle*[1]; [1]California Institute of Technology

Numerous materials problems demand large-scale computational resources and Alloy theory problems are no exception. This talk discusses two specific computationally-intensive problems and their solutions: (i) the calculation of multicomponent alloy phase diagrams and (ii) the automated determination of structure-property relationships in crystals. The proposed methods rely on input from first principles electronic structure calculations and employ the so-called cluster expansion formalism (which, in some applications, must be generalized to handle tensors). The key insight is the realization that these problems can be cast into the form of a large number of smaller-scale weakly coupled ab initio calculations. To ensure efficiency, these tasks need to be dynamically created based upon earlier calculation output, thus requiring flexible scheduling schemes that are nevertheless compatible with existing queuing systems. We explain how these calculations can be efficiently carried out on shared large-scale supercomputers, such as the ones available via the Teragrid infrastructure.

2:30 PM
Thermodynamic Description of the System Cu-Sn-P - Computational Thermodynamics and Experimental Investigation on the Systems CuSn and CuP: *Monika Grasser*[1]; Florian Mayer[1]; Andreas Ludwig[1]; Johann Riedle[2]; Udo Hofmann[2]; [1]Montanuniversity of Leoben; [2]Wieland-Werke AG

Technical bronzes are based on Cu-Sn-P alloys. Sn and P tend to from microsegregations and macrosegregations during DC-casting why the involved phase diagrams are of great interest for industry. The paper shows experimental investigations with Differential Scanning Calorimetrie measurements for the binary Cu-Sn and Cu-P system and diffusion experiments. Besides, numerical calculations have been performed. The DSC measurements allow the identification of phase transformation temperatures. For the identification of the phase distribution, SEM investigations are applied. Most of the phases were detected. Diffusion experiments have been performed for two binary diffusion partners, namely CuSn20 and CuP8.3, with a cylindrical geometry. Here information has been gained about diffusion coefficients of Sn and P under specific conditions. Besides, numerical calculations have been performed with ThermoCalc based on a user defined database. As a final step already published phase diagram data, resent experimental data, and numerical assessment work are compared.

2:50 PM
First Principles Study of the Thermodynamics of Carbynes: W. Luo[1]; *Wolfgang Windl*[1]; [1]OSU

Many years ago, a third solid carbon allotrope has been suggested to exist besides diamond and graphite called carbyne, consisting of linear carbon chains. Carbynes were suggested to form at high temperatures under pressure and have been suggested to be detrimental to the high-temperature performance of carbon materials. Even today, their existence is still under discussion, and no atomic structure for pure-carbon carbyne has been resolved. In this paper, we propose a structural model for carbynes, combining elements from previous work. Structural optimization within density functional theory shows that the crystallography of these structures agrees well with previous experimental results. The free energy of the proposed carbyne structures is calculated within wide ranges of temperature and pressure (1000 to 4000 K and 0 to 180 kbar) and is higher than that of graphite in the whole studied region. However, our calculations confirm that additional elements can stabilize carebynes.

3:10 PM
Graphical Representation for Isothermal Kinetics of Non-Equilibrium Grain-Boundary Segregation and Its Applications: *Tingdong Xu*[1]; [1]Central Iron and Steel Research Institute

A recent model for non-equilibrium solute segregation at the grain-boundary is expressed with graphs for the isothermal ageing at various temperatures after quenching from a solution temperature. Some new characteristics are found using the graphical representations. For the samples aged for a certain time at various temperatures, an ageing temperature exists at which solute concentration at grain boundaries reaches a maximum value and the critical time of the non-equilibrium segregation at the ageing temperature will be equal or close to the ageing time. These findings have important consequences for the analysis of grain boundary segregation during the common thermal cycles. A number of diverse and sometimes conflicting experimental results from a number of different labs are rationalized on the graphical representation. As the application of these new characteristics, a non-equilibrium grain-boundary segregation mechanism for intermediate temperature brittleness of metals and alloys is proposed.

3:30 PM
Thermodynamic Assessment of Ce-Cr, Cr-La and Cr-Y Systems: *Wren Chan*[1]; Michael Gao[2]; Omer Dogan[2]; Paul King[2]; [1]Carnegie Mellon University; [2]National Energy Technology Laboratory

In order to improve ductility and creep resistance, early rare earth elements are added to refractory metal based alloys to absorb residual oxygen in the alloy and form dispersion strengthening oxides. In this work, three binary systems, Ce-Cr, Cr-La and Cr-Y, were thermodynamically assessed based on available experimental data in the literature. The PARROT module of Thermo-Calc package was used to optimize the systems. Self-consistent and reasonable thermodynamic descriptions for all three systems were obtained.

3:50 PM Break

4:15 PM

Thermodynamic Calculations Predicting MgO Saturated EAF Slag for Use in EAF Steel Production: *Kyeising Kwong*[1]; James Bennett[1]; Rick Krabbe[1]; Arthur Petty[1]; Hugh Thomas[1]; [1]NETL, USDOE

The use of foamy slags is widely practiced in EAF steel production resulting in energy savings, productivity improvements, and enhanced refractory service life. Foamy slag requires the control of slag viscosity to sustain gas bubbles during processing. This is accomplished through the precipitation of magnesium wustite particles in the slag at the operating temperature. A thermodynamic program, Factsage®, was utilized to study the quaternary oxide system of MgO-CaO-FeO-SiO_2, to predict the dual and MgO saturated EAF slag chemistry under different oxygen partial pressures, temperatures, and slag basicity. These predictions indicate a linear relationship between oxide components and slag basicity, from which an accurate prediction of the optimum slag chemistry can be made. The results of the Factsage® calculations will be compared to models developed by other researchers and experimental data. Also discussed will be how these results can be used from practical slag management during EAF steel production.

4:35 PM

Thermodynamic Model as Double Function of Temperature and Molar Fraction for Aluminum-Tin Alloys: *Cristian-Aurelian Popescu*[1]; Dragos Taloi[1]; Liana Vladutiu[1]; [1]University POLITEHNICA Bucharest

In this paper, a new thermodynamic model developed as double function of temperature and molar fraction for characterization of thermodynamic behavior of aluminum-tin alloys is presented. The model offers the advantage of computing thermodynamic functions of the aluminum-tin alloys in every composition and temperature desired. The proposed model is a polynomial model having sixteen interactions parameters which were determined using the least squares method based on three sets of data: own experimental results, equilibrium diagram and literature data. The experimental data were obtained in our laboratory using electrochemical measurements of activity. Using the model developed the molar thermodynamic functions and partial molar functions were computed. The comparison of the results computed using the model and the experimental and literature data have shown a very good correlation.

4:55 PM

Estimation of Formation Enthalpies Using an Extended Miedema Approach: *Pratik Ray*[1]; Mufit Akinc[1]; Matthew Kramer[2]; [1]Iowa State University; [2]Ames Laboratory

Formation enthalpy , which is an indicator of phase stability, is an important parameter in materials research. A number of methods can be used to calculate enthalpy. Ab-initio calculations can often give highly precise results, but are time intensive. Models based on solution thermodynamics, like CALPHAD, are based on expressing the thermodynamic variables as a polynomial function of temperature. these approaches are fast; but they require a prior extensive database. Semi-empirical models like Miedema's method can be used for extremely fast calculation of enthalpies in situations where a large number of alloys have to be considered in absence of a prior thermodynamic database. While the original Miedema's model was postulated for binary alloys, in this paper we attempt to extend it to ternary systems and use it for estimating formation enthalpies of a large number of Ni based ternary alloys.

5:15 PM

Thermodynamic Calculation of CaSO4-Ca(OH)2-H2O System Phase Equilibriums: Xiaoyu Peng[1]; *Yunyan Wang*[1]; [1]Central South University

Pitzer theory was applied to calculate solubility in the ternary brine system of CaSO4-Ca(OH)2-H2O, and the phase diagram has been drawn at 298.15K. This diagram consists of one single-phase region and three two-phase regions. The single-phase region is the unsaturated solution zone, and three two-phase regions include the crystallization zone of CaSO4•2H2O, the crystallization zone of Ca(OH)2 and the coexist zone of CaSO4•2H2O and Ca(OH)2. The interaction characteristics between the solubility of CaSO4(s) and Ca(OH)2(s) was also obtained. The results would provide a theoretical basis for the treatment and reuse of industrial wastewater, especially for the wastewater containing sulfate which would be treated by lime-milk neutralization.

5:35 PM

Computing the Phase Diagram of the FeCr Binary Alloy by Path-Sampling Techniques: *Gilles Adjanor*[1]; Manuel Athènes[2]; [1]EDF R&D; [2]CEA

Due to their potential application as structural material for fusion and Generation IV reactors, high-chromium ferritic/martensitic steels have recently received considerable interest. Ab initio results have shown that due to magnetism the sign of the mixing enthalpy of the Fe-Cr system changes at low temperature. One of the most challenging tasks is now to establish how this effect influences the phase diagram of the system. In a recent study, Monte-Carlo simulations in the semi-grand canonical ensemble and the thermodynamic integration method were applied, with their respective limitations. We report on the first attempt to apply a path-sampling method on this system. This method can be seen as a combination of the former methods in the limiting cases of fast and slow switching rates respectively. In addition, the analysis of path histograms yields a built-in criterion for diagnosing the convergence of thermodynamic potential estimates.

Diffusion in Materials for Energy Technologies: Session IV

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS: Alloy Phases Committee, TMS: High Temperature Alloys Committee, TMS/ASM: Nuclear Materials Committee, TMS: Solidification Committee, ASM-MSCTS: Atomic Transport Committee
Program Organizers: Jeffrey LaCombe, University of Nevada, Reno; Yongho Sohn, University of Central Florida; Carelyn Campbell, National Institute of Standards & Tech; Afina Lupulescu, GE; Ji-Cheng Zhao, Ohio State University

Wednesday PM — Room: 3006
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Carelyn Campbell, National Institute of Standards and Technology; Jeffrey LaCombe, University of Nevada, Reno

2:00 PM Invited

Determination of the Diffusivity for Point Defects in Passivation Layer on NiTi and NiTiAl Thin Films: K. T. Liu[1]; *Jeng-Gong Duh*[1]; [1]National Tsing Hua University

The shape memory NiTi and NiTiAl alloys are widely used in the biomedical application. In the electrolyte, the passivation layer is formed on the NiTi and NiTiAl. A Point Defect Model (PDM) based on the movement of cation and anion defects in an electrostatic field was carried out to explain the growth or dissolution behavior of a passivation layer on a NiTi and NiTiAl thin films. The potential drop was related to the barrier film/electrolyte interface on the applied voltage and to estimate the diffusivity of defect. The calculated value of diffusivity was in range of 10^{-16} – 10^{-17} cm^2/s. This value was extracted from the potential relationship between the donor density and the film formation voltage. Besides, the Mott-Schottky (M-S) analysis also indicated that the movement of the major defect was contributed by the oxygen vacancy. The defect of oxygen vacancy revealed that the passivation film was an n-type semiconductor. Analysis has shown that the doping level within a passive film was rather large and in the order of 10^{20} - 10^{21} cm^{-3} film. In the electrochemical reaction, the lower donor density and the lower diffusion coefficient retarded the movement of defect in the passivation layer and improved the stability of the passive film during corrosion.

2:30 PM

Kinetic Monte Carlo Formation of Hollow Nanospheres Using the Kirkendall Effect: Alexander Evteev[1]; Elena Levchenko[1]; Irina Belova[1]; *Graeme Murch*[1]; [1]University of Newcastle

The experimental formation in 2004 of hollow nanospheres using the Kirkendall effect has attracted a great deal of interest because of the possible applications of these structures in a very wide range of technologies. An in-depth theoretical understanding of the phenomenon is lacking. Results of kinetic Monte Carlo simulations of the formation of a hollow nanosphere by interdiffusion from a core-shell binary system are presented. The faster diffusing species is located in the core whilst the slower diffusing species forms the shell. With its self-generated vacancies all stages of the hollow sphere formation process are observed in this model: interdiffusion, the supersaturation of the core of the

nanosphere by vacancies, precipitation of pores and eventual void formation. Results confirm the experimental conclusions that interdiffusion accompanied by the Kirkendall effect and Kirkendall porosity is one of the mechanisms responsible for the formation of hollow nano-objects.

2:50 PM

Molecular Dynamics Study of Surface Segregation in Bimetallic Ag-Ni Core-Shell Nanoparticles: Alexander Evteev[1]; Elena Levchenko[1]; Irina Belova[1]; *Graeme Murch*[1]; [1]University of Newcastle

Bimetallic nanoparticles have received considerable attention for their importance in catalysis and nanotechnology. Much of the research has focussed on binary metal systems that form ordered or random bulk alloys. But in another type of binary metal system such as Ag-Ni the two metal components do not mix appreciably in the bulk. Ag-Ni nanoparticles are expected to possess the surface character of Ag and the magnetic properties of Ni and have applications in many fields. At the nano-level, in addition to the tendency for phase separation, surface segregation of Ag is expected since it has a lower surface energy. Molecular dynamics is used to investigate the effect of surface segregation on the structure and atomic distribution of an initial Ag-Ni core-shell nanoparticle. It is found that Ag atoms diffuse through the Ni shell until they have completely covered the outer surface of this nanoparticle with a well-defined surface monolayer.

3:10 PM

Ab Initio Study of Reordering in Microtwinning Deformation Mechanism in Ni Base Superalloys: *Libor Kovarik*[1]; Ju Li[2]; Raymond Unocic[1]; Michael Mills[1]; [1]Ohio State University; [2]University of Pennsylvania

Microtwinning is an important deformation mechanism at intermediate temperature, low stress and low strain rate conditions in Ni base superalloys. The rate limiting process of the microtwining deformation mechanism is the diffusion-controlled reordering in γ' phase. It is shown that reordering requires very simple, vacancy-mediated exchange between Al and Ni atoms. The energy barriers for the different pathways that lead to vacancy-mediated exchanges have been studied using ab initio calculations. Based on the results it is possible to predict the most favorable reordering pathways. The current results indicate that the diffusion coefficient for reordering should be similar to that for Ni self-diffusion considering an ideal Ni3Al system. The currently investigated alloy Rene 104 is a multi-elemental system. It will be discussed that other factors such as segregation of heavy elements at the twin interface may have a significant influence on the kinetics of reordering.

3:35 PM Break

3:50 PM Invited

Interdiffusion Coefficients Extraction from Multicomponent Diffusion Couple Data: *Liang Jiang*[1]; Shamik Chaudhuri[2]; Jack Madelone[1]; [1]GE Global Research; [2]GE Bangalore Engineering Center

The behavior of multicomponent diffusion can be complex due to the inter-dependence of diffusion among multiple elements. Interdiffusion coefficients are critical to characterize diffusion in multicomponent systems. Multicomponent diffusion couples are typically used to extract the interdiffusion coefficient. In the present study, various interdiffusion coefficients determination procedures are reviewed and a numerical inverse method is proposed. The numerical inverse method along with others is utilized to extract interdiffusion coefficients from experimental data of multicomponent diffusion couples. In this approach we are using an analytical diffusion solver to predict diffusion profiles and try to minimize the difference of the predicted profile and the experimental profile to estimate diffusion coefficients.

4:20 PM

Accounting for Transient Ostwald Ripening in Creep Models of Multi-Modal Nickel-Base Superalloys: *James Coakley*[1]; Hector Basoalto[2]; David Dye[1]; [1]Imperial College; [2]QinetiQ

Traditionally aero and industrial gas-turbine disc materials have been developed to ensure high resistance to fatigue cracking and propagation, as well as high yield strength, tensile strength, ductility and fracture toughness. Creep resistance has been given less emphasis due to the lower operating temperatures, but as operating temperatures increase over the years, disc components are being pushed into regimes where significant creep can occur. However, at these temperatures the microstructure may not be stable; significant diffusion-controlled coarsening of the, potentially multi-modal, precipitate distribution often occurs. An expansion of the Dyson microstructure–based creep model is presented in this talk that accounts for these effects using a modified LSW model to account for the evolution of the precipitate distribution. Creep data and microscopy of the precipitate distribution obtained for the multi-modal nickel superalloy Nimonic 115 is compared to model predictions.

4:45 PM

Thermal Stability of Ta-Based Diffusion Barriers: Julien Nazon[1]; Bernard Fraisse[1]; Marie-Hélène Berger[2]; Jean-Claude Tedenac[1]; *Nicole Fréty*[3]; [1]Institut Charles Gerhardt - Université Montpellier II; [2]Centre des Matériaux P.M. Fourt - Ecole des Mines de Paris; [3]Institut Charles Gerhardt - Université Montpellier II

There has been recently an increasing interest in the development of diffusion barriers against electrical wiring copper used in the thermoelectric and microelectronic device technologies. In this context, the barrier efficiency of TaN(50 nm)/Ta(50 nm)/TaN(50 nm) multilayers against copper diffusion has been investigated and was compared with that of TaN(150 nm) single layers. Tantalum-based and copper thin layers were successively deposited using the sputtering process. The thermal stability of these Ta-based thin layers was experimentally studied from high temperature in-situ Glancing Angle X-ray Diffraction experiments, which were conducted in the temperature range of 773 to 973 K. The diffusion coefficient of Cu through the TaN single layer was calculated from these experiments. These analyses were associated to a microstructural characterization using Scanning and Transmission Electron Microscopies. The TaN/Ta/TaN barrier appeared to be more efficient in preventing Cu diffusion than the TaN single layer.

5:10 PM

Site Preference and Diffusion in Ni3Al Alloyed with Ir, Ta or Re at 1200°C: *Narayana Garimella*[1]; Yongho Sohn[1]; [1]University of Central Florida

Diffusion in L12-Ni3Al with ternary alloying additions of Ir, Ta and Re was investigated at 1200°C using solid-to-solid diffusion couples, and examined with respect to site preference in ordered intermetallic compound. Average effective interdiffusion coefficients were determined directly from the experimental concentration profiles. Ni has the largest magnitude of average effective interdiffusion coefficient, followed by Al, Ir, Re and Ta. The average effective interdiffusion coefficients for Ir, Re and Ta are much smaller than those for Ni and Al. Tracer diffusion coefficients determined by extrapolation technique, and available literature also followed the same trend. The relative tendency of Ni, Al, Ir, Re and Ta to occupy the a-Ni and ß-Al sites are correlated to these diffusion coefficients, with due consideration for diffusion mechanisms as well as the size and coordination of atoms.

5:35 PM

Isothermal Oxidation of γ (fcc) Ni-Cr-X and γ (fcc) Fe-Ni-Cr-X (X = Al, Si, Ge or Pd) Alloys at 800°C: *Narayana Garimella*[1]; Michael Brady[2]; Yongho Sohn[1]; [1]University of Central Florida; [2]Oak Ridge National Lab

Isothermal oxidation of several γ (fcc) Ni-Cr-X and Fe-Ni-Cr-X (X = Al, Si, Ge or Pd) alloys was examined to assess the influence of alloying additions, Al, Si, Ge or Pd, with respect to ternary and quaternary interdiffusion behavior in these alloys. Alloys having various compositions in Ni-Cr-X and Fe-Ni-Cr-X (X = Al, Si, Ge or Pd) systems were arc-melt, chill-cast, and homogenized for 168 hours at 900°C. Isothermal oxidation was carried out in air at 800°C up to 1,008 hours. Change in specimen weight, microstructure of oxide scale and compositional changes in the alloy underneath the oxide scale were examined by using thermogravimetric analysis, X-ray diffraction, scanning electron microscopy, energy dispersive spectroscopy and electron probe microanalysis. The results from isothermal oxidation are discussed and related to our previous studies on ternary and quaternary interdiffusion studies where the influence of alloying additions were quantitatively reported via multicomponent interdiffusion analysis.

Electrode Technology for Aluminum Production: Electrode Connections and Cathode Studies

Sponsored by: The Minerals, Metals and Materials Society, TMS Light Metals Division, TMS: Aluminum Committee
Program Organizers: Barry Sadler, Net Carbon Consulting Pty Ltd; John Johnson, RUSAL Engineering and Technological Center LLC

Wednesday PM Room: 2003
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Marilou McClung, Century Aluminum Co

2:00 PM Introductory Comments

2:05 PM
FEM Analysis of Voltage Drop in the Anode Connector Assembly: *Hugues Fortin*[1]; Mario Fafard[1]; Nedeltcho Kandev[2]; Patrice Goulet[1]; [1]Laval University; [2]IREQ

During their service life, steel stubs are exposed to an extremely aggressive environment involving thermomechanical stresses and chemical attacks, causing their shape to change. These phenomena affect the thermo-electro-mechanical contact at carbon/cast-iron/steel interfaces and have a significant effect on anode voltage drop. To estimate this voltage drop, a 3D finite element model (FEM) of a whole anode was developed and solved with the in-house code FESh+. Different models of stub shapes representing degradation were compared with a new stub to determine variations in voltage drop. The analyzed parameter was diameter of the steel stubs.

2:30 PM
Electrical Losses in the Stub-Anode Connection: Computer Modeling and Laboratory Characterization: *Nedeltcho Kandev*[1]; Hugues Fortin[1]; [1]Hydro-Quebec

The Joule heat dissipated in the anode connection is an important part of the anode power losses and it should be minimized. In this work, the electrical losses in the anodic connector were investigated through laboratory tests, microscope analysis and simple electrical 3D finite elements model (FEM). The electrical resistivity and the thermal expansion have been characterized for the three constitutive materials (steel, cast iron, carbon) as a function of the temperature. Special attention has been paid to the impact of the phase transition period on the electrical resistivity and the thermal expansion for steel and cast-iron. The interfacial contact resistance between the steel stubs and cast-iron has also been studied in the laboratory as a function of the temperature in the range from ambient to cell operating temperature. Ideas for reduction of anodic losses are suggested in this paper to improve the power efficiency of the aluminium reduction process.

2:55 PM
Challenges in Stub Hole Optimisation of Cast Iron Rodded Anodes: *Daniel Richard*[1]; Patrice Goulet[2]; Olivier Trempe[2]; Marc Dupuis[3]; Mario Fafard[2]; [1]Hatch; [2]Aluminium Research Centre - REGAL; [3]GéniSim Inc.

Reduction of cell voltage through redesign of the stub holes of cast iron rodded anodes is an attractive idea. In practice, stub hole optimisation is not an easy task and in situ trials may yield what seem to be counter-intuitive results. A closer examination reveals a complex behaviour of the steel stub - cast iron - carbon joint. It was shown in previous work to be a non-linear thermal-electrical-mechanical coupled system. Minimisation of the stub-to-carbon voltage drop is a balancing act between contact surface area and electrical contact resistance. To gain insights into the merits of different designs, a finite element demonstration model was built using the in-house code FESh++. In order to take into account the rodding shop productivity, alternative configurations with a constant volume of cast iron were studied. The impact of cast iron mean thickness is also examined. Potential industrial applications are discussed.

3:20 PM
New Solutions for Stub-Anode Connection at Egyptalum: *Adel Nofal*[1]; Mohamed Waly[1]; Mahmoud Agour[2]; Shaher Mohamed[2]; [1]CMRDI; [2]Aluminium Company of Egypt

This work studies the possibility of replacing the high phosphorus cast iron with other cast iron grades for casting of stub-anode connection collars in the aluminum electrolytic cells to avoid the harmful effect of phosphorus on electrical resistivity of cast iron and contamination of primary aluminum. Bench scale measurements on the steel stub/cast iron collars/anode carbon combinations were conducted using different cast iron alloys such as high-and low-phosphorus grey irons and ductile iron with low and high carbon equivalents as well as other alloyed irons. The electrical resistivity and voltage drop values were measured at different temperatures and times. The alloy with the best performance was used in actual production conditions over a complete life cycle of the anode up to 850 °C and the results compared with the current situation.

3:45 PM Break

3:55 PM
Use of Cell Autopsy to Diagnose Potlining Problems: *Richard Jeltsch*[1]; [1]Jeltsch Consulting

The technique of cell autopsy, commonly used to determine the mode of failure of a single reduction cell, can be used to dig more deeply into potlining problems. Identification of such problems in a timely manner is important due to the long time horizon for implementation of the solutions. In this paper examples of problems with lining design, materials, cell construction, cell startup and cell operations found through the cell autopsy technique are described and illustrated.

4:20 PM
Autopsies of Spent Refractory Pot Linings – A Revised View: *Kati Tschöpe*[1]; Christian Schøning[2]; Tor Grande[1]; [1]NTNU; [2]SINTEF

Cathode autopsies performed after pot failures or shutdowns of cells have frequently been used to investigate pot failure and degradation of cathode lining. The sequence of materials observed from the cathode to the non-reacted refractory lining has been assumed to reflect the situation before the pot was taken out of service. Here we demonstrate that this is not necessary the case. Based on annealing experiments, X-ray diffraction and electron microscopy we propose that the thermal gradient in the lining is reversed during cooling and that the physical appearance of the lining reflects a combination of cooling and operation of the cell. The presence of molten phases below the carbon cathode may therefore solidify from the top rather than towards the bottom of the lining. Finally, we show experimental evidence that Na (g) infiltrate the refractory lining and is the main attacking chemical specie in the reaction front.

4:45 PM
Energy Recovery and Amperage Increase in Aluminium Cells by Active Cooling of the Anode Yokes: *Asbjørn Solheim*[1]; Bjørn Moxnes[2]; Kristin Vamraak[2]; Elin Haugland[2]; [1]SINTEF; [2]Hydro Aluminium

The anode stubs and yoke assembly acts as an important heat sink in aluminium electrolysis cells. Removing heat by cooling the yokes gives increased heat flow out of the cell. Experiments in an industrial cell indicated that 2.5-3.0 kW could be removed from each anode by using compressed air supplied to channels at the outside of the yoke, and there were positive correlations between air flow, amount of heat collected, and heat loss through the stubs. By covering the yoke with thermal insulation, 4.3 kW was collected at a temperature of 320°C, which is interesting with a view to electricity production from waste heat. Besides the inherent potential for amperage increase and energy recovery, a number of benefits can be anticipated, such as improved cell control (new manipulated variable), lower exhaust gas temperature, and improved electrical conductivity of the steel parts of the anode assembly due to lower temperature.

5:10 PM
Modelling of Collector Bar Sealing in Cathode Blocks with Cast-Iron: *Benedicte Allard*[1]; Serge Lacroix[1]; Jean-Philippe Noyel[2]; Loig Rivoaland[1]; [1]Carbone Savoie; [2]Ecole Catholique des Arts et Métiers

Cast iron rodding is the most spread technique of collector bar sealing in the cathode blocks for the aluminium electrolysis cells. This operation needs to be carefully mastered, either because of all the safety issues involved in the operation, or because of the quality impact of the rodding on the future electrical performance of the cathode blocks in the pots. During cast-iron sealing the cathode blocks are submitted to thermomechanical stresses that can be very important and that may induce block cracking. A model has been developed with ANSYS® software to describe firstly the thermal distribution inside the cathode blocks and secondly the resulting stresses. The influence of different parameters related to the contact between the different products and to the thermal transfert mechanism is studied. First results on the thermal validation of the model and the level of stresses obtained in different types of conditions are also presented.

5:35 PM
Evaluation of Contact Resistance in Electrodes of Hall-Heroult Process: *Laszlo Kiss*[1]; Lyne St-Georges[1]; Mathieu Rouleau[1]; [1]University of Quebec

In the Hall-Héroult cells, the electrodes are made of carbon blocks where steel inserts are sealed with cast iron. The contact resistance between these solids influence the energy efficiency of the process and potentially the life of the cell. An experimental device has been developed where the thermal and electrical contact resistances are determined simultaneously from room temperatures up to 1000ºC under mechanical loads corresponding to those in the real cell. Special attention is paid to the fabrication of the test samples to reproduce realistic cast-iron/carbon and cast-iron/steel interfaces. A procedure representative of industrial cathode and anode sealing is used for the fabrication of the contact surfaces. The conditions of friction (static and dynamic) between the two solids can also be analysed. Using an inverse mathematical method, the evolution of the friction mechanism as a function of the relative displacement between the samples can be followed.

Fatigue: Mechanisms, Theory, Experiments and Industry Practice: Fatigue at High-Temperature and in Harsh Environments

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Computational Materials Science and Engineering Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS/ASM: Nuclear Materials Committee

Program Organizers: Koenraad Janssens, Paul Scherrer Institute; Corbett Battaile, Sandia National Laboratories; Brad Boyce, Sandia National Laboratories; Luke Brewer, Sandia National Laboratories

Wednesday PM — Room: 3008
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Brad Boyce, Sandia National Laboratories

2:00 PM Invited
Low-Cycle Fatigue Properties of Single-Crystal Silicon Films in Harsh Environments: *Pierre-Olivier Theillet*[1]; Olivier Pierron[1]; [1]Georgia Institute of Technology

Understanding the mechanisms for fatigue crack initiation and propagation in micron-scale silicon is of great importance to improve MEMS reliability in harsh environments. Accordingly, this investigation studies the low-cycle fatigue properties of single-crystal Si films using kHz-frequency resonating structures. The influence of resonant frequency (4 vs. 40 kHz) and environment (30C/50%RH vs. 80C/90%RH) on the resulting S-N curves and resonant frequency evolution is monitored. During each fatigue test, consisting of successive bursts of cycles at large stresses, the resonant frequency is precisely measured (0.01Hz resolution) at low stress between bursts. We observe a continuous, monotonic decrease in resonant frequency for each fatigue test. The damage accumulation rates are not significantly influenced by the environment for fatigue lives shorter than 10^7 cycles, although they clearly are for longer fatigue lives. The underlying mechanism for the low-cycle fatigue behavior of single-crystal Si films is discussed in light of these experimental data.

2:30 PM
The Effect of Processing, Microstructure, and Texture on the Elevated-Temperature Fatigue and Creep Behavior of Ti-6Al-4V-xB Alloys: *Wei Chen*[1]; Carl Boehlert[1]; Andrew Payzant[2]; Seshacharyulu Tamirisakandala[3]; Daniel Miracle[4]; [1]Michigan State University; [2]Oak Ridge National Laboratory; [3]FMW Composite Systems Inc.; [4]US Air Force

The effect of nominal boron additions on the elevated-temperature fatigue and creep deformation behavior of Ti-6Al-4V was evaluated. The alloys were evaluated in the as-cast and cast-then-extruded conditions. The creep resistance of the as-cast alloys significantly improved with increased B concentration, where almost an order of magnitude decrease in the secondary creep rate was observed between the Ti-6Al-4V-1B(wt.%) and Ti-6Al-4V(wt.%) as-cast alloys. For the same nominal B contents, the cast-then-extruded alloys exhibited significantly greater creep and fatigue resistance than the as-cast alloys. This was explained to be an effect of the TiB phase and α-phase texture, and the decreased lath width in the cast and extruded alloys compared with the as cast alloys. The cast-then-extruded alloys exhibited four times smaller lath widths than the as-cast alloys, and the α-phase basal plane normal and TiB whisker axis were preferentially oriented parallel to the extrusion axis.

2:50 PM
Effects of Microstructure on Fatigue Crack Growth Rate of Alloy 10 at Elevated Temperatures: *Gilbert Mora*[1]; Pete Kantzos[1]; [1]Honeywell International

The effects of microstructure on fatigue crack growth rate (FCGR) behavior have been studied in a powder metal, Alloy 10. It was processed in two ways, As-Hip and extruded and isothermal forge, and given subsolvus and supersolvus heat treatments to obtain a variety of microstructures. FCGR testing was performed using a triangular waveform at 20 cycles per minute. Dwell FCGR testing was performed using a 90 second dwell at max load. Test temperatures ranged from 315 to 760C. Fractography and metallography was used to document failure modes and microstructures. The biggest overall driver controlling dwell and non-dwell FCG was grain size, with coarse grain supersolvus microstructures displaying better FCGR behavior. Under dwell and non-dwell conditions, the failure mode for subsolvus microstructures was intergranular above 649C. For supersolvus microstructures the transition from mixed mode to intergranular was between 649 and 760C. These transitions were accompanied by an increase in FCGR.

3:10 PM
Mechanistic Studies on Fatigue of Steels Welded with IN625 Alloy: *Neeraj Thirumalai*[1]; Raghavan Ayer[1]; Cary Marzinsky[1]; Russell Mueller[1]; Dan Lillig[2]; Mark Crawford[2]; Geoff Dunn[2]; Thomas Gnaupel-Herold[3]; [1]ExxonMobil Research & Engineering Co; [2]ExxonMobil Development Company; [3]NIST Center for Neutron Research

Superior fatigue performance of welded structures is critical for offshore applications in the oil and gas industry. Recently, it was shown that X-65 (65 ksi yield strength) steel tubulars joined with IN625 weld metal illustrated superior fatigue performance compared to those welded with carbon steel weld metal. The current study was initiated to understand the mechanisms underlying the difference in the fatigue performance in these two systems. We have conducted weld and base metal fatigue crack growth studies, microstructural analysis and full-scale residual stress measurements by neutron scattering, in steels welded with carbon steel and IN625 weld metals. In this presentation we will discuss the results of this study.

3:30 PM
The Effect of Grain Boundary Microstructure on Hold-Time Crack Growth in Allvac 718Plus Superalloy: *Leif Viskari*[1]; Thomas Hansson[2]; Krystyna Stiller[1]; [1]Chalmers University of Technology; [2]Volvo Aero Corporation

High temperature Low Cycle Fatigue (LCF) tests incorporating a hold-time at maximum tensile load have shown to increase Crack Growth Rate (CGR) in Ni-base superalloys. For Allvac 718Plus, hold-time crack propagation has been shown to be predominantly intergranular. It is thus of interest to investigate any correlation between grain boundary microstructure and hold-time crack growth. Two conditions of 718Plus were investigated; a lean condition with very low amount of grain boundary phases and a rich condition with higher amount of grain boundary phases. The phases were characterized by Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) with Energy Dispersive X-ray (EDX) analyses. Hold-time testing was performed at 704ºC using a load cycle of 10Hz with intermittent hold-times of 100s at maximum tensile load. The obtained microstructural characterization results were then correlated to results from hold-time fatigue testing.

3:50 PM Break

4:20 PM Invited
High Temperature Fatigue of Nickel-Base Superalloys Single Crystals: Bernard Fedelich[1]; Rainer Sievert[1]; Hellmuth Klingelhöffer[1]; Birgit Skrotzki[1]; *Pedro Portella*[1]; [1]BAM

Due to their relatively simple microstructure, single crystal superalloys allow a useful insight into deformation mechanisms. On the other hand their intrinsic anisotropy can pose difficult questions to the measurement and interpretation of strain. In this paper we describe the mechanical response of uncoated specimens of different superalloys to fatigue loading including thermo-mechanical and biaxial fatigue. The concomitant changes in the gamma / gamma prime microstructure as well as the damage evolution are presented and set into relation to the observed mechanical behavior. On the basis of this extensive experimental

basis, a viscoplastic anisotropic model was developed and calibrated for different alloys. Finally, some practical applications are considered.

4:50 PM
Investigation of Fatigue Crack Growth Mechanisms in a Monocrystalline Ni-Based Superalloy: *Clarissa Yablinsky*[1]; Katharine Flores[1]; Michael Mills[1]; James Williams[1]; [1]Ohio State University

Historically, the critical design parameter for Ni-based superalloy turbine blades has been creep resistance. Recently, because of modern airfoil designs, the focus has broadened to include fatigue resistance, resulting in the need to better understand fatigue behavior. In this study, compact tension specimens of monocrystalline Ni-based superalloy René N5 were tested under cyclic loading conditions. Test temperature and frequency (0.5 Hz / 10 Hz) were varied in order to examine the effects of plastic zone size, recovery, and other time dependent processes on crack growth. Fracture surfaces and microstructures were characterized using a scanning electron microscope in order to examine crack path selection and the γ/γ' morphology along the crack wake and in the bulk material. Dislocation arrangements were characterized via transmission electron microscopy using conventionally prepared and site-specific foils prepared by focused ion beam techniques, in order to understand the damage mechanisms active during fatigue crack growth.

5:10 PM
Microstructural Effects on Fatigue Behavior of Nickel-based Superalloy René 88DT at 593°C: *Jiashi Miao*[1]; Tresa Pollock[1]; J Jones[1]; [1]University of Michigan

Fatigue behavior of polycrystalline nickel-based superalloy, René 88 DT, was investigated at 593°C using an ultrasonic fatigue testing apparatus. Within the testing stress range of 500 - 760MPa, all fatigue failures initiated internally. Critical microstructural features controlling fatigue crack initiation and early stage of small crack growth were identified by the combination of serial sectioning, orientation imaging microscopy (OIM) and quantitative fractographic analysis. Cyclic deformation substructures were characterized by transmission electron microscopy. Fatigue cracks initiated and propagated along {111} slip planes. Twin boundaries within favorably oriented large grains are important sources for cyclic damage in this alloy at 593°C. The influence of different microstructural factors on fatigue crack initiation and the fatigue life variability of this alloy will be described.

5:30 PM
Creep Fatigue Behavior at High Temperature of a Udimet 720 Nickel-Base Superalloy: *Thomas Billot*[1]; Patrick Villechaise[1]; Mustapha Jouiad[1]; José Mendez[1]; [1]LMPM ENSMA

Nickel-base superalloys employed for components in the hot parts of aircraft engines are subjected to both steady and fluctuating stresses due to high temperature, centrifugal force and high frequency vibrations. The present study concerns a polycrystalline cast and wrought Udimet 720 elaborated and forged by Aubert & Duval corp. and provided by Turbomeca. It focuses on the interaction between creep and fatigue at 700°C. For that, tests have been performed with a trapezoidal signal and a dwell time ranges from 1 to 50 seconds. Results are compared to "pure" creep and fatigue tests. The damage process relative to the different loadings is described from SEM observation. Deformation mechanisms (dislocations structures) have been studied by TEM. A special attention is paid on the role of the hold period to investigate the transition of mechanical behaviour and durability from the "pure" creep to the fatigue testing conditions.

Friction Stir Welding and Processing-V: Session VI

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Shaping and Forming Committee
Program Organizers: Rajiv Mishra, Missouri University of Science and Technology; Thomas Lienert, Los Alamos National Laboratory; Murray Mahoney, formerly with Rockwell Scientific

Wednesday PM — Room: 2014
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Anthony Reynolds, University of South Carolina

2:00 PM Invited
Microstructure Development of Friction Stir Welded Joints in a AlMgSc Alloy: Cesar Weis Olea[1]; *Jorge dos Santos*[1]; Telmo Strohaecker[2]; [1]GKSS Forschungszentrum; [2]UFRGS

Scandium is well known due formation of a large second phase Al3Sc which is responsible for significant microstructural control, promoting a high increase in material strength added to recrystallization inhibition. In this work, AlMgSc alloy 4 mm thick plates developed and provided by Airbus were joined by friction stir welding (FSW). Mechanical behaviour and local properties of the weld seam were evaluated by microhardness measurements, conventional tensile tests and, microflat tensile tests. Structural changes and precipitation features were investigated mainly using transmission electron microscopy, in order to understand microstructural evolution in the weld zones and to establish the relationships with mechanical behaviour. The Al3Sc precipitates present in the base material were very stable and the thermal cycle produced during welding was not able to deteriorate significantly the strengthening effect as evidenced by the mechanical testing. The AlMgSc joints presented in general similar mechanical behaviour to the base material.

2:20 PM
Monotonic and Cyclic Deformation Behavior of Friction Stir Welded (FSW) Mg/Mg- and Al/Mg-Joints: *Guntram Wagner*[1]; Dietmar Eifler[1]; Otmar Klag[1]; [1]University of Kaiserslautern

In this research project the friction stir weldability of die casted AZ31 and AZ91 was investigated. The FSW system was equipped with a measurement unit to record online temperatures and welding forces. In monotonic tensile tests with AZ31/AZ31-joints and AZ91/AZ91-joints a tensile strength at the value of the parent materials could be realized. By light microscopical investigations and two-dimensional hardness measurements it could be demonstrated that the FSW process leads to an extreme grain refinement of about 85% compared to the parent status. Furthermore lower hardness values and a significantly higher ductility was measured in the joining area. To describe the cyclic deformation behavior of the AZ31/AZ31- and AZ91/AZ91-joints, high resolution stress-strain-hysteresis as well as temperature and electrical resistance measurements were performed. As a result of the grain refinement and the higher ductility of the joints the cyclic deformation curves are characterized by cyclic hardening until macro crack growth.

2:40 PM
Effect of Interface Characteristics on Transverse Tensile Strength of Dissimilar Metal Al-Mg Friction Stir Welds: Perumal Venkateswaran[1]; Y. Chen[2]; *Anthony Reynolds*[1]; [1]University of South Carolina; [2]General Motors R&D

A series of friction stir welds was made between AZ31 magnesium alloy and 6063 aluminum alloy sheets. Weld quality was assessed via metallographic analysis, fractography, and transverse tensile testing. Transverse tensile strengths varied between 80 and 120 MPa; however, the ductility of all welds was low. Al-Mg intermetallic phases were identified by XRD and were observed to form a continuous film at the interface between the base metals. Transverse tensile strength was correlated to several interface features including: (1) maximum intermetallic layer thickness, (2) actual interface length, (3) extent of interpenetration between the aluminum and magnesium base materials, and (4) area fraction of micro-void coalescence on the fracture surfaces. Results indicate that maximizing the extent of interpenetration, promoting mechanical interlocking between the metallic phases, is the key to attaining reasonable transverse strength in Al-Mg dissimilar metal welds.

3:00 PM

Dissimilar Friction Stir Welding of AA2024-T3 and AA7075-T6 Aluminium Alloys: *Antonio Monaco da Silva*[1]; Egoitz Aldanondo[1]; Pedro Alvarez[1]; Ainhoa Lizarralde[1]; Alberto Echeverría[1]; [1]Centro de Investigación en Tecnologías de Unión LORTEK

The scope of this investigation is to evaluate the effect of joining parameters on the mechanical and microstructural properties of dissimilar aluminium alloys (3 mm thick AA2024-T3 and AA7075-T6 sheets) joints produced by friction stir welding. Material flow under different parameters and the effect of the location of the base materials during welding on the flow pattern have also been investigated. Microstructural features have been analysed; while mechanical performance has been investigated in terms of hardness and tensile testing. Onion ring formation has been associated with high rotational speeds; while at low rotational speeds the boundary between both base materials is clearly delineated. Failure of tensile specimens always occurs at the 2024 side (independent of the material location related to the pin rotation and welding direction). Microstructural observation has revealed the development of a recrystallised fine-grained stir zone, with two different grain sizes resulting from the two different base materials.

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Fatigue Behavior of Friction Stir Welded (FSW) Aluminium Joints: Guntram Wagner[1]; *Dietmar Eifler*[1]; Markus Gutensohn[1]; Masahiro Endo[2]; [1]University of Kaiserslautern; [2]Fujuoka University

For the application of FSW in monotonically and cyclically loaded components, a detailed knowledge of the deformation behavior of the joints is required. The present work provides an overview of the deformation behavior of aluminium 5454 FSW-joints. In monotonic tensile tests the yield strength of the friction stir welded joints reaches up to 90% of the value of the parent material. In fatigue tests high-resolution plastic strain amplitude and electrical resistance measurements were performed to describe the cyclic deformation. With FSW-joints welded at different welding forces single step tests were carried out at constant stress amplitudes. With higher welding forces the plastic deformation during the first cycles increases as a result of the increasing size of the softened process zone and simultaneously the fatigue life of the joints increases. The development of the change in electrical resistance correlates in detail with the fatigue life as function of the welding force.

3:40 PM Invited

Time-Dependent Variations of Residual Stresses in a Friction Stir Welded 6061-T6 Al Alloy: *Wan Chuck Woo*[1]; Zhili Feng[1]; Xun-li Wang[1]; Ke An[1]; Camden Hubbard[1]; Stan David[1]; [1]Oak Ridge National Laboratory

We report the time-dependent variations of residual stresses observed in a friction stir welded (FSW) 6061-T6 aluminum alloy using neutron diffraction. 6.5-mm thick FSW Al alloy samples were prepared and installed in the residual stress diffractometer (NRSF2) at High Flux Isotope Reactor, Oak Ridge National Laboratory. The residual stress measurements were performed as a function of time from 2 to 10,000 hours after welding at 17 locations at different distances from the weld centerline along the mid-thickness of the plate. The results show that the tensile longitudinal stress noticeably decreased from 100 to 60 MPa near the weld centerline within about 50 hours after FSW. Causes of such time-dependent behavior could be attributed to the natural aging phenomenon of precipitates and the low temperature creep process in FSW Al alloys.

4:00 PM Break

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Friction Stir Welding of Sc-Modified Al-Zn-Mg-Cu Alloy Extrusions: *Carter Hamilton*[1]; Stanislaw Dymek[2]; Oleg Senkov[3]; [1]Miami University; [2]AGH University of Science and Technology; [3]UES, Inc.

Small additions of Sc to Al-Zn-Mg-Cu 7000 series alloys can significantly improve mechanical properties and augment the strength retention at low and elevated temperatures. This research program evaluates the residual mechanical and corrosion properties of Sc-modified Al-Zn-Mg-Cu alloy extrusions joined through friction stir welding. SSA038-T6 extrusions were friction stir welded at 175, 225, 250, 300, 350 and 400 RPM (weld velocity and force were held constant). Mechanical tests demonstrated that the highest joint efficiency was achieved at 250 RPM, but that the exfoliation corrosion resistance was lowest for this weld condition. Microstructural investigations through light, scanning electron and transmission electron microscopy correlate the residual properties of the welded alloy with the microstructure in each of the unique weld regions.

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Friction Stir Welding Characterization of Double-Welded Al-7075 Joints: *Meysam Mirazizi*[1]; Amir Hosein Kokabi[1]; [1]Sharif University of Technology

Double-welded friction stir welding of 7075 aluminium alloy was performed to investigate the effects of the second pass of weld on the microstructure of first pass and microstructure of final weld. Also the effect of pin length that influences the amount of overlapping of two stir zones was studied. Furthermore, the effect of heat input on microstructure of weld investigated by using various rotating speeds and welding travel speeds that results to various amount of heat input (hot, average, cold). Tensile test and shear punch test (SPT) were conducted to determine mechanical properties of joints. Experimental results indicated that second pass of weld and amount of heat input had significant effects on microstructure of weld. Somewhat overlapping of two passes in the middle of joint improved the mechanical properties of joint.

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Reducing Tool Axial Stresses in HSLA-65 during the Plunge: *Kenneth Ross*[1]; [1]Brigham Young University

Using friction stir welding to join high-carbon steels would be more common if the PCBN tool had a longer tool life. Because PCBN tools appear to fail due to normal loading, reducing normal stress in the tool should increase tool life. The highest stresses occur during the plunge. A broad study of z stresses during the plunge, first in Aluminum 7075 then in HSLA-65 steel, was conducted. In aluminum the lowest stresses were produced using pilot holes and a force control program. In HSLA-65 steel pilot holes greatly reduced stresses. It was discovered that increasing weld power decreases z-stress while the pin is engaged. When the shoulder is engaged increasing power increases z stress. A statistical analysis was performed and equations were derived to model stress during the plunge. Parameters are given for running a plunge with the least possible stress. Other findings are also discussed.

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Material Flow and Texture Analysis of Friction Stir Welded Aluminum Alloys: *Suk Hoon Kang*[1]; Kee Bum Kim[1]; Heung Nam Han[1]; Kyu Hwan Oh[1]; Jae-Hyung Cho[1]; Chang Gil Lee[1]; Sung-Joon Kim[1]; [1]Seoul National University

Friction Stir Welding (FSW) is an intricate process because threaded tool rotates and moves forward at the same time. Many researches on numerical flow simulations and direct flow visualization have been conducted for understanding the material flow behavior during the FSW process. In this study, material flow of Al6061-T651 sheet joined by FSW was investigated by electron backscattered diffraction (EBSD). It was confimed that the forward movement of the tool pin resulted in a loose contact between the tool pin and the receding material during FSW. The amount of incompletely rotated material due to the loose contact was estimated from the tilt angle of the shear texture which could be obtained from a pole figure.

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Investigation of a Donor Material in Friction Stir Welding: Justin Rice[1]; Saptarshi Mandal[1]; *Abdelmageed Elmustafa*[1]; [1]Old Dominion University

Excessive tool wear caused during the plunge phase of friction stir welding (FSW) is hindering the application of FSW of hard materials such as steel. This research uses a finite element model of the Johnson-Cook material constitutive law to investigate the shear stresses and axial forces experienced by the tool during the entire plunge stage of FSW. The model in this research consists of both a deformable workpiece and a deformable tool. By implementing the concept of using a donor material, we are able to have localized pre-heating and minimize the forces throughout the tool, therefore reducing tool fracturing and production costs. The numerical simulation data supports the concept of using a donor material to reduce tool wear and the need to implement this concept to experimental work for further verification.

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Tool Degradation during Friction Stir Welding of Aluminum: *Kazutaka Okamoto*[1]; Akihiro Sato[1]; Seung Hwan Park[1]; Satoshi Hirano[1]; [1]Hitachi, Ltd

Friction stir welding is well matured process and already implemented to aluminum alloys in various industries especially of transportation. Tool steel is a common material for the welding tool, which demonstrates acceptable performance as well as cost impact. However, tool failure such as pin snap off is sometimes observed even in aluminum welding. In this study, tool degradation is discussed considering several fundamental aspects; fatigue damage, thermal damage (tempering), chemical damage (Al diffusion) and impact damage.

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

Sponsored by: 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: Energy Committee, TMS: Nanomaterials Committee, TMS: Superconducting and Magnetic Materials Committee, TMS: Thin Films and Interfaces Committee
Program Organizers: Long Qing Chen, Pennsylvania State University; Mark Palmer, Kettering University; Sung Kang, IBM Corp

Wednesday PM Room: 2022
February 18, 2009 Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM
Alumina Coated Ssteels by the Sol Gel Method for a New Lamellar Soft Magnetic Composite: *Patrick Lemieux*[1]; Roderick Guthrie[1]; Mihaiela Isac[1]; [1]McGill University

A new soft magnetic composite has been developed. It is produced by sintering or forging lamellar particles of steel obtained from a cut ribbon. Prior to cutting, the steel ribbon is coated with a Sol-Gel solution to form a refractive dielectric. The composition, viscosity and concentration of the sol Gel solution must be adapted to the coating process (spray or dip) and to the overall soft magnetic composite process and application. An alumina-PVP composite sol-gel solution was developed in order to coat ribbons at high speed and to give a final 0.5 µm thick layer of refractive electrical insulator. Final properties of the soft magnetic composite obtained are reviewed.

2:20 PM
Correlation between the Morphological and Electrochemical Properties IrO_2 Based DSA® Anodes: *Ozgenur Kahvecioglu*[1]; Servet Timur[1]; [1]Istanbul Technical Univ

In this study, IrO_2 was coated onto titanium anodes both by thermal and sol-gel procedures. Surface morphology of these coatings was investigated by SEM and the phase determination was carried out by thin film XRD analysis. The electrodes were electrochemically characterized by means of applying potentiodynamic polarization tests in 1.0 M H_2SO_4 solution. Although the surfaces of the electrodes obtained with two coating procedures show variant morphologies (nano-structured, mud crack, nodular based flats) and it was expected that these electrodes show radical variation in the electrochemical activation due to having different *S* (active site), no difference was seen. That is to say, *a* (materials coefficient) and *b* (Tafel slope) values of the materials didn't demonstrate a radical change with the morphology. Increasing current densities resulted in an increase in the Tafel slope from 59 mV/dec to 90 mV/dec.Keywords: IrO_2,DSA®,Electrochemical Activation

2:40 PM
Hetero and Homoepitaxy of ZnO by Metalorganic Chemical Vapor Phase Epitaxy: *Tommy Ive*[1]; Tammy Ben-Yaacov[1]; Chris Van de Walle[1]; Umesh Mishra[1]; Steven DenBaars[1]; James Speck[1]; [1]University of California, Santa Barbara

Thin films of Zn-face ZnO(0001) were grown by metalorganic chemical vapor epitaxy on Si(111), Al_2O_3(0001), GaN(0001) and on high quality ZnO(0001) substrates. The focus of our study was on homoepitaxy which yielded excellent results. Atomic force microscopy revealed that our homoepitaxial films exhibited monolayer steps on the surface. The root-mean-square roughness was 0.09-0.15 nm for a 20×20 µm² area. The x-ray rocking curve full-width-at-half-maximum was ≤36 arcsec across both the (0002) and the (20-21) reflections. These full widths are comparable to the corresponding values for the bare ZnO bulk substrates. We found that a growth temperature >950°C was needed to obtain a smooth two-dimensional surface. Samples grown below 950°C exhibited a very rough three-dimensional surface morphology. This indicates that with respect to the comparatively low growth temperatures reported in the literature, much higher growth temperatures might be needed in order to obtain a smooth two dimensional surface morphology.

3:00 PM
Nonvolatile Memory Property of WSi_2 and $TiSi_2$ Nano-Particles in SiO_2 Dielectrics: Ki Bong Seo[1]; Seung Jong Han[1]; Dong Uk Lee[1]; Seon Pil Kim[1]; *Eun Kyu Kim*[1]; [1]Hanyang University

We have studied nonvolatile nano-floating gated memory (NFGM) device with WSi_2 and $TiSi_2$ nano-particles, and characterized electrical properties such as threshold voltage, endurance, and retention time. Silicide films with thickness of 5 ~ 10 nm were deposited by dc sputter on tunnel oxide layer with 4.5-nm-thickness, which was grown by thermal oxidation process. Then, the silicide nano-particles were formed by RTA process at temperature range of 600 to 1000°C for 1 min under nitrogen gas ambient. Finally, SiO_2 control oxide layer with 30-nm-thickness was sputtered and then an Al gate electrode was evaporated. These nano-floating gate capacitors with nano-particles in SiO_2 dielectrics were characterized by capacitance-voltage and current-voltage measurements. The memory windows were ranged from 1 V to 4 V by applying the gate voltages from ± 3 V to ± 7 V. Then, the programming and erasing speeds were measured about 50 ms and 600 ms, respectively.

3:20 PM Break

3:40 PM
Quantum Dots Shape Effects on Surface Plasmons Propagating in Chains: *Jimena Vergara Mojica*[1]; Angela Camacho[1]; [1]Universidad de Los Andes

We are interested in the study of collective excitations in quantum dot chains because these can be used to effectively transmit information at nano scale and to control spontaneous and stimulate electromagnetic emission in the quantum dots. [1] This work is centered in the study of semiconductor and metallic one-dimensional quantum dot arrays. We analyze how the geometry of the dot affects the collective oscillation of charge and its propagation. Furthermore, we introduce the Coulomb interaction between charges and compare our results with the ones where this interaction is neglected. We find out that Coulomb interaction plays an important role in these systems, which are good candidates to be used as nanometric devices. [1] A.V.Akimov, A.Mukherjee, C.L. Yu, D.E Chang, A.S.Zybrov, P.R. Hemmer, H Park and M.D Lukin, Generation of Single optical plasmons in metallic nanowires coupled to quantum dots, Nature 450, 402 (2007).

4:00 PM
Study of Properties in MgB_2 Doped with NbB_2 by High Energy Ball Mill (Spex 8000D): *Yenny Cardona-Quintero*[1]; Richard Perez[1]; Oswald Uwakweh[1]; Eric Hellstrom[2]; David Larbalestier[2]; [1]University of Puerto Rico-Mayaguez; [2]Florida State University

The doping of Magnesium Diboride (MgB_2) by the addition of Niobium Diboride (NbB_2) at 5 at% is carried out by high-energy ball milling at 60, 120, 180 and 300 minutes of milling. All the samples are processed in SPEX 8000D followed by cold and hot isostatic pressing. The obtained samples are characterized by measuring critical current density (J_c), critical temperature transition (T_c) and critical magnetic field (H_{c2}), while structural characterization will be undertaken with X-ray diffraction. The values of these properties will be compared with the ones obtained in previous studies carried out on bulk and doped MgB_2. In addition, doping with other transition metal diborides such as Titanium Diboride (TiB_2), Chromium Boride (CrB_2) and Tantalum Boride (TaB_2) will be carried out. The results of this work will be an important contribution towards the development of improved superconductors with better J_c capable of performing at higher T_c.

4:20 PM
Ultraviolet Photoconductive Properties of TiO2 Nanotubes Grown by Atomic Layer Deposition: *Yung-Huang Chang*[1]; Chih Chen[1]; [1]National Chiao Tung University

Self-organized TiO2 nanotubes are grown on Si substrates using anodic aluminum oxide as a template and atomic layer deposition (ALD) for the deposition of TiO2. With the aid of the ALD, the deposition temperature can be as low as 400°C and no any metal catalysts or seed layers are needed. Each nanotube is perpendicular to the Si substrate. We controlled the deposited cycles of ALD to modify the morphology of TiO2 nanotube arrays. The structure of the TiO2 appears to be polycrystalline, and their grain size become larger as deposited cycles increase. The Ultraviolet (UV) photoconductive properties of TiO2 nanotubes were investigated to obtain the best transformation efficiency for the application of UV detector. The results of photoluminescence, transmission

electron microscope, and the performances of UV photoconduction for the TiO2 nanotube arrays will be presented in the conference.

General Abstracts: Materials Processing and Manufacturing Division: Session II

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: Surface Engineering Committee
Program Organizers: Thomas Bieler, Michigan State University; Neville Moody, Sandia National Laboratories

Wednesday PM Room: 3022
February 18, 2009 Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM
Bulk Nanoscale Hydroxyapatite Structures Using Microwave Sintering: Sudip Dasgupta[1]; Amit Bandyopadhyay[1]; *Susmita Bose*[1]; [1]Washington State University

Nanostructured hydroxyapatite (HA) is of significant interest because of the nanoscale feature of the inorganic part of natural bone. In our research, nanostructure HA compacts were processed with below 200 nm average grain size using microwave sintering which showed improved mechanical and biological properties. Nano HA compacts showed compressive strength of 395 ±36 MPa, indentation hardness of 8.4 ± 0.4 GPa and indentation fracture toughness of 2.0 ± 0.1 MPa $m^{1/2}$. These numbers are significantly higher compared to pure HA compacts with micron size grains. Nano HA compacts were assessed for cell material interaction using SEM, MTT and immunochemistry assays using protein expression studies with human osteoblast cell line for 1, 5 and 11 days. These studies show better cell material interaction on nano grain HA compared to compacts with larger grains. The presentation will focus on processing and characterization of microwave sintered HA compacts.

2:20 PM
Dilatometric and Differential Scanning Studies of Cryogenic Treatment Methods and Cryogenically Treated Samples: *Harish Sivasankaran*[1]; Bensely Albert[2]; Mohan Lal D.[2]; Nagarajan G.[2]; [1]Arizona State University; [2]College of Engineering Guindy

Even though cryogenic treatments are widely used as strengthening mechanisms for metals, especially tool steels, the underlying metallurgical principles of the same have remained unexplained. The present investigation aims at determining the actual metallurgical metamorphosis during the cryogenic treatment of a case hardened gear material, En353.A differential scanning calorimeter (DSC) and a thermomechanical analyzer (TMA) are used and the relevant parameters are measured during the course of the treatment procedure itself. Simultaneous measurement by DSC and TMA enables the comparison of the cryogenic treatment methods, namely Deep Cryogenic Treatment (DCT) and Shallow Cryogenic Treatment (SCT) and the data obtained is studied. Possible means of reducing the retention time for Deep Cryogenic Treatment (DCT) process is discussed. Quantitatively, the values from TMA are used to validate the treatment procedures.

2:40 PM
Effect of Pulse Magneto Oscillation on the Solidification Structure of Pure Aluminum During Crystal Growth Stage: *Yongyong Gong*[1]; Qijie Zhai[1]; [1]Shanghai Univ

The effect of Pulse Magneto Oscillation (PMO) on solidification structures of pure aluminum at different solidification stages has been researched and the refinement mechanism has been analyzed. The previous experimental results show that PMO can not refine the solidification structure of pure aluminum when it is only applied to high temperature liquid metal. However, the solidification structures are significantly refined when PMO is applied from the end of nucleation till beginning of crystal growing. But if cooling ability of the mold and treatment frequency of PMO are increased, the solidification structures are also greatly refined when PMO is applied during the stage of crystal growth.

3:00 PM
Finite Element Analysis and Experimental Investigation of the Effect of Spatial Energy Distribution on Dual-Beam Welding of Aluminum Alloys: *Rouzbeh Sarrafi*[1]; [1]Southern Methodist University

The effect of beam splitting on the laser welding of aluminum in conduction and keyhole modes is studied through a FEM model as well as experiments. For the conduction mode, four types of energy distribution were compared, 1) single laser beam, 2) the same laser beam split into two equal beams, 3) laser beam split unequally and the weaker beam leads, and 4) laser beam split unequally and the stronger beam leads. The results showed the variation of molten pool profile with the spatial energy distribution. Furthermore, the setup with strong beam ahead can reduce porosities in the weld since it provides a notably slower cooling rate for the molten pool before solidification compared to the other cases of spatial energy distribution. The analysis results showed reasonable agreement with experiments. In addition, the effect of spatial energy distribution on keyhole laser welding of aluminum alloys was experimentally investigated.

3:20 PM
Lamellar Decomposition in U-Nb Alloys: *Robert Hackenberg*[1]; Heather Volz[1]; Robert Dickerson[1]; Pallas Papin[1]; Robert Forsyth[1]; Ann Kelly[1]; Tim Tucker[1]; Robert Field[1]; [1]Los Alamos National Laboratory

Lamellar decomposition products result when U-Nb alloys are transformed between about 300C and the 650C monotectoid temperature. As such microstructures give undesirable properties, TTT diagrams were generated for U-5.6 wt% Nb and U-7.7 wt% Nb alloys. Detailed kinetic studies of these reactions were undertaken to better understand their mechanisms. The relevant parameters for the initial discontinuous precipitation as well as the succeeding discontinuous coarsening reactions were investigated via light microscopy, X-ray diffraction, SEM, and TEM. These results will be compared with theory. Attention will be paid to the rates and degrees to which these various aging reactions drive the system toward its final equilibrium state.

3:40 PM
Monitoring of Spatters by Using Microphone during Gas Tungsten Arc Welding of Galvanized High-Strength Steel DP980: *Wei Huang*[1]; Shanglu Yang[1]; Dechao Lin[1]; Radovan Kovacevic[1]; [1]Southern Methodist University, Research Center for Advanced Manufacturing

Galvanized high-strength steels have broad applications in automobile industry. However, during the welding process, many spatters are produced due to a lower boiling point of zinc coat (907°) than the melting point of steel (over 1300°). Since spatters greatly deteriorate the quality of welds, it is necessary to monitor the presence of spatters and achieve quality control. In this paper, a microphone was used to acquire the airborne acoustic signals during gas tungsten arc welding of galvanized high-strength steel DP980. The signals were denoised by spectral subtraction and analyzed in wavelet domain. The results show that by applying noise reduction, spatters can be detected in a noisy environment. In addition, the wavelet analysis shows a good correlation between the signals in the frequency range [0:100 Hz] and the quality of welds, which can be explained by the frequency characteristic of the oscillation behavior of molten pool.

4:00 PM
Porous and Permeable Metal Membranes from Slurry Precursors: *Kevin Hurysz*[1]; Jason Nadler[1]; [1]Georgia Tech Research Institute

Oxide slurries present a unique opportunity for fabricating thin, porous and permeable metal structures. Powder raw material precursors are mixed with a binder and solvent into a viscous slurry. Air is then introduced into the structure via high shear mixing. By employing traditional tape casting and doctor blading techniques, thicknesses of 100 µm can be achieved. The tape is then dried and fired in a controlled atmosphere to form a metal membrane. The advantage to this process is that feature sizes can be substantially decreased through shrinkage: evaporation of the solvent, binder burnout, reduction shrinkage, and sintering of the structure. Thicknesses of 30 µm with pore sizes on the order of 10 – 50 µm have been achieved.

4:20 PM
Rapid Materials Processing and Surface Sculpting Using Electron Beam and Laser Processes: *Bruce Dance*[1]; [1]TWI Ltd

For up to fifty years high energy density power beams have been used in many industrial processes. More recently, 'Surfi-Sculpt®' processing has been

developed. This novel process typically uses a focused energy beam to displace material in the liquid phase. By means of controlled and usually repeated movements of the beam with overlapping or intersecting paths, extraordinarily complex 3-D geometries may be generated rapidly and precisely. The Surfi-Sculpt process can be implemented using electron beams and laser beams, in materials ranging from metals to polymers. Feature sizes from tens of mm to tens of μm may be created quickly and efficiently. Novel materials thus processed are under evaluation for a wide range of applications including composite to metal 'Comeld®' bonding, materials for enhanced heat transfer and flow control, as well as biocompatible/bone ingrowth materials. Examples of different types of treatments are presented, together with some recent developments.

4:40 PM
Solidification Structure Transformation of Bearing Steel under Electric Current Pulse: Ma Jianhong[1]; Li Jie[1]; Gao Yulai[1]; Chen Zhongxin[2]; Li Zheng[2]; *Zhai Qijie*[1]; [1]Shanghai University; [2]Special Steel Branch, Baoshan Iron & Steel Co., Ltd, P.R. China

The solidification structure transformation of bearing steel with different patterns of electric current pulse (ECP) was carried out. The results showed that the ratio of equiaxed dendrites could only be slightly increased and little reduction of the primary dendritic arm was obtained under the effect of high voltage and low discharge frequency of ECP. In contrast, high ratio of equiaxed dendrites and large reduction of the primary dendritic arm were generated by low voltage and high discharge frequency of ECP. It was deemed that grain refinement could be attributed to the heterogeneous nucleus formed on the top surface and their falling by the agitation resulted from the ECP. For high voltage ECP, non-crowded agitation and lower accumulated heat were generated and therefore little nucleus dissociated from the top surface. Contrarily, much more nucleus falling due to combined action of dense agitation and more accumulated heat by employing low voltage ECP.

5:00 PM
The Effect of Feedstock Composition on Defect Evolution in Powder Injection Molded Ceramic Microarrays: *Sachin Laddha*[1]; Sundar Atre[2]; Kevin Simmons[1]; [1]Pacific Northwest National Laboratory; [2]Oregon State University

Powder injection molding is a cost-effective to manufacture Microchannel arrays (MCA), which are used as the major component and design feature for many microsystems in a large variety of applications, such as microfluidics, micros optics, etc. In this paper, characterization of feedstock consisting of alumina nanopowder (average particle size of 400nm) with ethylene-propylene/wax and polyacetal binder systems for micro-powder injection molding is reported. It is found that the wax-based binder system performs the lowest viscosity and heat capacity as well as greater pseudo-plasticity than the polyacetal binder system. However, the results from Moldflow simulations inferred that the polyacetal-based system, though having a higher viscosity than wax-based system at higher shear rate, fills the microcavities (50μm) in a more efficient way. Also, a design of experiments involving variations in powder injection molding conditions were carried out to map the homogeneity at the macroscale (part level) and microscale (particle level) in MCA.

5:20 PM
Viscosity – Structure Relationships in Copper Foams: *Kevin Hurysz*[1]; Jason Nadler[1]; [1]Georgia Tech Research Institute

Metallic foams can be constructed by mixing oxide precursors with a binder and solvent, introducing air into the resulting slurry by mechanical mixing, drying, and heat treating in controlled atmospheres. Slurry viscosity plays a key role in the ability to form a green porous structure. The combined action of drainage, coalescence, and Ostwald ripening among bubbles will cause foams made from fluids of low viscosity to collapse. High viscosity slurries will not foam because mixing that is turbulent enough to incorporate air into the structure is impossible to achieve. A critical target viscosity range for foaming clearly emerges: 1 000 to 5 x 10^5 cP. This paper will investigate the structure of thin, tape cast foams made from slurries of cuprous oxide (Cu_2O), polyvinyl alcohol (PVA), and water.

General Abstracts: Structural Materials Division: Session III

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

Wednesday PM — Room: 2018
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM
A New Microstructure-Sensitive Crystallographic Constitutive Model for Creep of Ni-Base Single -Crystal Blade Alloys: *Yoon Suk Choi*[1]; You-Hai Wen[1]; Triplicane Parthasarathy[1]; Christopher Woodward[2]; Dennis Dimiduk[2]; [1]UES Inc; [2]Air Force Research Laboratory

We developed a microstructure-sensitive, mechanism-based crystallographic constitutive model to predict creep behavior of Ni-base single-crystal blade alloys over a wide range of stress and temperature. The new creep constitutive model accommodated major slip-system-based dislocation micro-mechanisms to predict primary and secondary creep, and adopted a phenomenological creep formulation to predict tertiary creep. In particular, the new model was directly linked to a γ'-rafting model, which predicts the evolution of the γ'-precipitate morphology during creep, in order to update the microstructural information at every time step of the creep simulation. We implemented the new creep model into the FEM package ABAQUS through the User MATerial subroutine (UMAT) and performed creep simulations for selected stress and temperature regimes with the microstructural information as variables. The model predictions were primarily intended to clarify the effect of the microstructural heterogeneity on the creep variability for various stress and temperature regimes.

2:20 PM
Characterisation of Microplasticity in TiAl Based Alloys: *Francisco Garcia-Pastor*[1]; Hui Jiang[2]; David Hu[2]; Xinhua Wu[2]; Michael Loretto[2]; Michael Preuss[1]; Philip Withers[1]; [1]The University of Manchester; [2]The University of Birmingham

Three different microstructural variants (nearly fully lamellar, fully lamellar and duplex) of two-phase (α_2+β) Ti-44Al-8Nb-1B (at%) have been studied by in-situ loading, coupled with acoustic emission and image correlation, electron backscatter diffraction and transmission electron microscopy. The observations show that the onset of microyielding occurs at different stress levels: the lamellar materials yielding at the lowest stress and duplex material withstanding the highest stress before any microplasticity was observed. The early microyielding observed in the lamellar microstructures is explained by the strong strain heterogeneity seen at early stages during loading using image correlation and post-mortem TEM analysis. It was possible to relate the level of microyielding to the orientation of the lamellae in respect of the loading direction. In contrast, duplex microstructures show no strain heterogeneity until its macroscopic yield point. This strain heterogeneity may lead to stress concentrations and early cracking detected by acoustic emission.

2:40 PM
Effect of Strain Rate, and Deformation Temperature on the Microstructure of an Equal Channel Angular Extrusion (ECAE) Processed Ti-6Al-4V Alloy: *Rabindra Mahapatra*[1]; Shankar Sastry[2]; [1]Naval Air Systems Command; [2]Washington University

The Ti-6Al-4V alloy was ECAE processed to produce ultra-fine grains of 1-2μm. The uni-axial compression experiments to simulate forging parameters subsequent to ECAE processing were carried out to elucidate whether, the ultra-fine grained microstructure of the alloy can be sustained after the deformation. The ECAE processed Ti-6Al-4V alloy showed no significant grain growth when

deformed at 750°C, at a strain rate of 0.1"/sec., where as when deformed at same temperature, a strain rate of 0.001"/sec., both the recovery and grain growth were observed.

3:00 PM

Quasi-Static and Dynamic Torsional Deformation Behavior of API X70 and X80 Linepipe Steels: *Yongjin Kim*[1]; Yanggon Kim[1]; Sunghak Lee[1]; [1]Pohang University of Science & Technology

This study aimed at investigating quasi-static and dynamic torsional deformation behavior of three API X70 and X80 linepipe steels fabricated by varying alloying elements and hot-rolling conditions. Quasi-static and dynamic torsional tests were conducted on these steel specimens, which had different grain sizes and volume fractions of acicular ferrite and polygonal ferrite, using a torsional Kolsky bar, and then the test data were compared via microstructures, tensile properties, fracture mode, and adiabatic shear band formation. The dynamic torsional test results indicated that the steels rolled in the single phase region had the higher maximum shear stress and fracture shear strain than the steel rolled in the two phase region because their microstructures were composed of acicular ferrite. Particularly in the X80 steel rolled in the single phase region, the increased dynamic torsional properties could be explained by the decrease in the overall effective grain size.

3:20 PM

High-Temperature Surface Stability and Creep Behavior of Ni-Based Alloys in Impure-He Environment: *Raghavendra Adharapurapu*[1]; Deepak Kumar[1]; Chris Torbet[1]; Tresa Pollock[1]; J Jones[1]; Gary Was[1]; [1]University of Michigan

The high-temperatures and long lifetimes expected in the very high temperature reactor (VHTR) for the Next Generation Nuclear Plant (NGNP) necessitate the use of alloys with good surface stability as well as corrosion and creep resistance. However, the understanding of alloy stability and creep strength in an environment of helium containing impurities such as $CO+CO_2$ is currently insufficient for long term predictions of crucial properties. New alloying strategies to improve strength and creep resistance over Alloy 617 through the addition of solution hardening elements such as Re and W are presented. Laboratory air cyclic oxidation behavior at 1000°C is described for a set of 11 experimental alloys. Creep behavior and the surface stability of selected alloys exposed to an impure helium environment (in carburization, decarburization atmospheres) at 1000°C is also examined.

3:40 PM

Low Torsional Ductility Induced by Shear Deformation Localization of a High Strength Steel Wire: *Youshi Hong*[1]; Xiaolei Wu[1]; Zhijia Wang[1]; [1]Institute of Mechanics, Chinese Academy of Sciences

Adequate torsional ductility is required by high strength steel wire products because the wires are inevitably subjected to cabling and brunching in applications. The torsional ductility of the wire is characterized by the number of twists in torsion test with a given gauge length. In this investigation, the values of torsional ductility for a high strength steel wire with different heat-treatment states were tested and examined. Torsion deformation characteristics, i.e. the values of shear strain along the longitudinal axis of the wire specimen were measured. Torsion rupture modes and fracture surfaces of the wire specimens were examined. A numerical simulation was performed to illustrate the temperature field of wire cross section. The results indicate that the low torsional ductility of the wire originates from shear deformation localization due to nonuniform microstructure distribution resulted from nonuniform temperature field, which eventually leads to delamintion and final fracture of the wire specimen.

4:00 PM

Microstructural Characterization Of High-Explosive Driven Tantalum: *Veronica Livescu*[1]; John Bingert[1]; Thomas Mason[1]; [1]Los Alamos National Laboratory

Three-dimensional high-explosive (HE) shock loading results in a triangular wave profile. While shock wave interaction in three-dimensions is a complex phenomenon, post-mortem interrogation and quantification of damaged microstructures provides insight towards the understanding of dynamic damage evolution. This work investigates microstructural damage and deformation in Tantalum specimens tested in HE drive experiments. Volumetric reconstruction of the damage field revealed substantial differences compared to the damage structure observed in plate-impact experiments on equivalent material. Electron Backscatter Diffraction (EBSD) was used to capture microstructural changes due to shock loading, for identification and quantification of twinning, and for characterization of potential orientation-related phenomena contributing to void nucleation processes. These results offer insight on the variables affecting damage features, which is essential to the validation of predictive damage models.

4:20 PM

Modeling the Room Temperature Deformation Behaviour of a Commercial Ti-6Al-4V Alloy for Low and High Strain Rates: *Frederik Coghe*[1]; Luc Rabet[1]; Paul Van Houtte[2]; [1]Royal Military Academy; [2]Katholieke Universiteit Leuven

This work will show some results on the modeling of the plastic deformation behaviour of a Ti-6Al-4V alloy. Cylindrical specimens, originating out of a rod in the mill-annealed condition and machined along different sample orientations, were compressed (at room temperature) at low and high strain rates using a servo-hydraulical testing machine and a Split Hopkinson Pressure Bar setup. The texture of the specimens before and after deformation was determined by the use of XRD and EBSD techniques. The Visco-Plastic Self-Consistent plasticity code (VPSC7) was used in order to try to simulate the texture evolution during deformation and to identify the active slip and twinning systems as a function of initial texture and strain rate. This work will focus in particular on the determination of the active twinning systems and on the influence of twinning on the overall deformation behaviour.

4:40 PM

Studies on Transient-Stage-Scale Growth on Fe-22 wt.% Cr Alloys Containing 120 ppm La + 270 ppm Ce: Laura Maria Fernandez Diaz[1]; *Jingxi Zhu*[1]; G.R. Holcomb[2]; P.D. Jablonski[2]; D.E. Alman[2]; Sridhar Seetharaman[1]; [1]National Energy Technology Laboratory - Carnegie Mellon University; [2]National Energy Technology Laboratory

Reactive elements (RE), such as Ce, La or Y, are known to improve oxidation resistance of Fe based alloys that form Cr2O3 scales. The current investigation aims to characterize the oxide scale in a Fe-22 wt.% Cr alloy containing 120 ppm La and 270 ppm Ce (added during melt-stage processing) as a function of oxidation times (at 800C in dry air) during the transient stage of scale formation. The surface oxidation processes were imaged in-situ through a Confocal Scanning Laser Microscope (CSLM). The results are correlated with post-experiment characterization through FEG-SEM and dual beam FIB-SEM. The evolution of the reactive-elements-containing scale, its morphology and composition are determined.

5:00 PM

Development and Ballistic Testing of a New Class of Autotempered High Hard Steels under Military Specification MIL-DTL-46100E: *William Gooch*[1]; Dwight Showalter[1]; Matthew Burkins[1]; Jonathan Montgomery[1]; Richard Squillacioti[1]; Andrew Nichols[2]; Larry Martin[2]; Ronald Bailey[2]; Glenn Swiatek[2]; [1]US Army Research Laboratory; [2]Allegheny Technologies Inc

The US Army Research Laboratory (ARL) was directed to expand current steel armor plate production as the large military demand for armor plate exceeded the production capacity at US steel facilities for quench and tempered high hard(HH)steel armor plate. The solution was to expand the availability of HH steels under the current military specification to include a new class of air-quenched, autotempered steels that do not use existing water quench and temper facilities. Allegheny Technologies Incorporated (ATI) developed the autotempered steel alloy ATI500 that has physical and mechanical properties that meet the HH specification. ARL procured sufficient amounts of ATI500 plate to allow acceptance testing and certification of ATI500 as complying with First Article requirements in a new MIL-DTL-46100E specification. This paper documents the development of ATI500 and subsequent ballistic testing and inclusion into the specification as Class 2 auto-tempered HH armor steel.

Magnesium Technology 2009: Alloys IV: Yttrium and Tin

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

Wednesday PM Room: 2006
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Menachem Bamberger, Israel Institute of Technology; Alan Luo, General Motors Corp

2:00 PM Introductory Comments

2:05 PM
Microstructure and Mechanical Properties of Mg-Al-Mn and Mg-Al-Sn Alloys: *Alan Luo*[1]; Anil Sachdev[1]; [1]General Motors Corp

The Mg-Al-Mn (AM) based cast alloys were optimized for balanced tensile properties (strength and ductility) and reasonable response to heat treatment. 1-3% tin addition was found to be effective in strengthening the AM alloys due to the precipitation of Mg2Sn phase in the Mg-Al-Sn (AT) alloys. The new AT72 (Mg-7%Al-2%Sn) offers a well balanced strength and ductility as well as good corrosion resistance. T5 (artificial aging after as-cast condition) is recommended for additional strengthening effect of AM and AT alloys.

2:25 PM
Directionally Controlled Precipitation on Twin-Boundaries in Mg-Zn-Y Alloys: *Julian Rosalie*[1]; Hidetoshi Somekawa[1]; Alok Singh[1]; Toshiji Mukai[1]; [1]National Institute for Materials Science

Precipitation strengthening in Mg-Zn(-X) alloys occurs via the formation of a fine dispersion of β_1' rods aligned parallel to the hexagonal axis. In this study, controlled deformation has been used to alter the precipitation behaviour in a Mg-2.8at%Zn-0.4at%Y alloy. The resulting microstructures were studied via conventional and high-resolution transmission electron microscopy. Texture was developed through extrusion at 300°C, followed by controlled room-temperature deformation to generate microstructures where yield occurred either with, or without, twinning. The ageing response was monitored using hardness testing. Both compressive and tensile deformation accelerated the ageing response. Compression parallel to the extrusion axis also generated a high volume fraction of twins. Precipitates on the twin boundaries assumed a low aspect-ratio morphology different from the usual high-aspect ratio rods. The change in precipitate morphology was due to the reduced matrix symmetry at the twin boundary.

2:45 PM
Effect of Extrusion Temperature on the Microstructure and Mechanical Properties of Mg97Y2Zn1 Alloy: Bin Chen[1]; *Xiaoqin Zeng*[1]; Dongliang Lin[1]; [1]SJTU

The microstructure and mechanical properties of as-cast and extruded Mg-Y-Zn alloys were investigated. It was found that the addition of yttrium and zinc not only influence the microstructure of Mg-Y-Zn alloys but also their mechanical properties. With increasing yttrium or yttrium and zinc total alloy content, the amount of secondary phases is also increased. The long-period stacking (LPS) structure was observed at both as-cast and extruded Mg-Y-Zn alloys. The formation of LPS structure in matrix was attributed to the dissolution of a certain amount of Y and Zn. The yttrium and zinc addition play very important role in mechanical properties of extruded Mg–Y–Zn alloys. The extruded Mg-Y-Zn alloys exhibit excellent mechanical properties at both ambient temperature and elevated temperature. Their excellent mechanical properties were thought due to the strengthening by the grain refinement, solid solution, formation of LPS structure and distribution of fine Mg24Y5 phase.

3:05 PM
Effect of Trace Elements on Age Hardening Behavior of Mg-Sn Alloy: Do Hyung Kim[1]; Hyun Kyu Lim[1]; Joon Seok Kyeong[1]; Won Tae Kim[2]; *Do-hyang Kim*[1]; [1]Yonsei University; [2]Cheongju University

Mg-Sn alloy system is well known as one of precipitation hardening type Mg-based alloys due to high solubility of Sn at high temperature. In the present study, to improve age hardening response of Mg-Sn alloy, 0.2at% of various trace elements such as Si, Ge, Ca, Ti and Zr are added in Mg-1.5at%Sn alloy. All of trace elements remarkably improve the yield strength level of Mg-Sn binary alloy after aging treatment. From the result of TEM analysis, the strength improvement is attributed to the formation of higher number of density of Mg_2Sn precipitates. However, their hardening mechanisms are different each other. It is considered that the trace elements of Si, Ge and Ca suppress the growth of Mg_2Sn precipitates by reducing lattice mismatch between α-Mg and Mg_2Sn phase, while the trace elements of Ti and Zr encourage the increase of nucleation site of Mg_2Sn phase.

3:25 PM
Effects of Sb Additions on the Microstructure and Mechanical Properties of As-Cast Mg-5%Sn Alloy: *Ghazal Nayyeri*[1]; Reza Mahmudi[1]; [1]Tehran University

Mg-Sn based alloys have great potential for creep resistance because of the formation of thermally stable phase Mg2Sn in the as cast condition. In the present investigation, for further enhancement of the creep resistance 0.15, 0.4 and 0.7 wt.% of Sb was added to the base Mg-5%Sn alloy. The dendritic structure of the base alloy was refined after the addition of Sb, the effect being more pronounced in Mg-5%Sn-0.4%Sb. EDS analysis of the phases indicated that in addition to Mg2Sn, the new Mg-Sn-Sb rich particles are formed along grain boundaries. Impression creep tests were carried out at 175°C under constant stresses of 300 and 350 MPa. The Mg-5%Sn-0.4%Sb alloy had the lowest creep rates and thus the highest creep resistance among all materials tested. This is attributed to the higher volume fraction of Mg-Sn-Sb rich second phase precipitates which acts as the main strengthening agent in the investigated system.

3:45 PM Break

4:00 PM
Thermodynamic Modeling and Its Applications to Mg-Sn Based Alloy Development: *In-Ho Jung*[1]; D.H. Kang[2]; Nack J. Kim[2]; Woo-Jin Park[3]; SangHo Ahn[3]; [1]McGill University; [2]POSTECH; [3]RIST (Research Institute of Industrial Science and Technology)

Recently an Mg–Sn based alloy system has been investigated actively in order to develop new Mg alloys which have a stable structure and good mechanical properties at high temperatures. In the present study, thermodynamic modeling of the Mg–Sn–Al–Mn–Sb–Si–Zn system has been performed based on available thermodynamic, phase equilibria and phase diagram data. With the aid of the optimized database, unexplored complex phase equilibria/phase diagrams and Scheil cooling solidifications in multicomponent system can be readily calculated. It shows that the microstructural evolutions of Mg–Sn–Al–Zn alloys with additions of Si and Sb can be well explained by the thermodynamic calculations, which proves the applicability of thermodynamic calculations for new Mg alloy design. All calculations were performed using FactSage thermochemical software.

4:20 PM
Strengthening Mechanisms in Mg-Al-Sn Based Alloys: Shaul Avraham[1]; *Menachem Bamberger*[1]; [1]Technion

Intermetallic phases in Mg alloys determine the elevated temperature mechanical properties. Alloying elements dissolved in the α-Mg matrix, thermally stable intermetallics in the matrix and at the grain boundaries can hinder plastic deformation by solid solution strengthening, dispersion hardening and pinning of the grain boundaries respectively. Thermodynamics, X-ray diffraction and electron microscopy analysis indicate that the formation of γ-$Mg_{17}Al_{12}$, creep resistance deteriorating phase, is suppressed. The presence of different types of precipitates (Al-X) at the grain boundaries may serve as a source for grain boundary pinning. Nucleation of thermally stable intermetallics (Mg_2Sn) in the matrix can serve as obstacles for dislocation motion. The correlation between the microstructure evolution, hardening mechanisms and mechanical behavior will be discussed.

4:40 PM

Relation between the Microstructure and the Plastic Deformation Behavior in $Mg_{12}ZnY$ with the LPSO Structure: *Koji Hagihara*[1]; Akihito Kinoshita[1]; Yuya Sugino[1]; Naoyuki Yokotani[1]; Michiaki Yamasaki[2]; Yoshihito Kawamura[2]; Yukichi Umakoshi[1]; [1]Osaka University; [2]Kumamoto University

In the recent development of some high-strength Mg alloys, the role of $Mg_{12}ZnY$ strengthening phase with long-period stacking ordered (LPSO) structure is focused. Some ideas to explain the strengthening mechanism with the LPSO phase have been reported, but the detail is not enough clarified yet. Recently we investigated the plastic deformation behavior of 18R LPSO phase by using the directionally solidified single-phase crystals, and clarified that the (0001)<11-20> basal slip is dominantly operative in it. In compression at the loading orientation where the Schmid factor for the basal slip is negligible, on the other hand, the deformation proceeds accompanied by the formation of deformation kink. These results indicate that the LPSO phase exhibits strong plastic anisotropy. In this study, some LPSO single-phase alloys with different microstructures were prepared by casting, extrusion and heat-treatment under several conditions, and the variation in plastic deformation behavior depending on microstructure was examined.

5:00 PM

Effect of Rare Earth Elements Addition and T6 Heat Treatment on Creep Properties of Mg-Al-Zn Alloy: *Kaveh Meshinchi Asl*[1]; Farzad Khomamizadeh[2]; [1]Clemson University; [2]Sharif University of Technology

This paper focuses on creep properties of Mg-Al-RE and heat treated AZ91 magnesium alloy. The influence of heat treatment and rare earth elements addition on microstructure and mechanical properties were also investigated. The steady state creep rates were measured and for the AZ91 alloy, the results indicate a mixed mode of creep behavior, with some grain boundary effects contributing to the overall behavior. However for the cerium rich misch metal added samples, the sliding of grain boundaries was greatly suppressed due to morphological changes and the dislocation climb controlled creep was the dominant deformation mechanism at high temperatures. As a result, the grain boundaries were less susceptible for grain boundary sliding at higher temperatures. Effect of decreasing Al content on creep resistance of Mg-Al-RE alloys was also investigated. It was found that by decreasing the Al content to 4 wt%, the steady state creep rate was even decreased.

5:20 PM

Influence of RE Elements on Microstructure and Mechanical Properties of the quaternary Mg-Zn-Y-RE systems: *Jonghyun Kim*[1]; Yoshihito Kawamura[2]; [1]Kumamoto Technology & Industry foundation; [2]Kumamoto University

Magnesium alloys are known for their light weight and specific stiffness which are greatly attractive to the automotive and aerospace industries. However, the application of the Mg alloys is limited due to their lower mechanical properties. Recently, Kawamura et al. have developed the Mg-Zn-Y (at. %) alloys with LPSO phase. These alloys have the excellent mechanical properties. The investigation reported here focused on the influence of RE elements, which were also effective in strengthening Mg-Zn-Y alloys, on the microstructure and mechanical properties of the Mg-Zn-Y-RE alloys.The microstructure of the Mg96Zn2Y2-xREx alloys (RE= La, Ce, and Yb) with RE content in the range 0.1 to 1.0 at. % was composed of α-Mg, compounds and LPSO phases. However, Mg96Zn2Y1RE1 alloys (RE= Nd, Pr, and Sm) not detected LPSO phase. The tensile yield strength of Mg96Zn2Y2-xREx alloys with LPSO phase was higher than that of Mg96Zn2Y2-xREx alloys without LPSO phase.

5:40 PM

Changes in Microstructure and Mechanical Properties of Mg-Zn-Y Alloy with Long Period Stacking Ordered Structure during Annealing: *Masafumi Noda*[1]; Yoshihito Kawamura[2]; [1]Kumamoto Technology and Industry Foundation; [2]Kumamoto University

Changes in the mechanical properties and structure of extruded Mg-Zn-Y alloy with a long period stacking ordered structure on annealing at various temperatures were examined. The grain size of the extruded alloy increased to 9.4 μm on annealing between 473K and 773K for 3.6ks. However, the long period stacking ordered structure in the α-Mg matrix inhibited grain growth, and alloy annealed at 573K had similar mechanical properties to the extruded alloy, showing that the annealed alloy retained its strength.

Magnesium Technology 2009: Wrought Alloys

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

Program Organizers: Eric Nyberg, Pacific Northwest National Laboratory; Sean Agnew, University of Virginia; Neale Neelameggham, US Magnesium LLC; Mihriban Pekguleryuz, McGill University

Wednesday PM — Room: 2007
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Tyrone Jones, US Army Research Laboratory; Chamini Mendis, National Institute for Materials Science

2:00 PM Introductory Comments

2:05 PM

Age Hardening Response and Microstructures of ZK60 Alloy with Li Additions: *Chamini Mendis*[1]; Keiichiro Oh-ishi[1]; Kazuhiro Hono[1]; [1]National Institute for Materials Science

Mg-Zn based ZK60 alloy is a widely used wrought magnesium alloy that has a good combination of strength and ductility. However, only a small increment in strength is achieved by precipitation hardening due to the formation of coarse $MgZn_2$ rod-like precipitates parallel to $[0001]_{Mg}$. In this work, we have found that systematic additions of 1-3at%Li to the ZK60 alloy enhanced the age hardening response and the peak hardness is doubled compared to that of Li free alloy. TEM investigations have revealed that the improved age hardening is attributed to the refinement of the precipitates and the increase of their aspect ratio, thereby hindering the motion of basal dislocations. The precipitates remain $MgZn_2$ phase. The role of Li in increasing the aspect ratio and number density of precipitates will be discussed.

2:25 PM

High Temperature Deformation Behavior of Three Rolled Sheets of Magnesium Alloy AZ31: *Ravi Verma*[1]; Jon Carter[1]; Paul Krajewski[1]; [1]General Motors Corp

Magnesium AZ31 sheet alloys from three different sources, 2 DC (Direct-Chill) and 1 CC (Continuous-strip cast), were assessed for elevated temperature formability by tensile testing. Both monotonic and step-strain tensile tests were conducted at several different temperatures and strain rates. The paper discusses the relationship between initial sheet microstructure and tensile deformation behavior in terms of ductility to failure, strainrate sensitivity of flow stress, necking characteristics, and failure modes. The three alloys exhibit very different deformation behaviors, suggesting microstructural features in addition to the grain size influencing the deformation behaviors.

2:45 PM

Physical Metallurgy of Mg AZ80 Alloys for Forging Applications: Chris Sager[1]; Igor Yakubtsov[2]; William MacDonald[3]; *Scott Shook*[4]; Brad Diak[1]; Marek Niewczas[2]; [1]Queen's University; [2]McMaster University; [3]Canmet MTL; [4]Timminco Corp.

The Dow Chemical Company originally developed Mg AZ80 alloy in the 1950's for forging applications. The physical metallurgy and microstructural makeup of AZ series alloys can be quite varied and are dependent on alloy content, casting parameters, cooling rate, heat treatment, and thermomechanical processing. Early attempts to improve the ductility of this alloy focused on reduction of the as-cast grain size and volume fraction of brittle second phase particles. We have studied mechanical properties and the microstructure of Mg AZ80 alloys after different thermomechanical processing and assessed its suitability for the forging applications. The stability of secondary phases was evaluated in this alloy by modeling phase equilibria and examining its microstructure using a range of experimental approaches. The recrystallization behaviour of the alloy was studied to optimize homogenization treatments, deformation processing and the microstructure of the as-cast billets to achieve the full potential of AZ80 in applications for automotive wheels.

3:05 PM
Effects of Alloying Elements on Texture and Mechanical Properties of Extruded Mg-Zn-Al Alloys: *Jung Woo Choi*[1]; Ji Hoon Hwang[1]; Kwang Seon Shin[1]; [1]Seoul National University

Mechanical properties of magnesium alloys are significantly influenced by their microstructure and texture. In recent years, there have been numerous attempts to improve the mechanical properties by controlling microstructure and texture using different manufacturing processes. Little study, however, has been carried out to examine the effects of alloying elements on texture in magnesium alloys. In the present study, the changes in texture and mechanical properties were examined in the extruded magnesium alloys with different Zn and Al contents. The effects of Zn and Al on the texture of magnesium alloys were examined systematically using the X-ray diffraction method. The effects of Zn and Al on the mechanical properties were examined by tensile and compressive tests. Using these experimental results, simulations were conducted based on a visco-plastic self-consistent model in order to predict the changes in major deformation modes and textures during tensile and compressive tests.

3:25 PM
Formability of Magnesium Sheet: *Dietmar Letzig*[1]; Lenka Fuskova[1]; Kerstin Hantzsche[1]; Gerrit Kurz[1]; Sangbong Yi[1]; Jan Bohlen[1]; [1]GKSS Forschungszentrum

Processing of magnesium and its alloys such as sheet-rolling causes significant changes in microstructures, especially in their crystallographic texture. Further, these changes influence on the mechanical properties such as strength, formability as well as the anisotropic behaviour. Rolled AZ31 alloy sheets typically have a strong basal texture, which limits their formability especially at room temperature. On the other hand, aluminium-free magnesium alloys with additions of rare-earth-elements exhibit different crystallographic texture, in terms of main texture component and its strength. Thus, the improvement in sheet formability can be achived in rare-earth-elements added alloys, comparing to AZ31 alloy. In this study the formability of both AZ31 and ZE10 alloys at different temperatures is examined. Based on the present results, the influence of texture on the mechanical behaviour is discussed. It will be shown how the formability can be improved by weakening the crystallographic texture by means of alloy modification.

3:45 PM Break

4:00 PM
Application of Incremental Forming Technique to Mg-AZ31 Sheet: *Jong Park*[1]; [1]Hongik University

Magnesium alloy has a good strength-to-weight ratio. However, the material is so brittle that its application is limited to casting. Recently, as the formability of the material was found to be improved at warm temperatures, various sheet-metal forming techniques such as incremental forming and deep drawing at warm temperatures have been applied to this material. In the present study, the incremental forming technique was applied to Mg-AZ31 sheet in order to form various shapes, including cones, pyramids and curved surfaces. During forming, the sheet was continuously heated by hot-air blowers to keep the temperature to be consistent. As a result, these shapes were found to be successfully formed that were almost impossible by other forming techniques. Methodologies to utilize the material's formability, to overcome the forming limit and to compensate the springback were explored.

4:20 PM
Grain Size Effect on Hot Forging of Mg Alloys: *Yong Nam Kwon*[1]; [1]Korea Institute of Machinery and Materials

Magnesium alloys still have a lot of technical challenges to be solved for more applications. In the present study, effect of grain size on hot forging characteristics of Mg alloys using both cast and extruded forging stocks. For this purpose, three different Mg alloys such as AZ31, AZ61 and ZK60 were used. A general deformation behavior was gathered through a conventional compression test with the variation of strain rate and temperature. Both numerical and experimental works have been carried out on a model which contains both upsetting and extrusion geometries. Forgeability of magnesium alloys was found to depend greatly on grain size. Also, forging speed seemed to be more influential than temperature.

4:40 PM
Deformation Mechanisms in AZ31 Magnesium Alloy Tube Bending: *Wenyun Wu*[1]; Li Jin[1]; Shoushan Yao[1]; Alan Luo[2]; Anil Sachdev[2]; [1]Shanghai Jiao Tong University; [2]General Motors Research & Development Center

Abstract:This paper investigates the plastic deformation mechanisms of AZ31 magnesium alloy tubes under a rotary bending process at room and warm temperatures. The results suggest extensive twinning occurred at both intrados and extrados, leading to crack initiation and limited bendability at room temperature. With increasing bending temperature, less twinning is evident and the dislocation slip became more important in the bending deformation. When the bending temperature is too high, dynamic recrystallization occurs at the prior grain boundary and results in softening which leads to plastic instability and premature failure by cavity linkage.

5:00 PM
Elevated-Temperature Gas-Pressure Bulge Forming of Magnesium AZ31 Sheet: Theory and Experiment: *Eric Taleff*[1]; Ravi Verma[2]; Louis Hector[2]; Jung-Kuei Chang[1]; John Bradley[2]; Paul Krajewski[2]; [1]University of Texas; [2]General Motors R&D Center

Accurate prediction of strain fields and cycle times for fine-grained Mg alloy sheet forming at high temperatures (400-500°C) is limited by a lack of accurate material constitutive models. This paper details a first step toward addressing this issue by evaluating material constitutive models, developed from tensile data, for high-temperature plasticity of a fine-grained Mg AZ31 sheet material. The finite element method was used to simulate gas pressure bulge forming experiments at 450°C using four constant gas pressures. The applicability of the material constitutive models to a balanced-biaxial stress state was evaluated through comparison of simulation results with bulge forming data. Simulations based upon a phenomenological material constitutive model developed using data from both tensile elongation and strain-rate-change experiments were found to be in favorable accord with experiments. These results provide new insights specific to the construction and use of material constitutive models for hot deformation of wrought, fine-grained Mg alloys.

Materials for High Temperature Applications: Next Generation Superalloys and Beyond: Advanced Coatings II and Intermetallics

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS: High Temperature Alloys Committee, TMS: Refractory Metals Committee
Program Organizers: Joseph Rigney, GE Aviation; Omer Dogan, National Energy Technology Laboratory; Donna Ballard, Air Force Research Laboratory; Shiela Woodard, Pratt & Whitney

Wednesday PM — Room: 3010
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Brian Gleeson, University of Pittsburgh; Patrick Martin, Air Force Research Laboratory

2:00 PM Invited
High-Temperature, Environmental-Resistant Coatings for Current and Future Alloys: *Bruce Pint*[1]; [1]Oak Ridge National Laboratory

The implementation of current and new high-temperature materials is often hampered by their lack of oxidation or environmental resistance, particularly due to ever-increasing performance demands. Environmental-resistant coatings can increase service lifetime but often perform best on alloys with some inherent environmental resistance. So-called "prime-reliant" coatings that could prevent catastrophic degradation of substrates optimized for high-temperature strength are a noble research goal, however, there is considerable risk associated with inevitable coating defects and reliability in general. Further, other properties that high-performance coatings must demonstrate, such as chemical and mechanical compatibility with the substrates, assume greater importance as operating temperatures are increased. Examples are given for conventional alloys, superalloys and advanced materials in a variety of power generation applications.

2:25 PM Invited

Hot Corrosion Degradation of Alumina-Forming Coatings on Ni-Base Superalloys: *Gerald Meier*[1]; Frederick Pettit[1]; Brian Gleeson[1]; Michael Task[1]; [1]University of Pittsburgh

The components of gas turbine engines operating in many environments are susceptible to deposit-induced accelerated attack. In this study, relevant coatings and bulk alloys are being studied under conditions that are representative of Type I and Type II aggressive hot corrosion environments and cyclic oxidation. Experiments are also being performed to evaluate the effects of intermittent hot corrosion exposures on cyclic oxidation resistance of the alloys and coatings. The coatings and base alloys are relevant to current state-of-the-art systems. In order to examine the degradation of these systems without the complication of coating-substrate interactions, bulk alloys with the coatings compositions are also being studied. The effects of coating composition and phase distribution on the relative resistance to the various exposure environments and corresponding degradation mechanisms will be described.

2:50 PM

Industrially Prepared EQ Coating Systems for Advanced Ni-Base Superalloys: *Rudder Wu*[1]; Kyoko Kawagishi[2]; Kazuhide Matsumoto[2]; Hiroshi Harada[2]; [1]Imperial College London; [2]National Institute for Materials Science, Japan

"EQ coating" as a new coating system, has been introduced by NIMS-Japan in 2006. In this system, phases in thermodynamic equilibrium with the substrate (i.e. γ' phase) are used as coating materials to improve oxidation-resistance of substrates and suppress the formation of secondary reaction zone (SRZ).In our latest study, two EQ systems (standard and Pt-containing EQ) for the TMS-138A superalloy have been industrially prepared. The systems have further been coated by an electron beam-physical vapour deposited (EB-PVD) ZrO_2/7wt% Y_2O_3 (YSZ) top coat. Thermal-cycling experiments have shown that standard EQ systems can offer performances comparable to industrially used thermal barrier coatings (TBC). By the addition of Pt in the EQ system, the spallation-resistance of the YSZ coated system have been increased by approximately three times and exceed the majority of the current leading bond coat materials. Characterization of the coating cross-sections after spallation has confirmed the substrate-coating thermodynamic compatibility.

3:10 PM

Fluorine Treatment for Improved Adherence of Eb-Pvd Thermal Barrier Coatings on TiAl Alloys: *Alexander Donchev*[1]; Reinhold Braun[2]; Michael Schütze[1]; [1]DECHEMA; [2]German Aerospace Center

TiAl-alloys are promising candidates for high temperature applications in e.g. aero turbines. The capability of TiAl to withstand high temperature environmental attack is limited to temperatures of about 800°C. The fluorine effect is one possibility to enhance the oxidation resistance of TiAl alloys by forming a protective alumina scale. This scale can work as a bond layer for thermal barrier coatings (TBCs). To investigate the potential of the fluorine treatment in combination with ceramic YSZ-coatings (yttria stabilised zirconia) disk-shape specimens were treated with fluorine and preoxidised in air to form an Al2O3-scale. On these samples a ceramic YSZ top coat was deposited by electron-beam physical vapour deposition (EB-PVD). The oxidation resistance of these samples was studied performing cyclic oxidation tests for up to 2400 1-h cycles between 60°C and 900°C, 950°C or 1000°C in air. The TBCs exhibited good adhesion to the pre-treated specimens. Failure only occurred onthose areas which had not been protected by the F- treatment before the EB-PVD process.

3:30 PM

Resistance of Y_2SiO_5 to CMAS Degradation by Apatite Formation: *Kendra Grant*[1]; Stephan Kramer[1]; Carlos Levi[1]; [1]University of California, Santa Barbara

Environmental barrier coatings (EBCs) protect ceramic matrix composites (CMCs) from volatilization in high temperature combustion environments containing water vapor. Degradation of EBCs by siliceous deposits known as CMAS may limit the durability of these coatings. Yttrium monosilicate, a candidate EBC, has been shown to dissolve into molten CMAS and re-precipitate as a Ca:Y Apatite phase, with minor additional crystalline phases. The formation of a dense Apatite layer along the CMAS/EBC interface offers promise as a barrier to protect the underlying Y_2SiO_5 from further chemical reaction with the CMAS melt. These results are particularly significant in light of earlier findings that EBCs based on barium-strontium-alumino-silicate (BSAS) do not form a reaction product with similar protective potential, and are susceptible to CMAS penetration along grain boundaries to substantial depths below the interface. No such penetration has been observed in Y_2SiO_5. The observations, underlying mechanisms and implications for EBC durability are discussed.

3:50 PM Break

4:00 PM Invited

Development Pathways to Engineering "Beta Gamma TiAl" Alloys: *Young-Won Kim*[1]; Sang-Lan Kim[1]; Dennis Dimiduk[2]; Christopher Woodward[2]; [1]UES Inc; [2]AFRL

"Beta gamma" alloys are a new class of TiAl alloys that may offer improved processibility, refined microstructures, and increased strength without loss of ductility. The alloy design concept selected for the present investigations was to determine alloy compositions that are beta solidified but yield gamma- and beta-phase volume fractions of >85% and <5%, respectively, below 1000°C. Such alloys were found to exist within compositional ranges of Ti-(42-45)Al-(2-6)Nb-(1-6)(Cr, Mn, Mo, V)-(0.2-0.4)(B,C) in the proximity of the ternary phase field ($\gamma + \beta/B2 + \alpha/\alpha_2$). Employing various processing methods and analytical tools, together with phase diagram constructions, the results showed that this alloy system exhibits the desired attributes and improvements, at least for medium-scale ingot processing. However, there are numerous issues/challenges that need to be overcome for beta gamma alloys to become viable as high-temperature structural materials. This presentation reports our ongoing development pathways toward beta gamma materials technology, and discusses the challenges.

4:25 PM

Damage Evolution and Fatigue after Impact of the TiAl-Alloy TNBV3B: *Susanne Gebhard*[1]; P.W.M. Peters[1]; Dan Roth-Fagaraseanu[2]; Heinz Voggenreiter[1]; [1]German Aerospace Center; [2]Rolls-Royce Germany

TiAl-alloys show a low ductility, which limits their application especially in components prone to impact damage like turbine blades or vanes. Therefore, the influence of impact damage on the mechanical behavior of a cast and a forged TNBV3B alloy has been investigated. Ballistic tests were performed with impact energies up to several joules. In order to approach the impact situation with real blades or vanes, a blade-like geometry was chosen for the specimen edges. The caused damage has been evaluated as a function of the impact speed, the particle weight and the impact location. Fatigue experiments at different temperatures were performed to determine the threshold for crack growth as a function of the impact damage size. For these tests, specimens showing cracks with lengths up to some millimeters on the specimen back side as well as specimens showing other damages like blow-outs were chosen.

4:45 PM

Development of Ti-Al-Nb Intermetallic Alloys from Accumulative Roll Bonding and Reaction Annealing: *Peng Qu*[1]; Viola Acoff[1]; [1]University of Alabama

The TiAl intermetallic compound has long been considered as a next-generation structural material and a replacement for traditional Ni-based superalloy not only for its high oxidation resistance, creep strength and excellent mechanical properties at elevated temperatures, but also because of its low density and light weight. In this paper, a ternary Ti-46Al-9Nb intermetallic alloy (at. %) was produced by accumulative cold roll bonding (ARB) followed by reaction annealing. X-ray diffraction (XRD), scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS), and microhardness testing were used to characterize the phases that appeared after subjection to a two-stage annealing process. The diffusion mechanism for the first annealing stage was studied. The final lamellar structure that was obtained after the two-stage annealing process resulted in desirable properties.

5:05 PM

The Effects of Cooling Rates on Phase Transformation and Microstructural Evolution in Ti-44Al-4Nb-4Zr Alloy: *Hongwei Yang*[1]; [1]Delaware St Univ

Phase transformation and microstructural evolution has been studied in Ti-44Al-4Nb-4Zr-0.2Si-0.1B alloys that were cooled from the alpha + beta phase region with various cooling rates. It has been shown that the cooling rates have different influence on the morphology of the transformation products for three phase transformations studied, alpha -> alpha 2, B2 -> omega and alpha -> gamma. In furnace-cooled samples all three transformations are fulfilled completely, while in water-cooled samples, B2 -> omega is partially detained and a diffuse omega phase forms as metastable phase, and alpha -> gamma is completely suppressed, which support that the gamma lamellae formation is diffusion-controlled.

5:25 PM
Beyond Near-Gamma Alloys: Development of Gamma+Sigma Alloys: *Fereshteh Ebrahimi*[1]; Michael Kesler[1]; Sonalika Goyel[1]; Hans Seifert[2]; [1]University of Florida; [2]Freiberg University of Mining and Technology

The performance of aircrafts can be significantly improved by reducing the weight of their engines. Two-phase (γ-TiAl+α-Ti_3Al) near-gamma alloys with densities less than half of Ni-based superalloys are presently being developed but their application is limited due to their low creep resistance at high temperatures. We have developed alloys based on Ti-Al-Nb-X (X = Cr, Mo,W) system with γ-TiAl+σ-Nb_2Al microstructures, which exhibit superior creep resistance. One of the shortcomings of gamma phase is its low ductility. In the presents study ductility improvement is achieved through significant refinement of the gamma grains, whose sliding at high temperatures is inhibited by the presence of a fine distribution of the hard sigma-phase. In this presentation, microstructural evolution in alloys based on Ti-Al-Nb-X systems is discussed and the dependency of toughness on microstructural scale and volume fraction of phases is elucidated. The support by NSF/AFOSR under grant number DMR-0605702 is greatly appreciated.

Materials for the Nuclear Renaissance: Materials: Applications and Characterization

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS/ASM: Nuclear Materials Committee, TMS: Refractory Metals Committee
Program Organizers: Raul Rebak, GE Global Research; Robert Hanrahan, National Nuclear Security Administration; Brian Cockeram, Bechtel-Bettis Inc

Wednesday PM Room: 2009
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Brian Cockeram, Bechtel-Bettis Inc; Raul Rebak, GE Global Research

2:00 PM
Beryllium Use in Commercial Nuclear Reactors: *Edgar Vidal*[1]; [1]Brush Wellman, Inc.

Beryllium has unique nuclear properties that make it an attractive metal for use in commercial nuclear reactors. Beryllium has been extensively used in advanced test reactors around the world as both a moderator and reflector of neutrons. Published work report that the use of beryllium improves the "neutron efficiency" of power generating nuclear reactors, thus reducing the fuel enrichment requirements in light water reactors, and heavy water inventory in heavy water reactors. Beryllium in solid form, like many industrial materials, poses no special health risk if safe handling practices are followed.

2:20 PM
Hydride Redistribution and Delayed Hydride Cracking in Spent Fuel Rods during Dry Storage: *Young Suk Kim*[1]; [1]Korea Atomic Energy Research Institute

The aim of this work is to investigate the effect of thermal creep during vacuum drying of the spent fuel rods on redistribution of hydrides and their delayed hydride cracking (DHC) susceptibility. To this end, we analyzed Tsai's thermal creep results of irradiated Zircaloy-4 cladding segments from two pressurized water reactors and Simpson and Ells's observation where zirconium alloy cladding tube failed during a long-term storage at room temperature. On cooling the spent fuel rods, it is found that hydrogen moves from the peak temperature regions toward the cooler parts such as both ends of the cladding tubes, causing DHC cracks to grow there. When the spent fuel rods are cooled to below 180oC during their dry storage, this study demonstrates that according to Kim's DHC model spent fuel rods may fail by DHC only if the stressed regions with a higher tensile stress are present inside them.

2:40 PM
In-Situ Studies and Modeling the Fracture of Zircaloy-4: *Brian Cockeram*[1]; K. Chan[2]; [1]Bechtel Bettis Inc; [2]Southwest Research Institute

In-situ fracture studies were performed on non-irradiated Zircaloy-4 using tensile specimens and pre-cracked Compact Tension (CT) specimnes to clarify the mechanism for fracture initiation in the constrained and non-constrained state. Similar approaches have been reported in the literature to understand the role of hydrides on the fracture of Zircaloy-4, but hydride-free Zircaloy-4 has received little study. Both annealed and beta-treated Zircaloy-4 were tested in the longitudinal, transverse, and short-transverse orientations to study the role of microstructure and orientation. Unstable crack extension is shown to occur under plastic constraint by a process of void nucleation, growth, and coalescence initiating from the Laves phase particles in the microstructure. A micromechanical model is developed for ductile tearing by void growth and coalescence. Excellent agreement between the model and experiments are observed. Aspects of the fracture mechanism and model are discussed.

3:00 PM
Influence of Grain Boundary Character on Creep Void Formation in Alloy 617: *Thomas Lillo*[1]; James Cole[1]; Megan Frary[2]; Scott Schlegel[2]; [1]Idaho National Laboratory; [2]Boise State Univ

Alloy 617, a high temperature creep-resistant, nickel-based alloy, is in the process of being code qualified for the primary heat exchanger for the Next Generation Nuclear Plant (NGNP) which will operate at temperatures above those for materials listed in ASME Boiler and Pressure Vessel Code for nuclear power applications. Orientation imaging microscopy (OIM) is used to characterize the grain boundaries in the vicinity of creep voids that develop during high temperature creep tests terminated at relatively low creep strains (800-1000°C at creep stresses ranging from 20-85 MPa) so only the sites most prone to void formation are present. Grain boundary character of the boundaries comprising triple junctions, the primary location for creep void nucleation, is reported. Also, the grain boundary character of triple junctions that appear to be resistant to void formation, as indicated by the absence of voids during creep tests carried out to fracture, are surveyed.

3:20 PM
Precipitate Redistribution during Creep of Alloy 617: Scott Schlegel[1]; Thomas Lillo[2]; James Cole[2]; Sharla Hopkins[1]; Evan Young[1]; *Megan Frary*[1]; [1]Boise State University; [2]Idaho National Laboratory

The next-generation nuclear plant (NGNP) will require materials that can operate at very high temperatures (e.g., in heat exchangers). Alloy 617 is known for its high temperature strength and corrosion resistance; however, during creep, carbides that are supposed to retard grain boundary motion are found to dissolve and re-precipitate on boundaries in tension. To quantify the redistribution, we have used electron backscatter diffraction and energy dispersive spectroscopy to analyze the microstructure of 617 after creep testing. The data were analyzed with respect to location of the carbides (e.g., intergranular vs. intragranular), grain boundary character, and precipitate type (i.e., Cr-rich or Mo-rich). We find that grain boundary character is the most important factor in carbide distribution; some evidence of preferential distribution to tensile boundaries is also observed. If the role of grain boundary character on redistribution can be determined, materials could be engineered to have microstructures resistant to carbide redistribution.

3:40 PM Break

3:50 PM
Microstructural and Corrosion Characteristics of Austenitic Stainless Steels Containing Silicon: Peter Andresen[1]; Martin Morra[1]; Peter Chou[2]; *Raul Rebak*[1]; [1]GE Global Research; [2]Electric Power Research Institute

Austenitic stainless steels (SS) core internals components in nuclear light water reactors are susceptible to irradiation assisted stress corrosion cracking (IASCC). One of the effects of irradiation is the hardening of the SS and a change in the dislocation distribution in the alloy. Irradiation also alters the local chemistry of these austenitic alloys, for example in the vicinity of grain boundaries. The segregation or depletion phenomena at near grain boundaries may enhance the susceptibility of these alloys to environmentally assisted cracking (EAC). The objective of the present work was to perform laboratory tests in order to better understand the role of Si on microstructure, electrochemical behavior and susceptibility to EAC. Experimental results are presented for two main types of especially prepared stainless steels: (1) Type 304L SS + 1-5% added Si and (2) 12% Cr Steel + 5% added Si.

4:10 PM

On Processing and Orientation Effects on the Viscoplastic Constitutive Laws of Nanostructured Ferritic Alloys: Michael Salston[1]; *G. Robert Odette*[1]; Charles Eiselt[2]; Kurt Van Nugtren[1]; [1]University of California, Santa Barbara; [2]Forschungszentrum Karlsruhe

The creep strength of nanostructured ferritic alloys (NFA) is controlled by a high density of Y-Ti-O nanofeatures, dislocations and grain structures that depend on alloy composition and thermomechanical processing treatments. Extruded NFA have strong axial and weak transverse orientations. High temperature viscoplastic properties are evaluated for a range of NFA and different orientations using strain rate jump (SRJ) creep tests: a low strain rate is imposed until the stress reaches steady-state, exhausting the primary creep strain, followed by a series of increases in the imposed strain-rates and corresponding steady-state stresses. The creep data are fitted to a threshold stress model. The creep rates vary for the different NFA and orientations, but the threshold stresses are found to be significant fractions of the at-temperature yield stress. The NFA SRJ data are compared to those for 9Cr tempered martensitic steels (TMS) including constant stress creep data.

4:30 PM

Titanium Aluminides for Advanced Fission Plants?: *Wolfgang Hoffelner*[1]; Jiachao Chen[1]; Per Magnusson[1]; [1]Paul Scherrer Institute

Titanium aluminides are well accepted elevated temperature materials. In conventional applications their poor oxidation resistance limits the maximum operating temperature. Advanced reactor environments operate in non-oxidizing environments which could widen the applicability of these materials to higher temperatures. The behaviour of a cast gamma-alpha-2 TiAl in was investigated under thermal and irradiation conditions. Irradiation creep was studied in-beam using helium and proton irradiation. Thin strip samples of 100 micrometer thickness were investigated in a temperature range of 300-500 C under irradiation and significant creep strains were detected. At temperatures above 500 C thermal creep becomes the predominant mechanism. Thermal creep was investigated at temperatures up to 950 C with non-irradiated and irradiated material. No significant effect of sample geometry was detected. Irradiation induced damage and creep damage were studied with the transmission electron microscope. The results are compared with similar tests performed with a ferritic oxide dispersion strengthened material.

4:50 PM

Effect of Grain Boundary Engineering on Microstructure and Properties of Alloy 800H: *Lizhen Tan*[1]; Loic Rakotojaona[2]; Kumar Sridharan[1]; Todd Allen[1]; [1]University of Wisconsin; [2]Mines ParisTech

Grain boundary engineering (GBE) has been demonstrated as an effective technique to improve the properties of polycrystalline metals. This technique essentially increases the population of low-sigma coincidence site lattice boundaries (CSLBs) with sigma less than 29 and interrupts the connectivity of general/random boundaries, by means of a carefully designed thermomechanical processing route. GBE has been successfully applied to Incoloy alloy 800H in this study. The supercritical water exposure and cyclic air oxidation tests showed significant improvement in oxide scale integrity with limited oxide exfoliation on the GBE-treated samples. The tensile and impact tests showed enhancement in strength at room and elevated temperatures. The microstructural evolution including precipitates and dislocations induced by the GBE treatment has been characterized by transmission electron microscopy and atomic force microscopy. This study provides insights on the property improvements due to the microstructural evolution induced by the GBE treatment.

5:10 PM

Advanced Finite Element Flaw Growth Analysis of Stress Corrosion Cracks in Dissimilar Metal Butt Welds: *Aladar Csontos*[1]; David Rudland[2]; Do-Jun Shim[2]; [1]U.S. Nuclear Regulatory Commission; [2]Engineering Mechanics Corporation of Columbus

On October 13, 2006, Wolf Creek Nuclear Operating Corporation performed inspections on the pressurizer surge, spray, relief, and safety nozzle-to-safe end dissimilar metal (DM) welds. The inspection identified five circumferential indications in the surge, relief, and safety DM welds that were attributed to primary water stress corrosion cracking (PWSCC). These indications were significantly larger than previously seen in commercial pressurized water reactors. As a result of the initial U.S. Nuclear Regulatory Commission (NRC) flaw evaluation study, the nuclear power industry agreed to complete pressurizer nozzle DM weld inspections on an accelerated basis. The industry then conducted an advanced finite element analyses (AFEA) using more realistic assumptions to address NRC's concerns regarding the potential for rupture without leakage from circumferentially oriented PWSCC. This talk will dicuss the modeling approach and a comparison of the industry's AFEA results to the NRC's confirmatory AFEA research program.

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Advanced Materials for PEM Fuel Cells and Batteries - Session II

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Zi-Kui Liu, Pennsylvania State University

Wednesday PM — Room: 3005
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Xingbo Liu, West Virginia University

2:00 PM Introductory Comments

2:05 PM Invited

Atomic-Scale Structural and Compositional Characterization of Alloy Catalyst Particles for PEM Fuel Cell Cathodes: *Karren More*[1]; Lawrence Allard[1]; K. Reeves[1]; [1]Oak Ridge National Laboratory

High angle annular dark field (HAADF)-STEM (Z-contrast) imaging with sub-Å resolution is being used to image individual Pt-alloy catalyst particles having catalytically-relevant surface features, i.e., crystallographic/atomic ordering, surface faceting, surface 'skin' or 'skeleton' structures, and core-shell morphologies. In this study, several Pt-alloy catalysts, such as Pt-Co, Pt-Cr, Pt-Ti, and Pt-W, have been characterized by HAADF-STEM, high-resolution analytical TEM, and X-ray diffraction, in order to identify the crystallographic structures and predominant particle shapes and to correlate these observations with cathode durability and performance. Results from in-situ heating experiments of Pt-alloy catalysts, used to assess durability, will be discussed. Research sponsored by the U.S. Department of Energy, Office of Hydrogen, Fuel Cells, and Infrastructure Technologies Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. Research conducted at ORNL's SHaRE User Facility was sponsored by the Division of Scientific User Facilities, Office of Basic Energy Sciences, U.S. Department of Energy.

2:40 PM

A Novel Non-Platinum Group Electrocatalyst for PEM Fuel Cell Application: *Jin Yong Kim*[1]; K. Scott Weil[1]; [1]Pacific Northwest National Laboratory

Recent economic studies conducted indicate that cost of PEMFC stacks can be reduced dramatically by decreasing or eliminating the amount of platinum required in the cell electrodes; the largest quantity of which is employed in the cathode where it catalyzes the oxygen reduction reaction (ORR). While substantial progress has been made in understanding why platinum is such an effective catalyst for the ORR and in explaining the catalyst degradation mechanisms that currently limit the operational lifetimes of PEMFCs, less success has been achieved in identifying promising alternative electrocatalysts. We have recently completed a series of preliminary experiments on a composite material that shows promise as an alternative ORR electrocatalyst, in partial or full replacement of platinum. We will present the synthesis approach used in preparing the material, describe results from half-cell measurements, and discuss the reasons for the observed high catalytic activity based on current interpretations of supporting microstructural data.

3:00 PM Invited

Metallic Bipolar Plates for Direct Methanol Fuel Cells: *Christian Trappmann*[1]; Martin Mueller[1]; Juergen Mergel[1]; Detlef Stolten[1]; [1]Forschungszentrum Juelich

The results of the current research into design and development of novel metallic bipolar plates for direct methanol fuel cells (DMFCs) will be presented. The bipolar plate is one of the most important components of the fuel cell. In

conventional DMFCs the bipolar plates fabricated from graphite based materials are used. Since such plates mainly contribute more than 70% to the entire size and weight of the cell, the investigations into possibilities to utilize alternative metallic materials and advanced manufacturing methods are crucial for the reduction of the size and weight of the cell. The anodic and cathodic flow fields in the metallic bipolar plates recently developed at Forschungszentrum Juelich (IEF-3) are shaped into a 0.1mm thick foil material by a hydroforming process. Various ways to enhance the corrosion stability of the plates are discussed. To reduce the contact resistance full or partially coating of the active areas is utilized.

3:35 PM
Effect of Annealing on Microstructures of Nb-Clad 304LSS and Nb-Clad 434 SS for PEMFC Bipolar Plates: Sung-tae Hong[1]; *K. Scott Weil*[2]; Jung Pyung Choi[2]; [1]University of Ulsan; [2]Pacific Northwest National Laboratory

Two different Niobium (Nb)-clad stainless steels (SS) manufactured by cold rolling are currently under consideration for use as bipolar plate materials in polymer electrolyte membrane fuel cell (PEMFC) stacks. In the manufacturing process of Nb-clad SS, annealing was needed to reduce the springback induced by cold rolling. Two different annealing conditions were required due to the two different SS substrates. For Nb-clad 304L SS, the annealing developed an interfacial layer between the Nb cladding and the SS core and the interfacial layer plays a key role in the failure of the Nb-clad 304L SS as reported earlier. For Nb-clad 434 SS, the development of interfacial layer was insignificant even though the recovered ductility was similar. For Nb-clad 304L SS, the effect of interfacial layer on the bulk electrical resistance was investigated.

3:55 PM Break

Materials in Clean Power Systems IV: Clean Coal-, Hydrogen Based-Technologies, and Fuel Cells: Solid Oxide Fuel Cell Materials, Session I: Membranes, Electrodes, and Seals

Sponsored by: The Minerals, Metals and Materials Society, ASM International, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Corrosion and Environmental Effects Committee, TMS: Energy Harvesting and Storage Committee
Program Organizers: K. Scott Weil, Pacific Northwest National Laboratory; Michael Brady, Oak Ridge National Laboratory; Ayyakkannu Manivannan, US DOE; Z. Gary Yang, Pacific Northwest National Laboratory; Xingbo Liu, West Virginia University; Zi-Kui Liu, Pennsylvania State University

Wednesday PM Room: 3005
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Ayyakkannu Manivannan, US DOE; Xingbo Liu, West Virginia University

4:00 PM Invited
Reliability Prediction of SOFCs Anode Material Exposed to Fuel Gas Contaminants: Modeling & Experiment: Gulfam Iqbal[1]; Huang Guo[1]; *Bruce Kang*[1]; [1]West Virginia University

Solid Oxide Fuel Cells (SOFCs) operate under harsh environment which deteriorate anode material properties and reduce its service life. In addition to electrochemical performance, structural integrity of SOFCs anodes is essential for long-term operation. SOFCs anodes are subjected to stresses at high temperature, thermal/redox cyclic effects, and coal syngas contaminants. These mechanisms can degrade anode microstructure and decrease electrochemical performance and structural properties. In this research a anode material degradation model is developed and implemented in FE analysis. The model takes into account thermo-mechanical and coal syngas contaminants degradation mechanisms for prediction of long-term structural integrity of SOFC anode. The model will be validated using a NexTech ProbostatTM SOFC button cell test apparatus integrated with a Sagnac optical setup and infrared thermometer. The setup is capable of in-situ surface deformation and temperature measurement while measuring electrochemical performance of button cells under hydrogen or simulated coal syngas environment at operating conditions.

4:35 PM
First-principles Calculations and CALPHAD Thermodynamic Modeling of Defects in $La_{1-x}Sr_xCoO_{3-d}$: *James Saal*[1]; Venkateswara Rao Manga[1]; Mei Yang[1]; Zi-Kui Liu[1]; [1]Penn State University

To fully exploit the properties of $La_{1-x}Sr_xCoO_{3-d}$, a comprehensive and quantitative description of its defects is necessary. The parameters of the Gibbs free energy that make up thermodynamic models for defects in ionic systems are usually evaluated by fitting to experimental measurements, such as d. Such an approach reproduces experiments but is not always capable of uniquely describing the thermodynamic properties of the material. We report the progress in overcoming this difficulty by incorporating first-principles data, which allows us to predict not only measurable quantities but also properties that are difficult to determine experimentally, such as the valences of cobalt. This technology development has been supported in part by the U.S. Department of Energy under Contract No. DE-FC26-98FT40343. The Government reserves for itself and others acting on its behalf a royalty-free, nonexclusive, irrevocable, worldwide license for Governmental purposes to publish, distribute, translate, duplicate, exhibit and perform this copyrighted paper.

4:55 PM Invited
Development of Chromium Barrier Layers for Solid Oxide Fuel Cells: *Dilip Chatterjee*[1]; Samir Biswas[1]; [1]Corning Incorporated

Development of robust barrier layers is of prime interest for efficient operation of SOFC. High chromium content of ferritic stainless steels in electrolyte supported SOFC form gaseous oxides/hydroxides at the operating temperature and condense on various components of the stack, particularly on cathodes, resulting in performance degradation. Ideal barrier layer properties should include low diffusivity of chromium species in it, low thermal expansion mismatch of this layer with that of the substrate, durable at operating temperature, and most importantly, the preferred interface between the steel substrate and barrier layer should be continuous in nature. Primarily, two types of barrier layer were designed, produced and optimized for an electrolyte supported SOFC stack. These barrier layers were produced by diffusional and non-diffusional processes. This presentation will describe various barrier coatings, barrier properties provided by those coatings, and the transpiration measurements adopted to evaluate the efficiency of those coatings.

5:30 PM
Evaluation of Thermal Stresses in Intermediate and High Temperature Solid Oxide Fuel Cells as a Function of Thermo-Mechanical Properties of Conventional and Advanced Anode, Cathode and Electrolyte Materials: *T. Manisha*[1]; Miladin Radovic[1]; Nina Orlovskaya[2]; Beth Armstrong[3]; [1]Texas A & M University; [2]University of Central Florida; [3]Oak Ridge National Laboratory

Distribution of mechanical stresses in Solid Oxide Fuel Cells (SOFCs) is a complex function of geometry, temperature distribution, residual thermal stresses, external mechanical loads, etc. In the present work, we report on evaluation and distribution of thermal stresses as a function of thermo-mechanical properties of constituent materials at various temperatures for high temperature and intermediate temperature SOFCs. The materials studied include Ni-Y0.8Zr0.92O2 and Ni-Sc0.1Ce0.01Zr0.89O2, as anode materials, LaMnO3, (La0.7Sr0.3)0.98 MnO3, and La0.6Sr0.4Fe0.8Co0.2O3 as the cathode material and Yt0.08ZrO2, Sc0.1Ce0.01ZrO2, and Gd0.2Ce0.8O2 as the electrolyte materials. The thermo-mechanical properties namely coefficient of thermal expansion and elastic and shear moduli were determined in 25-900°C temperature range using thermal mechanical analyzer and resonant ultrasound spectroscope in nitrogen and air atmosphere respectively. These thermo-mechanical properties have been used to estimate and model the distribution of thermal stresses at different temperatures in the anode, electrolyte and cathode assembly of intermediate and high temperature fuel cells.

5:50 PM
Microstructure Design of Solid Oxide Fuel Cell Electrodes: Kei Yamamoto[1]; *R. Edwin Garcia*[1]; [1]Purdue University

The effects of connected porosity and functionally graded electrode material are analyzed for a typical YSZ|LSM SOFC cells. Optimal microstructure electrodes are proposed by identifying microstructural mechanisms that control the transport of oxygen in the porous cathode electrode. For the selected material parameters simulations show that a decrease in the spacing of interconnected porosity improves power delivery at moderate and high current densities. Mictrostructural mechanisms, such as self-induced starvation and the effect of the tortuousity of the LSM network to power generation are assessed.

Microstructures where LSM with engineered partcle density gradients and directly connected porosity networks are described.

Materials Processing Fundamentals: Aqueous and Liquid Processing

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

Wednesday PM Room: 2016
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Sutham Niyomwas, Prince of Songkla University

2:00 PM
A Study on the Mechanism of Magnetite Formation Based on Iron Isotope Fractionation: *Payman Roonasi*[1]; Allan Holmgren[1]; [1]Luleå University of Technology

Having knowledge of mechanism of magnetite formation is essential in a number of industrial processes including magnetite synthesis and corrosion of iron. In this study, magnetite nano-particle was synthesized via two different ways; coprecipitation of iron (II) and (III) and oxidation of ferrous hydroxide. The samples were characterized using X-ray diffraction (XRD), Mid-Far IR spectroscopy, scanning electron microscopy (SEM), chemical analysis for determination of FeII/FeIII ratio and ICP-MS for iron isotopic ratio ($^{56}Fe/^{54}Fe$) measurement. Since fractionation of iron isotopes depends on reaction rate and bonding strength, interpretation of the isotopic data with respect to the possible mechanisms is discussed. No fractionation of iron isotopes was observed for the magnetite synthesized by coprecipitation, whilst magnetite formed from ferrous hydroxide showed higher abundance of ^{54}Fe compared to ^{56}Fe in the beginning of reaction, implying the significance of the following reaction: $Fe(OH)_{2\,(solid)} <\!\!\longrightarrow [Fe(OH)]^{+}_{(aq)} + OH^{-}$.

2:15 PM
Kinetic Studies of Hydrochloric Acid Leaching Process of Ilmenite for Rutile Synthesis: Zengjie Wang[1]; *Jilai Xue*[1]; Haibei Wang[2]; Xunxiong Jiang[2]; [1]University of Science & Tech Beijing; [2]Beijing General Research Institute of Mining and Metallurgy

Kinetic study of hydrochloric acid leaching of Panzhihua ilmenite from China is presented. The leaching process is found to follow the spherical model f(a)=1-(1-a)1/3 and the apparent activation energy is 47.21 KJ/mol. The dissolving behaviors varied in different kinetic stages. At the beginning stage the dissolved amount of Fe and Ti increased monotonically with leaching time, while about 15 minutes later the dissolved Ti in the form of TiOCl2 began hydrolyzed into TiO2 powder reunited on the ilmenite ore surface, as observed by SEM and EDS. With the unreacted ilmenite particles covered by a layer of hydrolyzed TiO2, the leaching seemed as a selective leaching of iron and other impurities because Ti dissolved and hydrolyzed rapidly. The leaching process can end 4 hour later when most of Fe and other impurities have been dissolved and TiO2 remains as residues. The effects of acid concentration and leaching temperature were also investigated.

2:30 PM
Studies on the Anodic Dissolution Behavior of TiCxOy in Alkali Chloride Melt: *Xiaohui Ning*[1]; Hengyang Liu[1]; Hongmin Zhu[1]; [1]Beijing University of Science & Tech

The chemical and electrochemical anodic dissolution behavior of TiC_xO_y,which were prepared through carbothermic reduction of titanium dioxide,was investigated in alkali chloride melt. A mass spectrometer was used to on-line detect the anodic gas generated during the dissolution.The results showed that TiC_xO_y can dissolve by electrochemical way but not chemical dissolution. During the electrochemical dissolution, titanium dissolves as Ti^{2+} ion into the molten salt melt and carbon and oxygen form carbon oxide (CO, CO_2) simultaneously. The influence of ratio of O/C in TiC_xO_y on the dissolution behavior,as well as the gas component was also studied in detail.It is very interesting that the components of anodic gas changed with the change of ratio of O/C in TiC_xO_y.When the ratio of O/C is 1:1,the carbon monoxide was main production. And as the ratio of O/C in TiC_xO_y is more than 1, CO_2 was also detected.

2:45 PM
Synthesis and Chlorination of Titanium Oxycarbide: *Guangqing Zhang*[1]; Mohammad Dewan[1]; Andrew Adipuri[1]; Oleg Ostrovski[1]; [1]The University of New South Wales

Chlorination of titanium oxycarbide has an advantage over chlorination of titanium dioxide as it occurs at much lower temperatures. The paper presents results of a systematic study of synthesis of titanium oxycarbide and its chlorination.Titanium oxycarbide was produced by carbothermal reduction of titania in hydrogen, argon and helium. Formation of titanium oxycarbide started at 1200°C in all gases. The reduction was the fastest in hydrogen. Formation of titanium oxycarbide in hydrogen was close to completion in 120 min at 1300°C, 60 min at 1400°C and less than 30 min at 1500°C. Reduction in argon and helium progressed similarly and reached 90-95% after 300 min at 1400-1500°C. The chlorination of titanium oxycarbide was ignited at 150-200°C. Chlorine partial pressure and gas flow rate strongly affected the chlorination rate. Chlorination of titanium oxycarbide produced with carbon to titania molar ratio 2.5 at 235-400°C was close to 100% in 30 min.

Mechanical Behavior of Nanostructured Materials: Joint Session of Mechanical Behavior of Nanostructured Materials and Bulk Metallic Glasses VI: Mechanical Behavior of Nano and Amorphous Materials

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee
Program Organizers: Peter Liaw, The University of Tennessee; Hahn Choo, The University of Tennessee; Yanfei Gao, The University of Tennessee; Gongyao Wang, University of Tennessee; Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Wednesday PM Room: 3012
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Nathan Mara, Los Alamos National Laboratory; Julian Raphael, Columbus McKinnon

See page 283 for program.

Mechanical Behavior of Nanostructured Materials: Plasticity and Deformation Mechanisms at Small Length Scale III

Sponsored by: The Minerals, Metals and Materials Society, TMS Electronic, Magnetic, and Photonic Materials Division, TMS Materials Processing and Manufacturing Division, TMS Structural Materials Division, TMS: Chemistry and Physics of Materials Committee, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Nanomechanical Materials Behavior Committee
Program Organizers: Xinghang Zhang, Texas A & M University; Andrew Minor, Lawrence Berkeley National Laboratory; Xiaodong Li, University of South Carolina; Nathan Mara, Los Alamos National Laboratory; Yuntian Zhu, North Carolina State University; Rui Huang, University of Texas, Austin

Wednesday PM Room: 3024
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Nathan Mara, Los Alamos National Laboratory; Zhiwei Shan, Hysitron Inc

3:30 PM Invited
Creep, Superplasticity and Fracture Toughness in Nanocrystalline Ceramics: Dongtao Jiang[1]; Dustin Hulbert[1]; *Amiya Mukherjee*[1]; [1]University of California

A three-phase alumina based nanoceramic composite demonstrated superplasticity at a surprisingly lower temperature and at a higher strain rate. An alumina-carbon nanotube-niobium nanocomposite demonstrated fracture

toughness values that are three times higher than that for pure nanocrystalline alumina. It was possible to take advantage of both fiber-toughening and ductile-metal toughening in this investigation. A silicon nitride/silicon carbide nanocomposite, produced by pyrolysis and liquid polymer precursor, demonstrated one of the lowest creep rates reported so far in ceramics. This was primarily achieved by avoiding oxynitride glassy phase at the intergrain boundaries. One important factor in the processing of these nanocomposites was the use of electrical field assisted sintering method. This allowed the sintering to be completed at significantly lower temperatures and at much shorter times. These improvements in mechanical properties will be discussed in the context of results from microstructural investigations. Work supported by grants from ARO and ONR.

3:50 PM

Phase Transformation and Recrystallization during Creep of a Nanostructured Intermetallic TiAl Alloy: *Fritz Appel*[1]; Jonathan Paul[1]; Michael Oehring[1]; [1]GKSS Research Centre Geesthacht

The creep behavior of a novel type of nanostructured TiAl alloys with a modulated morphology has been investigated. The constitution and microstructure of the alloy result from decomposition reactions of the high-temperature ß/B2 phase. The characteristic constituents are structurally modulated laths that are comprised of several stable and metastable phases. Tensile creep tests have been correlated with characterization by high-resolution transmission electron microscopy. The creep behavior of the material is mainly limited by the early onset of tertiary creep at higher stresses and temperatures. The processes associated with this behavior are several phase transformations towards thermodynamic equilibrium, dynamic recrystallization and the relaxation of constraint stresses that exist between misfitting phases.

4:05 PM

Investigation of Creep Behaviour with a New Innovative Nanoindentation Tester: *Nicholas Randall*[1]; [1]CSM Instruments

Nanoindentation testing is particularly appropriate for creep and stress relaxation tests because it can measure materials whose properties are highly viscoelastic. However, the main drawback of nanoindentation tests is linked to the low thermal stability of most instruments. These instabilities introduce an uncontrollable penetration drift superimposed to the viscoelastic deformation of the sample. For some polymers thermal expansion of the instrument frame can be quite significant. The recent development of a new innovative instrument (the Ultra Nanoindentation Tester) has allowed such drawbacks to be avoided, and has allowed precise investigation of the creep behaviour of samples using very long duration tests. This results in almost complete elimination of the thermal drift in the measurement head. This study demonstrates that nanoindentation testing, when performed in good conditions with appropriate apparatus, constitutes a reliable tool to study the time dependent mechanical properties of materials.

4:20 PM Break

4:40 PM

Examining Nanomechanical Properties through Quantitative In Situ TEM Compression Testing: *Jia Ye*[1]; Raja Mishra[2]; Andrew Minor[1]; [1]Lawrence Berkeley National Laboratory; [2]General Motors R&D Center

In situ TEM nanocompression testing gives us insight into size effects in nanoscale volumes and also the ability to systematically measure the mechanical properties of small, well-defined single crystals. Using this technique, we have studied the origins of ductility in Aluminum alloys and the twinning process in Mg. In the first system, an AA6063 alloy was found to exhibit very different plastic deformation characteristics depending on the solute concentration in the matrix. For the second system, we studied Mg nanopillars. Due to their hexagonal structure, we will show that knowing the orientation of the Mg samples is particularly important. During in situ nanopillar compression testing the pure Mg demonstrated basal plane sliding and extensive twinning behavior. Importantly, our in situ technique allows for the stress state at the point of twin nucleation and during the progression of the twinning process to be measured directly.

4:55 PM

Modeling the In Situ TEM Deformation of CdS Nanospherical Shells: *Matthew Sherburne*[1]; Hillary Green[1]; D. Chrzan[2]; [1]University of California, Berkeley; [2]Materials Sciences Division, Lawrence Berkeley National Laboratory

Experiments performed by Shan et al. indicate that hierachically structured CdS nanospherical shells, composed of multiple nanograins, can be compressed up to 20% of their diameters before brittle fracture occurs. A finite element model was used to analyze the stress state within the shells; the shear stresses within the shell approach 2.2 GPa at the point of failure. The ideal shear stress for CdS was computed using density functional based total energy method. The computed ideal shear strength for CdS is 3.1 GPa. The stresses within the shell approach 71% of the ideal strength of the material. This unusual strength is attributed to the heirarchical structure of the nanospheres. Research supported by the Director, Office of Science, Office of Basic Energy Sciences (BES), of the US Department of Energy under Contract No. DE-AC02-05CH11231 and National Science Foundation under Grant No. DMR 0304629. Z. W. Shan et al., submitted for publication

5:10 PM

Systematic Study of Strain Rate Sensitivity of Nanostructured Pd Alloys Using Nanoindentation: *Insuk Choi*[1]; Ruth Schwaiger[1]; Anna Castrup[1]; Julia Ivanisenko[1]; Horst Hahn[1]; Oliver Kraft[1]; [1]Forschungszentrum Karlsruhe

Nanostructured metals have shown a strong strain rate sensitivity at room temperature. In this study, we performed nanoindenation tests with different strain rates to provide more quantitative and systematic understandings of the mechanisms behind this strain rate sensitivity by studying nanostructured metal alloys. Pd alloys with grain size ranging from a few nm to 150 nm were prepared, with Ag and Zr as alloying additions, by High Pressure Torsion (HPT), and by R.F. magnetron sputtering. For Pd based alloys, stacking fault energy varies with respect to the Ag content and is likely to affect deformation mechanisms. Furthermore, grain boundary sliding may be controlled by Zr, which segregates to the grain boundaries. For Pd-Ag alloys, the sensitivity becomes dramatically weaker with increasing Ag alloying content. Furthermore, the strain rate sensitivity itself was observed to be stronger for smaller strain rates indicating that different mechanisms are active in different strain rate regimes.

5:30 PM

Isostatic Pressing of a Nanocrystalline Al Alloy Powder: *Byungmin Ahn*[1]; Andrew Newbery[2]; Enrique Lavernia[2]; Steven Nutt[1]; [1]University of Southern California; [2]University of California, Davis

When consolidating nanocrystalline powder, it is important to obtain full densification without losing the beneficial microstructure. Although cryomilled Al alloy powder typically has very high thermal stability, the time at extended temperature and pressure during consolidation by hot isostatic pressing (HIP) results in moderate grain growth and reduction in the strength. In the work described in this paper, we carry out the isostatic pressing of cryomilled Al-5083 powder at lower temperatures, either by cold isostatic pressing (CIP) or by HIPping at low temperature. The effect of increasing CIP pressure was also investigated. The density and microstructure of the consolidated billets were characterized. A nearly 100% dense billet can be obtained at temperatures within the range of what is termed warm isostatic pressing. The resultant microstructure has much reduced grain size relative to material produced using previous HIP conditions. Mechanisms of hardening as well as microstructural development during processing were investigated.

5:45 PM

Mechanical Behavior of Fine-Grained Ductile Films on Polymer Substrates: *Megan Cordill*[1]; Gerhard Dehm[1]; F. Fischer[2]; [1]Erich Schimd Insitute of Materials Science; [2]Institute of Mechanics, Montanuniversitaet Leoben

Adhesion has been shown to be an important parameter in increasing the stretchability of flexible electronic devices. These devices are made on polymer substrates where the adhesion energies of the ceramic transistors and metal lines are difficult to determine. These interfaces need to be able to stretch as well as compress while maintaining good electrical conductivity. Common methods to measure adhesion energies of films on hard substrates (stressed overlayers, nanoindentation, and four point bend) cannot be easily implemented due to the compliance of the substrate. Cu films deposited onto polyimide are examined using an in-situ tensile test inside the scanning electron microscope to induce fracture and delamination of the films for measuring adhesion energies. Two interlayers, Cr and Ti, will also be studied to determine which increases the adhesion at the polyimide-metal interface.

6:00 PM

Hypersensitive Moisture-Assisted Debonding along Sol-Gel Coupled Oxide/Epoxy Interfaces: *Mark Oliver*[1]; Reinhold Dauskardt[1]; [1]Stanford University

Thin (~100nm) metal/epoxysilane sol-gel coupling layers exhibit excellent adhesive and cohesive fracture properties and have the potential to enable

new technologies that require high-performance epoxy/oxide interfaces. We demonstrate the existence of a new mechanism of moisture-assisted subcritical crack growth involving cohesive fracture of the sol-gel coupling layer wherein the crack growth kinetics exhibit a hypersensitivity to the moisture content of the environment at low growth rates. Rather than a threshold below which crack growth is dormant, persistence debonding is observed at growth rates below ~10 nm/sec. Existing models of moisture-assisted cracking, which have been successfully applied to numerous materials and interfaces, are unable to capture the observed behavior. A new model for moisture-assisted crack growth in hybrid organic-inorganic thin films will be proposed. Strategies for eliminating this behavior will be presented along with the implications of these findings for the reliability of sol-gel materials in general.

Nanocomposite Materials: Nanocomposites for Energy Conversion and Storage

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS Electronic, Magnetic, and Photonic Materials Division, TMS/ASM: Composite Materials Committee, TMS: Materials Characterization Committee, TMS: Nanomaterials Committee

Program Organizers: Jonathan Spowart, US Air Force; Judy Schneider, Mississippi State University; Bhaskar Majumdar, New Mexico Tech; Benji Maruyama, Air Force Research Laboratory

Wednesday PM — Room: 3020
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Terry Tritt, Clemson University; Michael Durstock, US Air Force

2:00 PM Introductory Comments

2:05 PM Invited

Nanostructured Materials for Energy Harvesting and Storage Devices: *Michael Durstock*[1]; [1]Air Force Research Laboratory, Materials and Manufacturing Directorate

The development of low-cost, lightweight, and flexible energy harvesting and storage devices are an enabling technology for many different types of applications. The fabrication of highly efficient conversion and/or storage devices with a high power and energy density, have yet to be achieved. In general, poor charge transport in organic and nanoparticle-hybrid devices is one of these factors and can result from low electronic charge carrier mobilities, relatively random thin film morphologies, and/or limited ionic intercalation and conduction pathways. Our efforts to address these issues for a variety of devices (including photovoltaics and battery electrode materials) include developing materials and fabrication methodologies that result in highly ordered structures to permit enhanced charge transport. This talk will discuss a number of these approaches including the fabrication and utilization of vertically aligned TiO2 and carbon nanotubes, as well as the assembly of mixtures of electroactive discotic liquid crystals.

2:30 PM

Electrochemical Lithium Storage of Li-Doped Titanate Nanotube: *Yi-Heon Jeong*[1]; Sun-Jea Kim[2]; Kyung Sub Lee[1]; [1]Hanyang University; [2]Sejong University

Li-doped titanate nanotubes were synthesized by hydrothermal lithium ion exchange processing from titanate nanotube precursor. To prepare the Li-doped titanate nanotubes, titanate nanotubes powder that had been treated with NaOH was mixed with LiOH aqueous solution and the resulting suspension was placed in a Ni-lined stainless-steel autoclave at 120°C for 24hrs, and subsequently fired at 100-500°C in vacuum to remove the hydrate in the nanotube. The electrochemical tests were performed by cycle voltammetry and galvanostatic method on a coin-type cell assembled with working electrode/separator/reference electrode (metallic lithium). The electrolyte solution was 1M LiBF4 dissolved in a propylene carbone(PC). Galvanostatic charge-discharge tests were performed at constant current density(10mA/g), with cutoff voltage of 1.0 to 3.0V. Systematic studies of effect of Li dopant and residual Na+ in the nanotube on lithium ion storage have been presented.

2:50 PM

Evaluation of Single-Wall Carbon Nanotube/Poly-(p-Napthaleneethynylene)-Based Composite Electronic Materials for Supercapacitor Applications: *Maria Abreu-Sepulveda*[1]; Mariem Rosario-Canales[2]; Pravas Deria[3]; Michael Therien[3]; Jorge Santiago-Avilés[2]; [1]University of Puerto Rico at Humacao; [2]University of Pennsylvania; [3]Duke University

Although substantial improvement in capacitance, energy densities, and discharge times has been achieved for redox supercapacitors over last several years, important challenges remain. These include high series resistance which limits the ability to quickly discharge these devices, excessive heat generation, and the coupled thermoelastic strain field which tends to deform the device materials. High stability composites based upon conjugated polymers that wrap single-wall carbon nanotubes (SWNTs) provide a platform to develop materials that can broadly impact these issues. This work discusses the preparation, characterization, and testing of new electrode materials based on polymer-wrapped SWNTs for the construction of supercapacitors. These polymer-wrapped SWNT composites exploit rigid, polyanionic poly(aryleneethynylene)s which provide unusual solubility and dispersion characteristics for carbon nanotubes in several solvents. Testing of the PNES/SWNT materials was done in sulfuric acid and potassium hydroxide aqueous solutions and in propylene carbonate-based solutions containing either tetrabutylammonium perchlorate or the ionic liquid 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide.

3:10 PM

Novel Activated Carbon Based Nanocomposites for Electrochemical Supercapacitors: *Prabeer Barpanda*[1]; [1]Rutgers University

Activated carbons are dominating electrode materials for commercial electrochemical supercapacitors. It majorly stores electrostatic charge via non-faradaic ionic double-layer formation at electrode-interface. The overall capacitance of activated carbons can be improved by modifying the surface morphology (specific surface area, porosity distribution etc) and improving the space charge capacitance in carbon. The current work is an attempt to combine these two routes to modify activated carbons, which has been achieved by in-situ gr-VII halides (iodine and bromine) doping via high-energy milling. The highly electronegative halides induce strong charge transfer reaction in carbon, thereby improving its electrical conductivity. As a result of halidation, a homogeneous carbon-halide nanocomposites is obtained which shows dramatic improvement in gravimetric (~100% rise) and volumetric capacity (~300% rise). The structural and electrochemical properties of these nanocomposites will be presented using a suite of techniques like XRD, DSC, BET, XRF, XPS, TEM, Raman spectroscopy and electrochemical measurements.

3:30 PM Break

3:45 PM Invited

Overview of Thermoelectric Properties of Bulk Nano-Composite Thermoelectric Materials: *Terry Tritt*[1]; Xiaohua Ji[1]; Jian He[1]; Bo Zhang[1]; Nick Gothard[1]; Paola Alboni[1]; Zhe Su[1]; [1]Clemson University

Recently, there has been an ever-increasing research effort on thermoelectric nanocomposite materials. Composites using a mixture of bulk thermoelectric materials with nanoparticles incorporated with the bulk are of specific interest. One of the main goals is to have additional design or tuning parameters for materials in order to manipulate and control the phonon scattering mechanisms, without significantly deteriorating the electrical transport properties. The ability to decouple the electron and phonon scattering mechanisms is very important in the development of higher efficiency thermoelectric (TE) materials, wherein the figure of merit, ZT, can be greater than unity. New opportunities are being explored in order to improve existing TE materials and significantly increase ZT. The role of phonons may be one of the most important parameters to understand in these novel materials. A review of recent results in nanocomposite TE materials from several research groups will be presented.

4:10 PM

ErAs Nanoparticles Embedded in (InGaAs)1-x(InAlAs)x for Thermoelectric Power Conversion: *Gehong Zeng*[1]; Hong Lu[2]; Je-Hyeong Bahk[1]; Ashok Ramu[1]; Arthur Gossard[2]; John Bowers[1]; [1]Electrical and Computer Engineering Department, University of California, Santa Barbara; [2]Materials Department, University of California, Santa Barbara

Erbium arsenide metallic nanoparticles are incorporated into InGaAlAs to create scattering centers for middle and long wavelength phonons, provide charge carriers, and form local potential barriers for electron filtering. The

thermoelectric properties of ErAs:(InGaAs)1-x(InAlAs)x were characterized by variable temperature measurements of thermal conductivity, electrical conductivity and Seebeck coefficient from 300 K to 800 K. The results show that the material's ZT is greater than 1.2 when the temperature is above 700 K. Generator modules of Bi2Te3 and ErAs:(InGaAs)1-x(InAlAs)x segmented elements were fabricated and an output power over 6 W was measured. Device modeling shows that the performance of thermoelectric generator modules can further be enhanced by the improvement of the thermoelectric properties of the materials, and reducing the electrical and thermal parasitic losses.

4:30 PM

Enhancement of Surface Morphology and Optical Properties of ZnO-Ag-ZnO Used as Transparent Conductive Thin Films: *Wen- Long Wang*[1]; Fei-Yi Hung[1]; S. J. Chang[2]; K. J. Chen[2]; Z. S. Hu[3]; [1]Institute of Nanotechnology and Microsystems Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University; [2]Institute of Microelectronics and Department of Electrical Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University; [3]Institute of Electro-Optical Science and Engineering, Center for Micro/Nano Science and Technology, National Cheng Kung University

Sputtered ZnO-Ag-ZnO films (100nm/30nm/100nm) on the quartz glass were used to investigate the effect of crystallized mechanism on their surface morphology, electrical properties and optical properties. The thin films were heated at 400ºC~500ºC for 1 hour in vacuum and in 6.9x10-1 Torr with pure O2. The analyzed results were also compared to investigate the interface structure, and the relation between oxygen atom concentration and optical characteristics. From XRD and FIB analysis, the ZnO-Ag-ZnO (ZAZ) thin films possessed the hexagonal structures and the Ag diffused layer was observed. After O2 annealed, the ZAZ not only raised the index of crystalline, but also reduced the electrical resistivity. Notably, increasing O2 concentration can improve the transparent effect of Ag diffused layer. For the PL spectra, both the interface structure and the index of crystalline increasing of ZAZ thin film made a strong UV emission band and a blue-shift.

4:50 PM

New WC-Co Electrode Materials with Additives of Al2O3 Nanopowder: *Sergey Nikolenko*[1]; Sergey Pyachin[1]; [1]Institute of Materials, Khabarovsk Scientific Centre, Far Eastern Branch, Russian Academy of Sciences

Hard alloys based on tungsten carbide with cobalt are used usually as electrode materials for the forming of strengthening coatings by electro-spark alloying (ESA). The ESA method can be improved using nanocrystalline materials. We have produced WC-8%Co alloys with 1-5% additives of Al2O3 nanopowder applied as a inhibitor of grain growth. Powders have been compacted at a pressure of 150 MPa. Sintering of compacted electrodes was performed in vacuum at temperature 1450°C for 60 min. The microstructure investigation of obtained alloys showed that Al2O3 additives from 1 to 5 wt% decreases the grain size of tungsten carbide from 2.5 to 1 micron. In our work, physicomechanical properties of coatings formed by ESA method with new electrode materials have been studied. The microhardness increased in 2 times and the wear resistance grown in 3-4 times in comparison with a coating produced using WC-Co alloy.

5:10 PM

Effect of Nb Addition on Magnetic Properties and the Microstructure of Fe3B / Nd2Fe14B Nanocomposite Permanent Magnets: *Junhua You*[1]; [1]Northeastern University

The microalloying effect of Niobium on the microstructure and magnetic properties of Fe3B / Nd2Fe14B nanocomposite permanent magnet has been investigated. As a result, Niobium addition stabilizes the amorphous phase and hinders the kinetics of the crystallization of the Fe3B particles. Niobium added in combination with Cu reduces grain size of Fe3B particles more remarkably than that without Niobium; with Niobium addition enhances magnetic properties of the alloy, but the amount must be suitable. Optimum magnetic properties with Br=1.15T, jHc=467kA/m and (BH)max=132.7kJ/m3 were obtained by annealing a melt-spun Nd5.5Fe70.0Co5Cu0.5Nb0.5B18.5 amorphous ribbon at 670°C for 40 min.

National Academies Propulsion Materials Study Community Town Hall Meeting: National Academies Propulsion Materials Study Community Town Hall Meeting

Sponsored by: National Academy of Sciences
Program Organizer: Erik Svedberg, National Academy of Sciences

Wednesday PM — Room: 3010
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Erik Svedberg, National Academy of Sciences

6:00 PM Town Hall Meeting

Near-Net Shape Titanium Components: Deformation and Machining Processes

Sponsored by: The Minerals, Metals and Materials Society, TMS: Titanium Committee
Program Organizers: Rodney Boyer, Boeing Company; James Cotton, Boeing Co

Wednesday PM — Room: 2010
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: John Fanning, TIMET

2:00 PM

Heat Treat and Cold Worked ATI-425® Properties: *John Hebda*[1]; [1]ATI Wah Chang

Early after the discovery that ATI-425® was cold workable, trials were also conducted to examine the heat treat response for strengthening via solution treatment and aging. While the alloy demonstrated capability for strengthening via STA, a full matrix of heat treat cycles and anneals had not been performed. This presentation examines the mechanical properties and microstructure from a matrix of four solution temperatures, three cooling rates, and six aging or annealing temperatures. The mechanical properties are compared to cold worked material in the cold worked state, various stress relief cycles, and fully annealed. In general, there exist a variety of opportunities to utilize ATI-425® to optimize strength and ductility.

2:20 PM

Hot Stretch Forming of Near Net Shape Titanium Profiles: *Martin Moffat*[1]; [1]Cyril Bath Company

The development of Hot Stretch Forming was motivated by the need to design and manufacture Titanium airframe structures for new aircraft with carbon fiber fuselage skins. These structures are contoured to fit against the inside radius of the fuselage curvature. By combining traditional stretch forming technology with hot metal forming techniques, the new technology of Hot Stretch Forming (HSF) was developed by the Cyril Bath Company. This new forming technology allows design engineers to develop a variety of Titanium structure profiles at reduced buyt to fly costs. The HSF technology is cost effective, repeatable, and available to be used for immediate production in volumes to meet aircraft build rates, now and in the future. The process saves both material and machining time; serious cost issues for today's aircraft budgets. The benefits of this process in controlling and minimizing residual stress allowing consistent machining will be discussed.

2:40 PM

Property Evolution of ATI 425®: From Ingot through Final Tubing: *Melissa Martinez*[1]; John Hebda[1]; [1]ATI Wah Chang

ATI 425® alloy (4Al-2.5V-0.2Fe-0.25O) is a cold workable titanium alloy showing similar properties to Ti 6Al-4V. Previous work on sheet has shown the alloy to have improved fatigue properties over alloys such as Ti 6Al-4V Super ELI and Ti 3Al-2.5V. The improved properties make the alloy a good candidate for a higher pressure hydraulic system with a reduced wall thickness and weight resulting in cost savings. Microstructure and mechanical properties are examined at various stages in processing from ingot to final sized hydraulic

tubing of ATI 425® alloy including: ingot breakdown, extrusion and rocking. Properties are compared to Ti 3Al-2.5V at various stages.

3:00 PM

Superplastic Formability of Ti-5Al-4V-0.6Mo-0.4Fe Alloy (TIMETAL®54M): *Yoji Kosaka*[1]; Phani Gudipati[1]; Vasisht Venkatesh[1]; [1]TIMET

Ti-5Al-4V-0.6Mo-0.4Fe alloy (Ti-54M) was developed at TIMET recently. The alloy exhibits superior machinability in most of machining conditions and strength comparable to that of Ti-6Al-4V. This is believed to be due to its lower flow stresses at elevated temperatures. The alloy has commercially been produced with Electron Beam Single Melt process for automotive forgings applications. Since the beta transus of Ti-54M is lower than Ti-6Al-4V, and the alloy contains iron, a fast diffuser, it is of interest to examine superplastic formability of the alloy. This paper will introduce and discuss preliminary results of SPF evaluation of Ti-54M sheets produced in a laboratory scale.

3:20 PM

A New Method of Laser Milling of Titanium for Rapid Manufacture of 3D Parts: *Gareth Littlewood*[1]; Lin Li[1]; Zhu Liu[1]; Malcolm Ward-Close[2]; [1]University of Manchester; [2]QinetiQ

A new method of rapid manufacture of near net shape Ti components has been developed based on laser ablation. The majority of current methods of rapid manufacture using lasers are based on additive techniques in which a part is built up layer-by-layer. The new method is faster than the current additive laser based rapid manufacture techniques and uses lower laser powers than the current laser milling processes. This is achieved through the choice of experimental conditions and laser parameters. Thin walls and delicate structures have been demonstrated which are possible as the process is non-contact and deep, high aspect ratio holes have also been produced which have can be difficult to produce by mechanical rapid manufacture techniques. This raises the possibility of application of the technique in the areas of medical and aerospace component manufacture.

3:40 PM

Machining Advantages of Ti-54M: *Rodney Boyer*[1]; James Cotton[1]; Stacey Nakayana[2]; John Fanning[2]; Megan Harper; [1]Boeing Co; [2]TIMET

TIMET has developed an alloy referred to as 54M (Ti-5Al-4V-0.6Mo-0.4Fe) which has demonstrated the potential for a significant advantage in the cost of machining over Ti-6Al-4V, and has properties slightly lower than those of Ti-6Al-4V, but comparable. TIMET first noted an advantage in the number of holes which could be drilled with a single tool in comparison to Ti-6Al-4V. Boeing has studied the milling characteristics of the alloy and found a noteworthy advantage in this area for some machining modes. The mechanism(s) behind this improvement are not well understood, but are assumed to include such factors as changes in microstructure, flow and fracture stresses, and chemistry; these will be discussed.

Neutron and X-Ray Studies of Advanced Materials: Advanced Imaging and Bio-Inspired Studies

Sponsored by: The Minerals, Metals and Materials Society, TMS Structural Materials Division, TMS/ASM: Mechanical Behavior of Materials Committee, TMS: Advanced Characterization, Testing, and Simulation Committee, TMS: Titanium Committee
Program Organizers: Rozaliya Barabash, Oak Ridge National Laboratory; Yandong Wang, Northeastern University; Peter Liaw, The University of Tennessee; Jaimie Tiley, US Air Force

Wednesday PM — Room: 3016
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Emil Zolotoyabko, Technion-Israel Institute of Technology; Yang Ren, Argonne National Laboratory

2:00 PM Invited

The Structure of Natural Bio-Composites: Combined X-Ray and Neutron Diffraction Study of Mollusk Shells: *Emil Zolotoyabko*[1]; [1]Technion-Israel Institute of Technology

Formation of natural bio-composites with superior characteristics attracts growing attention of researchers focusing on deeper understanding and potential mimicking of biomineralization. Organic macromolecules, supplied by organisms, play a crucial role in this sophisticated "processing". Our recent studies using high-resolution x-ray and neutron powder diffraction showed that the interaction between organic substance and ceramic mineral influences even the atomic structure of biogenic crystals. Specifically, we found that the unit cells in both mollusk-made aragonite and calcite are anisotropically distorted as compared to their geological counterparts. These distortions are the result of local forces imposed on mineral crystallites by intra-crystalline organic macromolecules. These forces also influence atomic positions in biogenic aragonite and calcite and, hence, atomic bonds, which are probed by neutron diffraction. For example, we found significant changes in the aplanarity of carbonate groups in biogenic aragonite, which are well correlated with frequency shifts in the Raman spectra.

2:20 PM Invited

Diffracted X-Ray Tracking (DXT) for Super Accurate Dynamic Observations of Single Molecular Motions: *Yuji Sasaki*[1]; [1]SPring-8/JASRI, JST/CREST Sasaki-Team

Recent progress in in-vivo or in-vitro observations of individual single protein molecules has been achieved with several single-molecular techniques and systems. However, it is difficult to measure intramolecular structural changes of single proteins molecules using visible lights due to the lack of monitoring precision and stability of the signal intensity in physiological conditions. One of the methods for the improvement of the positional decision accuracy is to shorten wavelength, for example, X-rays, electrons, and neutron. Recently, we succeeded picometer-scale slow Brownian motions of individual protein membranes (Bacteriorhodopsin (BR) and Potassium channel KcsA) in aqueous solutions from time-resolved Laue observations. In this single molecular detection system with X-rays, which we call Diffracted X-ray Tracking (DXT), we observed the rotating motions of an individual nanocrystal, which is labeled to the specific site in individual protein molecules.

2:40 PM Invited

Monte Carlo Simulation Study of Diffuse Scattering in $Pb(Zr,Ti)O_3$ (PZT): *T. R. Welberry*[1]; R.L. Withers[1]; K.Z. Baba-Kishi[2]; [1]Australian National University; [2]The Hong Kong Polytechnic University

Transverse polarized diffuse streaks have been observed in diffraction patterns of $Pb(Zr_{1-x}Ti_x)O_3$ ceramics for compositions ranging from x=0.3 (rhombohedral phase) to x=0.7 (tetragonal phase) including the important MPB region (x = 0.48). The streaks correspond to diffuse planes of scattering in 3D and these are oriented normal to the (cubic) $<111>_c$ directions. A Monte Carlo (MC) model has been developed that convincingly reproduces the observed diffraction patterns. In this the displacements of Pb ions running in chains along each of the $<111>_c$ directions are directed along the chain and are strongly correlated from cell to cell. There is no evidence of lateral correlation. Neighbouring chains are essentially independent. At this stage it is not clear what role the local order revealed by the scattering might play in governing the exceptional piezo-electric properties of the material but its presence requires the currently accepted models for the average structure to be reassessed.

3:00 PM Invited

High-Density Resolution Microtomography Using Synchrotron Radiation for Materials Science Applications: *Felix Beckmann*[1]; [1]GKSS-Research Center Geesthacht

The GKSS-Research Center Geesthacht is operating the user experiments for microtomography using synchrotron radiation at DESY, Hamburg, Germany. At the GKSS HARWI II wiggler beamline at DORIS III an intense and large X-ray beam in the photon energy range from 20 to 250 keV is available. Tomograms with high spatial resolution together with high density resolution are now routiney be obtained. Several applications for the characterizing new materials for leight weight construction and new welding techniques will be shown. Furthermore, the perspectives for micro- and nanotomography at the new undulator beamlines the imaging beamline (IBL) and the high energy materials science beamline (HEMS) at the new PETRA III storage ring will be given.

3:20 PM Invited

Mapping 3D Shape, Orientation and Strain State of Individual Grains in Polycrystals by X-Ray Diffraction Contrast Tomography: *Wolfgang Ludwig*[1]; Andrew King[2]; Peter Reischig[3]; Sabine Rolland du Roscoat[3]; Erik Lauridsen[4]; [1]Mateis, CNRS; [2]Manchester University; [3]ESRF; [4]Technical University of Denmark

X-ray diffraction contrast tomography (DCT) is a synchrotron based imaging technique that enables the mapping of 3D shape, orientation and the elastic strain

state of individual grains in the bulk of polycrystalline materials. The sample is illuminated using a monochromatic X-ray beam and both the direct and the diffracted beams are captured simultaneously on a single detector positioned closely behind the sample. The scanning and analysis procedure are based on the acquisition of Friedel pairs of diffraction spots - a concept enabling the implementation of robust indexing and accurate strain determination procedures. The shapes of the individual grains are obtained by tomographic reconstruction from the observed diffraction spots, employing algebraic reconstruction techniques (ART). The talk will illustrate the current possibilities and limitations of the technique with the help of some selected applications, including the analysis of stress-corrosion and fatigue crack propagation, grain growth and elastic deformation in metallic alloys.

3:40 PM Break

3:50 PM
X-Ray Micro-Tomography Imaging of Crack Propagation in Biological Samples Using an In Situ Mechanical Testing Device: *Holly Barth*[1]; Alastair MacDowell[2]; Robert Ritchie[1]; [1]University of California, Berkeley; [2]Lawrence Berkeley National Laboratory

Many extrinsic mechanisms are activated in a biological structure during crack growth on the length scale of a micrometer. The x-ray microtomography beamline at the Advanced Light Source allows for non-destructive three-dimensional imaging at this length scale. The process takes 2D projections and through a filtered back projection algorithm the images are reconstructed into a 3 dimensional image, which is representative of the sample's absorption. Using this technique the crack path is visualized as it interacts with the biological sample's microstructure. However, to really visualize the interactions it is important to do the crack growth in real time. The in situ mechanical testing device allows for the stable crack growth of these specimens as the tomographic 3-dimensional imaging is being done using either three point bending or tension. The specimens observed in the in situ device have included human cortical bone, bovine cortical bone, and dentin.

4:05 PM Invited
Properties of Dislocation Microstructures during Deformation under Single Slip: *Patrick Veyssiere*[1]; [1]LEM, CNRS-Onera

In a first part of this talk, effects of test temperature on dislocation organization will be examined in fcc-based ordered alloys such as TiAl and Ni3Al. It will be shown that TEM provides unique information on dislocation behavior that would remain unnoticed under indirect diffraction methods. The second part will be devoted to dislocation self-organization into entanglements. The property that dislocations of a given slip system are able to spontaneously engender obstacles to their own propagation has been the object of constant attention since the early 1950's. Entanglements are believed to stem from the sweeping of prismatic loops by mobile dislocations. Little is known though on the origin and organization of the prismatic loops and on the mechanism by which stable multipolar entanglements are formed. The present investigation concentrates on selected aspects related to self-organization under single slip in an effort to clarify its various constituents.

4:25 PM Invited
Study of Biomineralization of Fish Bone Using X-Ray Diffraction Microscopy: Huaidong Jiang[1]; Changyong Song[2]; Kevin Raines[1]; Rui Xu[1]; Bagrat Amirbekian[1]; Yoshinori Nishino[2]; Tetsuya Ishikawa[2]; *Jianwei Miao*[1]; [1]University of California, Los Angeles; [2]RIKEN SPring-8 Center

X-ray diffraction microscopy is a newly developed imaging modality that extends the methodology of X-ray crystallography to allow the structural determination of noncrystalline specimens. Herein, the biomineralization and development of 3D architecture of Alewife herring bone were investigated by using X-ray diffraction microscopy. To study the spatial relationships of mineral crystals to the collagen matrix of the fish bone, we performed nanoscale imaging of the mineral crystals inside collage fibrils at different stages of mineralization. The origin and distribution of mineral crystals inside the collagen matrix has been identified. Based on the experimental results, we proposed a dynamic structural model of bone to account for the nucleation and growth of mineral crystals in the collagen matrix with maturation of bone. These results will not only contribute to understand the biomineralization mechanism of fish bone, but also provide important design principles for hard tissue engineering and the development of biocompatible materials.

4:45 PM
The EDXRF Analysis of Components in Electric and Electronic Instruments in Accordance with RoHS Standard: *Denis Negrea*[1]; Catalin Ducu[1]; [1]University of Pitesti

The use of Cd, Pb, Hg and hexavalent Cr in electric and electronic products is limited within the framework of the RoHS directives. The limiting value for Cd is 100 mg/kg, for Hg, Pb, hexavalent Cr and the brominated flame retardants PBB and PBDE is 1000 mg/kg. The values must be below these limits for all components in an instrument, making regular monitoring necessary. An electronic circuit board was mapped using an EDXRF spectrometer and a Fundamental Parameters algorithm. Based on the obtained map, a point scan was done for Cd, Pb, Hg, Cr and Br in the highest intensity spots in order to obtain their highest concentrations within the analyzed sample area. In the end it was verified the compliance of the circuit board subjected to the analysis with the RoHS standard.

5:00 PM
An Analysis of Solidification Porosity in Atomized Al-Cu and Al-Fe Powders: Stephane Ablitzer[1]; Jon Johansson[1]; Denise Thornton[1]; Maryia Maizlin[1]; *Hani Henein*[1]; [1]University of Alberta

Porosity is a defect encountered in numerous casting operations. There are numerous efforts still underway to model this porosity formation. However, porosity in castings can occur due to numerous causes such as solidification shrinkage or poor feeding. Experimental data of solidification shrinkage porosity has not been easily available to date. X-ray tomography using synchrotron radiation with 0.27 micron beam resolution at the European Synchrotron Radiation Facility was used to generate three-dimensional (3-D) images for rapidly solidified Al-Cu and Al-Fe atomized droplets. The 3D image of the solidified droplets was observed and the porosity in these droplets were isolated. The volume fraction, interconnectivity and position of the porosity in the droplets was analyzed. The effect of gas in the atomizing chamber as well as particle size and alloy chemistry will be discussed.

5:15 PM Invited
High-Energy Synchrotron X-Ray Diffraction for Materials Research under Complex Sample Environments: *Yang Ren*[1]; [1]Argonne National Laboratory

The availability of synchrotron photons generated in high-flux and with energies much greater than 60 keV has significantly advanced the field of materials research because of the great penetration and low absorption. Obtaining high angular resolution for high-energy x-ray scattering provides still further research opportunities, especially in the study of bulk samples for both fundamental research and practical applications. Here we present a user facility, at the APS high-energy x-ray beamline 11-ID-C, where we combine a 2D and a point detector for both rapid and high-resolution x-ray diffraction studies. Both powder and single crystal samples can be studied in confined and complex sample environments with combined tunable external parameters (for example, combinations of temperature, pressure, stress, magnetic and electric fields). We will present the technical details and scientific research opportunities for this facility, as well as some recent results obtained with this instrument in different research areas.

5:35 PM Invited
Diffuse Scattering from Molecular Crystals: *Darren Goossens*[1]; [1]Australian National University

Diffuse scattering is a probe of the local ordering in a crystal, whereas Bragg peaks are descriptive of the average long-range ordering. This long-range average is made up of numerous local configurations whose population cannot be determined from analysis of the Bragg peaks. Diffuse scattering allows examination of this population and so offers a way to look at crystal structures in great detail. This is particularly the case when making use of the three-dimensional distribution of diffuse scattering. However, diffuse scattering is very weak and broad and often of similar intensity to experimental background, so data collection is demanding. Further, disorder can take on many forms and local configurations are not constrained by the average crystallographic symmetry. Here, the modelling of diffuse scattering from molecular crystals will be discussed, with particular reference to an X-ray study of pentachloronitrobenzene ($C_6Cl_5NO_2$) and a neutron diffraction study of *para*-terphenyl, $C_{18}H_{14}$.

Pb-Free Solders and Emerging Interconnect and Packaging Technologies: 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: Sung Kang, IBM Corp; Iver Anderson, Iowa State University; Srinivas Chada, Medtronic; Jenq-Gong Duh, National Tsing-Hua University; Laura Turbini, Research In Motion; Albert Wu, National Central University

Wednesday PM
February 18, 2009

Room: 2020
Location: Moscone West Convention Center

Session Chairs: Carol Handwerker, Purdue University; Fu Guo, Beijing University of Technology

2:00 PM Invited

Nucleation Control of Near-Eutectic Sn-Ag-Cu+X Solder Joints by Alloy Design: *Iver Anderson*[1]; Jason Walleser[1]; Joel Harringa[1]; Alfred Kracher[1]; [1]Iowa State Univ

Selecting a general purpose Sn-Ag-Cu (SAC) reflow solder remains a quandry. Instead of a eutectic microstructure found in Sn-Pb, the difficulty of Sn nucleation in typical SAC joint solidification promotes increased undercooling and Sn dendrites in normal reflow or, in slow cooling (e.g., BGA reflow), pro-eutectic Ag3Sn "blades" can form that may embrittle joints. SAC alloy designs with low Cu (0.5 wt.%) and very low Ag, down to 1%Ag (SAC105) have been proposed to promote Sn nucleation and inhibit nucleation of Ag3Sn, but this results in higher melting (226C liquidus) and decreased strength. Alternatively, this work focused on avoiding Ag3Sn blades with near-eutectic SAC3595 solder (220C liquidus) by promoting Sn nucleation with 4th elements (X=Zn, Mn, Al, and Fe) at low concentrations (<0.21%). These were selected by alloy design rules to form pro-eutectic intermetallic "seeds" that can reform during multiple reflows. Supported by Nihon-Superior through Ames Lab (contract no. DE-AC02-07CH11358).

2:20 PM

A Novel Solder Based on Metallic Nanoparticle Inks for Low-Temperature Packaging Technology: *Teymur Bakhishev*[1]; Vivek Subramanian[1]; [1]University of California, Berkeley

A novel solder technology based on gold (Au) nanoparticle ink was investigated. Inks consisted of surfactant passivated nanoparticles dissolved in a solvent. Optimized gold inks are able to sinter at temperatures as low as 120°C and achieve conductivities of up to 70% of bulk. Once sintered, the metallic structure reverts to bulk-like properties and approaches bulk reliability and performance. Thus nanoparticle-bases solders would operate at much lower homologous temperatures as compared to alloy-based solders. Nanoparticle inks under investigation were sintered at 180°C. These inks showed no significant contact resistance due to surfactant or solvent contamination. Electromigration studies were carried out and time to failure was investigated as a function of temperature. Electromigration activation energy was calculated through Black's equation to be 0.52eV, which is consistent with grain boundary diffusion. Nanoparticle inks are promising candidates as next-generation solders.

2:35 PM

Utilizing the Thermodynamic Nanoparticle Size Effects for Low Temperature Pb-Free Solder Applications: *John Koppes*[1]; Kevin Grossklaus[1]; Anthony Muza[1]; R. Rao Revur[2]; Suvankar Sengupta[2]; Hong-Sik Hwang[3]; Eric Stach[1]; Carol Handwerker[1]; [1]Purdue University; [2]MetaMateria Partners; [3]Indium Corporation of America

Development of lead-free solders with melting temperatures near traditional eutectic Pb/Sn solder is needed to limit damage to heat sensitive microelectronic components and devices. A possible method for reducing the melting temperature of Pb-free solders is by using nanoparticle solder pastes, in which the nanoparticles melt at temperatures far below their bulk counterparts due to the thermodynamic size effect. Particles with 5 nm diameters, observed to melt at temperatures below traditional Pb/Sn solder, are combined with flux (organic rosin used to prevent and remove oxide layers) to produce nanosolder pastes. The prototype nanosolder pastes display nanoparticle coalescence, in some cases to sizes with "bulk" melting temperatures, as characterized by differential scanning calorimetry (DSC).

2:50 PM

Novel Liquid Phase Sintered Solders for Next Generation Thermal Interface Materials and Interconnect Applications: Praveen Kumar[1]; C Nagaraj[2]; *Indranath Dutta*[1]; Rishi Raj[2]; Mukul Renavikar[3]; [1]Naval Postgraduate School; [2]University of Colorado; [3]Intel Corporation

At present, there is significant interest in low-temperature solders for thermal interface material and interconnect applications for packaging thermally sensitive next-generation devices. In this paper, we report on the processing and characterization of novel liquid phase sintered solders (LPSS), based on the Sn-In and Cu-In systems. The LPSS microstructure comprises grains of a majority high melting phase which is highly thermally and electrically conductive, and a minority low melting phase which gives the solder very high mechanical compliance. For the Sn-In system, solders with flow stresses close to that of In were obtained with 30-50 vol.% In, along with electrical and thermal conductivities which are about half those of In. Sintering characteristics, artifacts, and property evolution as a function of material parameters will be discussed. Preliminary results on the Cu-In system will also be reported. Finally, a phenomenological model for the thermal conductivity of the LPSS will be presented.

3:05 PM

Measurement Method of Liquidus Temperature of Lead-Free Solder Using Differential Scanning Calorimetry Curves: *Hiroshi Nishikawa*[1]; Yoshihito Hamada[2]; Tadashi Takemoto[1]; [1]Osaka University; [2]Uchihashi Estec Co., Ltd.

In general, liquidus temperature of lead-free solder is measured using cooling curve of differential scanning calorimetry (DSC). However, small amount of crystallization is not reflected in the cooling curve. Since DSC was essentially able to measure small amount of heat transfer, extrapolated end temperatures of endothermic peaks were measured in several heating rates in this study. As a result, the extrapolated end temperature of endothermic peak was a linear function of square root of the heating rate. It was found that intercept at a temperature axis of the linear function was concluded to be liquidus temperature.

3:20 PM

Modeling Reactive Wetting in Bi-Sn System: *Shun Su*[1]; Bruce Murray[1]; Ying Sun[1]; [1]Binghamton University

Reactive wetting, referred to as a liquid spreading on a substrate with reaction or diffusion, is involved in soldering, brazing, printing, and many other materials joining processes. In this work, a diffusive transport model is employed to study the dynamic behavior of Bi-Sn alloy drops wetting and spreading on Bi substrates. The simulations are performed on a millimeter scale in order to model experiments that are part of a collaborative study. The kinetics of the triple junction, the shape of the dissolution boundary and the final equilibrium stage are investigated as a function of initial Sn concentration, total contact angle, and diffusion coefficient. Also, the experimentally determined mobility relationship (i.e., contact angle versus contact line velocity) is incorporated into the model to better understand the solid/liquid interface evolution (i.e., dissolution depth and contact angle as a function of time). The impact of convective transport is investigated as well.

3:35 PM

Kinetics of Copper Dissolution in Liquid Lead Free Solders in Static and Dynamic Conditions: *Nader Dariavach*[1]; Jin Liang[1]; Vernorris Kelly[1]; Dongkai Shangguan[2]; [1]EMC Corp; [2]Flextronics

During lead free wave soldering for through hole components, high rate copper dissolution may occur to printed wired boards. Sn-Ag-Cu lead-free alloys, the most popular alloys in the electronic industry for surface mount technology (SMT), has more than twice the rate of copper dissolution compared to the Sn-Pb eutectic (63Sn-37Pb) solder alloy. In this study, copper dissolution was evaluated with four lead free alloys at temperatures from 245 up to 300°C with time duration fro 20 up to 600 seconds. Results show that K100LD and SN100C have the lowest rate of copper dissolution. A unique dynamic copper dissolution test was performed to investigate the effect of liquid solder flow speed, time and temperature on dissolution kinetics at motion speeds from 2 up to 15 ft/min. Obtained test results show that alloy selection and process window definition are critical for lead free soldering for through hole component assembly and repair operation.

3:50 PM Break

4:05 PM
Three-Dimensional (3D) Visualization and Modeling of Reflow Porosity in Pb-Free Solder Joints: *Martha Dudek*[1]; Stephen Kranz[1]; Aaron Hilger[1]; Jason Williams[1]; Nikhilesh Chawla[1]; Luke Hunter[2]; S. Lau[2]; [1]Arizona State University; [2]XRadia Corporation

In this paper we describe a non-destructive x-ray tomography technique for visualization of the microstructure of Sn-3.9Ag-0.7Cu/Cu joints. The system had a high resolution (1 µm) which was used to visualize the three-dimensional (3D) characteristics of reflow porosity. A set of experiments were conducted to examine the effect of several key reflow parameters, including flux activation, temperature, and time, on porosity in the joints. Flux activation temperature and time were varied between 170-210ºC and 30-120s, respectively. Solder joints were first visualized using the lab-scale x-ray microtomography system, followed by monotonic shear tests. The effect of pore fraction, size, distribution, and interconnectivity on the mechanical integrity of the solder joints will be discussed.

4:20 PM
Multi-Phase Field Simulation of Intermetallic Compound Growth during Lead Free Soldering: *Min Soo Park*[1]; Raymundo Arroyave[1]; [1]Texas A&M University

A multi-phase field simulation of the morphological evolutions of the intermetallic compounds (IMCs) formed during reaction between liquid Sn-based solder and copper substrate is presented. Cu-substrate, Cu^3Sn layer, Cu^6Sn^5) grains, and the Sn-liquid phase are considered. In order to determine the diffuse interface structure, we assume that all components of coexisting phases within the interfacial region have equal chemical potential, which is calculated from CALPHAD thermodynamic databases. Fast grain boundary diffusion in the IMC layer, the IMC grain coarsening along the IMC growth, and the dissolution of Cu from the substrate and IMC layer will be discussed and compared to previous works. The simulation will address the kinetics of the IMCs during soldering and the influence of fast GB diffusion and the concurrent coarsening (Cu_3Sn and Cu_6Sn_5) rate of the IMC grains. The kinetic behavior of the substrate/IMC/solder system as a function of model parameters will also be presented.

4:35 PM
Diffusion Multiples Technique in Studying the Mechano-Chemistry of Ni-Cu-Ag-Sn Solders: *Marek Danielewski*[1]; Magdalena Pawelkiewicz[1]; Bartek Wierzba[1]; Nick Bosco[2]; Jolanta Janczak[3]; [1]AGH University of Science and Technology; [2]National Renewable Energy Laboratory; [3]EMPA

Using the HIP technique four series of Ni-Cu-Ag-Sn quaternary diffusion multiples of various geometries were prepared. The kinetics of formation for selected intermetallic compounds and distributions of elements in 2D and 3D fragments are presented. The volume velocity is a unique frame of reference for the mass diffusion and allows the use of mass and momentum continuity equations to model reactive interdiffusion. This presented method allows for a self-consistent phenomenological description of multiscale phenomena and opens a vast number of entirely new possibilities. Comparison of experimental and modeling results are reviewed.

4:50 PM
Thermodynamic Studies of Liquid Cu-Sn-Sb Alloys Using e.m.f and Calorimetric Measurements: *Dominika Jendrzejczyk-Handzlik*[1]; Boguslaw Onderka[1]; Krzysztof Fitzner[1]; [1]AGH University of Science and Technology

The results of metal/oxide experiments proved that in the Sn-Sb-O system liquid alloy remains in equilibrium with pure, solid SnO_2. The thermodynamic properties of the liquid Cu-Sb-Sn alloys were determined using the following galvanic cell: Re+kanthal,Cu_x-$In_ySn_{(1-x-y)}$,SnO_2|ZrO_2+Y_2O_3|Ni_iNiO_i,Pt in the temperature range from 1000 to 1200K. Activities of tin were determined from the measured e.m.f's along chosen cross sections with the ratio x_{Cu}/x_{Sb} kept constant. The cell performance was checked by comparing Gibbs free energy change of the reaction of formation of SnO_2 taken from the literature with that determined from our experiments: $\Delta G^0_{f,SnO2}$ = -567902.68 + 200.33 T (J/mole)in the temperature range from 773K to 1198K. Additionally, to work out precise description of the properties of the liquid phase with Redlich-Kister-Muggianu formula, calorimetric measurements of the heat of mixing were done. Phase relations in the ternary system were calculated and compared with the available experimental data.

5:05 PM
Novel Microprobe Test Metrology to Assess the Mechanics of Complex Multilayer Interconnect Structures: *Alexander Hsing*[1]; Reinhold Dauskardt[1]; [1]Stanford University

Advanced semiconductor technology nodes require the integration of heterogeneous multilayer thin-film structures with vastly different mechanical properties. Quantitative characterization techniques for understanding the mechanical behavior of the overall multilayer stack structure to allow complex packaging is of critical importance but almost completely lacking. We describe a novel test metrology based on a micron-scale diameter probe and a piezoelectric actuator to accurately measure a range of key mechanical properties of multilayer thin-film device structures, including stack compliance, strength characteristics, fracture behavior, and fatigue resistance. With accurate microprobe positioning, we also demonstrate mapping of material properties over the entire die area and the ability to resolve stiffness and strength differences across a given die and between different dies. Micron-scale soldering enables localized tensile loading of the underlying multilayer interconnect structure. Deformation and defect evolution due to local tensile and compressive monotonic and cyclic contact stresses will also be considered.

5:20 PM
Microtensile Testing of Sn-Cu and Sn-IMC-Cu Lead Free Solder Films for Packaging Applications: Zi-Yi Wang[1]; Chi-Jia Tong[1]; *Ming-tzer Lin*[1]; [1]National Chung Hsing University

As the miniaturization continues, the reduced dimensions of soldering materials potentially play a greater role in defining the overall mechanical behavior of the interconnects. In practical, many of the leading solder alloy candidates for replacement of Pb-Sn solders are based on the Sn-Cu system. Therefore, the mechanical properties of Sn-Cu based intermetallics plays a significant role in determining the reliability in joints. Here, we had conducted the uniaxialtensile testing for the mechanical characterization of freestanding Pb-free solders films. We measured the elastic modulus and mechanical properties of intermetallics compounds commonly formed in reactions of Sn-IMC-Cu solder with Cu metallizations resulted in the formation of intermetallics as those formed in electronic joints. The experiments provide a useful database of mechanical properties of lead free Sn-based intermetallics, in terms of deformation mechanisms and reliability concerns in solder joints for the mechanical behavior of thin Pb-free solders film materials use for package technologies.

Progress in Computational Materials Science and Engineering Education: Session I

Sponsored by: The Minerals, Metals and Materials Society, TMS: Education Committee

Program Organizers: Gregory Olson, Northwestern University; Anter El-Azab, Florida State University; Katsuyo Thornton, University of Michigan; Laura Bartolo, Kent State University

Wednesday PM — Room: 3003
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Gregory Olson, Northwestern University; Laura Bartolo, Kent State University

2:00 PM Invited
Status of Computational Materials Education in the US: Results of Recent Surveys: *Mark Asta*[1]; Katsuyo Thornton[2]; R. Edwin Garcia[3]; [1]University of California; [2]University of Michigan; [3]Purdue University

This talk will describe results of recently completed surveys investigating the status of computational materials education at leading research universities in the US. The survey includes results such as number and types of courses taught, the extent to which computational tools are being integrated into core curricula and the perspectives of employers concerning future needs. The results of the survey will be presented along with examples exemplifying approaches taken to including computational material into both undergraduate and graduate courses.

2:25 PM Invited
Integrated Computational Materials Engineering: Opportunities and Needs for Educating Future Materials Engineers and Scientists: *John Allison*[1]; [1]Ford Research and Advanced Enginering, Ford Motor Company

Integrated Computational Materials Engineering (ICME) is considered a new discipline within the materials profession. ICME is the integration of materials information, captured in computational tools, with engineering product performance analysis and manufacturing-process simulation. As an emerging discipline, it is important that the education of future materials engineers and scientists includes an understanding of the potential of ICME and aptitude in use of the tools that are required for its development and utilization. Equally important is motivating their involvement in the design and development of the tools that will be required by this emerging discipline. This talk will attempt to provide an industrial perspective on these educational needs and opportunities. It will also report on findings of a recent National Academies study on ICME which identifies a number of critical needs for change in MSE curricula.

2:50 PM Invited
Computational Materials Science: From NIST to the Academy: *James Warren*[1]; Laura Bartolo[2]; [1]National Institute of Standards and Technology; [2]Kent State University

While NIST's mission is to deliver the highest quality measurement science tools to our customers in industry, other government laboratories and academia, NIST maintains an abiding interest in also transferring these tools to the classroom. In the case of Computational Materials Science, this transfer both enables the training of students in state-of-the-art software tools and, upon their transition to the workforce, the use of these tools by industry, where they can have a direct impact. The NIST Center for Theoretical and Computational Materials Science has been involved in this transfer through a joint effort a number of academic partners to develop Matforge, a repository for computational materials science software, data, and information exchange. In this talk I will discuss our current efforts to use Matforge and the associated tools to help achieve the CTCMS mission.

3:15 PM Invited
Computational Materials Science Education and Training at the University of Florida: *Simon Phillpot*[1]; Susan Sinnott[1]; [1]University of Florida

The integrated program of formal, in-class education and research-driven training in computational materials science at the University of Florida is presented. A review of the computational materials science components in the undergraduate and graduate curricula is given. An analysis is also given of the strengths and weaknesses of the two core classes in graduate computational materials science that are offered. Our approach to research-driven training, including peer-to-peer education at all levels from high school to postdoctoral, is also discussed. Lastly, the computational materials science program is put into the context of education and research on computational methods in other engineering disciplines and in physical sciences. This work is supported by the National Science Foundation (DMR-0426870).

3:40 PM Break

3:50 PM
Public Domain Simulation Tools for the Thermodynamics and Kinetics of Materials Education: *R. Edwin Garcia*[1]; Michael Waters[1]; Matthew Kasenga[1]; [1]Purdue University

In order to prepare future engineers to tackle increasingly complex technological problems and develop physical intuition that leads to a clear understanding on the Thermodynamics and Kinetics of Materials, teaching modules were developed to enrich the undergraduate curricula at Purdue University. The educational modules are learned sequentially by sophomores and juniors in an increasingly sophisticated level of expertise, so that at the end of the semester the student understands the intricacies of real thermodynamic and kinetic systems. Through the development of easy-to-use graphical user interfaces, driving forces, nanosize length scales, and multiphysical contributions on the thermodynamic equilibrium of materials are ununderstood. Given the mathematical intricacies of the state function equations and the time constraints imposed by a classroom setting, these tools have provided the students with a simple interface where they focus on understanding the physical aspects and behavior of matter, instead of dealing with the mathematics.

4:15 PM
From Passive Learners to Active Model-Builders: A Case Study in Computational Materials Science Education: *Paulo Blikstein*[1]; Uri Wilensky[1]; [1]Northwestern University

For the past 4 years, I have developed and tested in classrooms "MaterialSim", a undergraduate-level Computational Materials Science curriculum. In this paper, I investigate: (a) the cognition of students engaging in scientific inquiry through interacting with simulations; (b) the effects of students programming simulations as opposed to only interacting with ready-made simulations; (c) the characteristics, advantages, and trajectories of scientific content knowledge that is articulated in epistemic forms and representational infrastructures unique to Computational Materials Science, and (d) the principles which govern the design of computational agent-based learning environments in general and for materials science in particular. Data sources for the evaluation of these studies include classroom observations, interviews with students, videotaped sessions of model-building, questionnaires, and analysis of artifacts. Results suggest that by becoming 'model-builders,' students develop deeper understanding of core concepts in Materials Science, and learn how to better identify unifying principles and behaviors in the field.

4:40 PM Invited
Computational Materials Science and Engineering Curriculum at Penn State: *Zi-Kui Liu*[1]; Long-Qing Chen[1]; Coray Colina[1]; Vincent Crespi[1]; Tarasankar DebRoy[1]; Evangelos Manias[1]; Jorge Sofo[1]; [1]Pennsylvania State Univ

In the last few years, a number of new computational based courses have been developed at the department of materials science and engineering at Penn State along with the insertion of computational approaches to several existing undergraduate graduate and courses. These courses primarily are focused on thermodynamic and kinetic aspects of materials based on faculty expertise in the department. A range of computational approaches are introduced to students through class lectures, hand-on exercises, and individual and team projects. In this presentation, the overview of our course portfolio will be presented on several core courses. The connection between teaching and research activities in the department will also be briefed.

5:05 PM
Coaching Technical Design Teams in Materials Design Course: *Shengjun Zhang*[1]; Gregory Olson[1]; [1]Northwestern University

The materials design course at Northwestern University is comprised of lectures, computational labs, homework, a midterm and a final design project. The primary goal of the course is to teach design practices grounded in materials science through active learning in a group design project. Each junior-level team is coached by a graduate student or post-doctorial research associate who is performing research in that area. The Terminator 4 is a long term project which has been carried out in one team per year for more than 15 years. The Terminator 4 project leveraged an ongoing research project utilizing a post-doctorial research associate coach to develop a high specific-strength Mg-based alloy composite for aerospace applications that can self-heal. Through thermodynamic calculations, the team designed high performance Mg alloys with thermodynamically compatible shape memory alloy (SMA) wires, which demonstrates yield strengths greater than a commonly used Mg-based alloy.

Computational Materials Research and Education Luncheon Roundtable: FiPy

Sponsored by: National Science Foundation
Program Organizer: Laura Bartolo, Kent State University

Wednesday PM
February 18, 2009

Room: 3003
Location: Moscone West Convention Center

Session Chair: Laura Bartolo, Kent State University

12:45 PM Panel Discussion

Recycling—General Session: Session II: Waste Utilization

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizer: Joseph Pomykala, Argonne National Laboratory

Wednesday PM Room: 2024
February 18, 2009 Location: Moscone West Convention Center

Session Chair: Joseph Pomykala, Argonne National Laboratory

2:00 PM
Properties of Clay-Based Ceramics Added with Construction and Demolition Waste: *Wilson Acchar*[1]; Ana Segadaes[2]; Sonia Castanho[3]; Juliana Silva[1]; [1]Federal University of Rio Grande do Norte; [2]University of Aveiro, Portugal; [3]Institute of Energy and Nuclear Research

The accelerated growth of urbanization processes and current environmental concerns brought to light the existing enormous quantity of construction and demolition waste (C&DW). Considering the frequent lack of adequate landfill areas available for the disposal of these materials, their use in the manufacture of ceramic products has been attracting a growing interest from researchers and is becoming common practice, with the consequent relief in public administration concerns for the appropriate C&DW management. This work describes the study of clay-based products added with C&DW collected directly from the building industry. Samples containing up to 50 wt.% C&DW were uniaxially pressed and sintered in air in an electric furnace (950-1150°C, 1 hour). Sintered test pieces were characterized by X-ray diffraction, apparent density, open porosity and flexural strength. The results showed that up to 50 wt.% C&DW can be added into the clay material, with no degradation in the final product properties.

2:20 PM
Characterization and Recycling of Paper Sludge Waste into Clay Bricks – Industrial Test: *Carlos Maurício Vieira*[1]; Rubén Sánchez[1]; Sergio Monteiro[1]; Regina Pinheiro[1]; [1]State University of the Northern Fluminense

This work has for objective to both characterize a waste generated in the form of sludge during the treatment of the effluent of a paper making industry and to evaluate the effect of its incorporation in the amount of 10 wt.% on the properties and microstructure of a clayey brick. The characterization of the waste was done by X-ray diffraction, X-ray fluorescence, differential thermal analysis and termogravimetric analysis. The physical and mechanical properties evaluated of the bricks fired at 750°C were water absorption and compression strength. The microstructure of the fired ceramic was evaluated by optical microscopy. The environmental evaluation was made by solution test and atmospheric emission by monitoring the release of SO2, NOx and particulate material. The results showed that it is possible to recycle this type of waste with energy saving during the firing stage.

2:40 PM
Synthesis of Mullite from the Residue of Kaolin by Microwave: *Lisiane Santana*[1]; Maria Brasileiro[1]; Gelmires Neves[1]; Helio Lira[1]; Romualdo Menezes[1]; Heber Ferreira[1]; [1]UFCG

The production of waste for ore industry is a problem that is intensifying nowadays because of high productivity. The kaolin processing industries produce residue rich in Al2O3 and SiO2, oxides that are precursors to produce mullite. So, the aim of this work is to use the residue from the kaolin improvement industry to produce mullite ceramics bodies. In this study it was used as raw materials: alumina and residue from the kaolin industry. The powder compacts were prepared by uniaxial pressing at 30 MPa. The material was drying and after sintering in the microwave oven. The ceramics bodies formed by the microwave reaction were characterized by X-ray diffraction and scanning electron microscopy. The flexural strength was measured by the three point bending technique. Based on these results it was concluded that is possible the production of the mullite bodies using high quantity of the waste.

2:55 PM
Recycling of Steelmaking Slag Aiming at the Production of Thermo-Acoustic Insulation: *Joner Alves*[1]; Denise Espinosa[1]; Jorge Tenório[1]; [1]Universidade de São Paulo - USP

The aim of this work was to study the use of steelmaking slag as raw material to the production of rock wool and glass wool. Firstly, the chemical composition of the slag was analyzed. These results were used to infer the amount of additives to assemble the charge for the experiments. A laboratory-scale electric furnace was used to melt the mixtures used as charge. The melt material were turned into a water filled recipient, this process was performed in order to impose a fast freezing that is a characteristic of mineral wool production. Finally, with the rest of the melt material it was performed the Herty Viscosity Test. Samples of the produced material were characterized by different analyses. The results found in this research may lead to a technological innovation, because they indicate that the use of steelmaking slag to produce rock wool and glass wool is possible.

3:15 PM Cancelled
Moving Business to Clean Production and Triple the Bottom Line: *J. Michael Huls*[1]; [1]California Take It Back Partnership

3:30 PM
Production of High-Grade Potassium Chloride from a Sinter Plant Baghouse Dust: *Naiyang Ma*[1]; [1]Arcelor Mittal

A sinter plant baghouse dust has been treated as a hazardous solid waste, and disposed at a very high price. A series of leaching tests were conducted to examine the possibility to produce high grade potassium chloride from the solid waste. It is found that around 50% of the solid wastes can dissolve into water. By taking step precipitation, a deposit with more than 95% KCl can be produced.

3:50 PM
Life Cycle Inventory Analysis of Municipal Solid Wastes Management: *He Dewen*[1]; Song Dan[1]; Liang Dingming[1]; Du Lu[1]; [1]Central South University

Based on the principle of life cycle assessment (LCA), municipal solid waste management (MSWM) was analyzed by life cycle inventory method. According to the system expansion and substitution method, the content and frame of life cycle analysis is summarized and applied to municipal solid waste. On the basic of feasibility research of life cycle analysis on MSWM, the paper put forwards complete and concrete “three changes” measures under life cycle analysis on MSWM. Some aims of MSWM can be achieved under above conditions of LCA on MSWM: to improve the current system efficiency of MSWM; to enhance the serve quality of MSWM, to increase recycle garbage from MSW; to reduce waste amount and reach minimization and innocuity aim of MSWM; to help the departments of MSWM to get over all difficulties to realize their ideal goals.

4:10 PM Break

4:30 PM
Study on Selectively Separating Iron Constituents in Copper Smelting Slags: *Hongyang Cao*[1]; Li Zhang[1]; Cigong Wang[1]; Nianxin Fu[1]; Zhitong Sui[1]; Naixiang Feng[1]; [1]Northeastern University

The iron constituents selectively separated from the modified copper slags by magnetic separation and flotation were studied. The results of magnetic separation for modified copper smelting slags show that the grinding size, current intensity and dispersant agent had great influence on grade, and recovery ratio of TFe in preparation concentrate; The results of flotation for magnetic concentrate show that the positive flotation of sodium oleate negatively upgrade the grade of magnetic concentrate, but the reverse flotation of lauryl amine and soluble starch are effective. The grade of TFe can reach 54% and the recovery ratio can reach about 90% in magnetic concentrate, when the grinding size is 45.8μm and the current intensity is 2.5A. The optimal effect is obtained by using lauryl amine as collector and soluble starch as depressant to reverse flotate magnetic concentrate,and the grade of TFe be upgraded 2.37%.

4:50 PM
Preparation of Iron Oxide Red Pigment Powders from Pyrite Cinders by Hydrothermal Method: Zhaocheng Liu[1]; Yajie Zheng[1]; [1]Central South University

Iron oxide red pigment powders of high quality were prepared from pyrite cinders. Iron oxide red pigment powders were prepared by hydrothermal method from the precursor of Fe(OH)3 and Fe(OH)2 colloid, which were obtained after ammonia solution reacted with acidic leaching solution produced by the reaction

of pyrite cinders with sulfuric acid. Effects of reaction concentration, reaction temperature, reaction time, pH value and n(Fe2+)/n(Fe3+) on main properties of oxide iron red were studied by experimrents.The prepared particles were characterized by means of X-ray diffraction (XRD) and scanning electron microscope (SEM). The results revealed that the prepared iron oxide red particles were a-Fe2O3 and pseudocubic with particle size of 0.50~0.75μm. Furthermore, iron oxide red pigment powders reached the first grade of China standard (GB1863-89).

5:10 PM

Research on Treatment Project of Municipal Solid Waste of Changsha City: *He Dewen*[1]; Liang Ding-ming[1]; Du Lu[1]; Qin Yan[1]; [1]Central South University

The municipal solid waste is a by-product of the daily life of urban residents. The quantity and ingredients of municipal solid waste has exceeded the current management and disposal ability, which has done a great harm to municipal environment and sanitation quality and threatened people's health. On the basic of analysis on the amounts and main elements of municipal solid waste of Changsha City, the sanitation landfill, incineration, composting, reclamation and utilization technology of household waste were compared specifically in this paper. Based on the concrete conditions of Changsha City, the paper has proposed an disposal proposal combined with reclamation and landfill of municipal solid waste of Changsha City, and the environmental benefit, social benefit and economical benefit of the prepared processing routes were also analyzed in the paper

5:30 PM

Separation for Recycling of Spent Potlining by Froth Flotation: Chuanlin Fan[1]; Yongfeng Chang[1]; *Xiujing Zhai*[1]; Yan Fu[1]; Binchuan Li[1]; [1]Northeastern University

The process of flotation separation and recycling of spent potlining (SPL) was described. On the basis of the laboratory investigations, the effects of influencing factors in froth flotation process, such as grain size, flotation agent dosage, pH value, pulp density, and dissolved components concentration, etc, were carefully studied. And the influence on environment of the process was also discussed. It was found that flotation separation can be used as an environment-friendly method for treatment of SPL residue. Carbon and other compounds (include cryolite, sodium fluoride, calcium fluoride and diaoyudaonite, etc.) from SPL were separated and recycled as flotation products respectively.

RPV Embrittlement and Fusion Materials: Measuring, Modeling and Managing Irradiation Effects: RPV Embrittlement: Technical Contributions of Professor G. Robert Odette

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

Program Organizers: Matthew Alinger, GE Global Research; Kurt Edsinger, Electric Power Research Institute; Roger Stoller, Oak Ridge National Laboratory; Brian Wirth, University of California, Berkeley

Wednesday PM — Room: 2008
February 18, 2009 — Location: Moscone West Convention Center

Session Chairs: Brian Wirth, University of California, Berkeley; Roger Stoller, Oak Ridge National Laboratory

2:00 PM Invited

An Evolution of Understanding of Reactor Pressure Vessel Steel Embrittlement: *Glenn Lucas*[1]; [1]University of California, Santa Barbara

This paper will attempt to summarize the lifetime contributions of Prof. G. Robert Odette to our understanding of the effects of neutron irradiation on reactor pressure vessel steel embrittlement. These contributions span the entire range of phenomena that contribute to embrittlement, from the production and evolution of fine scale features by radiation damage processes, to the effects of this damage microstructure on mechanical properties. They include the development and application of unique and novel experimental tools (from Seebeck Coefficient to Small Angle Neutron Scattering to confocal microscopy/ fracture reconstruction), the design and implementation of large multi-variable experimental matrices, the application of multi-scale modeling to understand the underlying mechanisms of defect evolution and property change, and the development of predictive methodologies employed to govern reactor operations. The ideas and discoveries have provided guidance worldwide to improving the safety of operating nuclear reactor pressure vessels.

2:20 PM Invited

The Development of Mechanistic Understanding of RPV Embrittlement: *Colin English*[1]; [1]Nexia Solutions

Professor G. Robert Odette has and continues to make significant technical contributions to understanding of the mechanisms controlling the embrittlement of thermally aged and irradiated RPV steels and the application of such understanding to the development of physically-based models that predict the embrittlement of RPV steels. In recognition of the 65th birthday of Professor Odette this paper highlights these technical contributions; in particular his role in the founding of IGRDM and his seminal contribution to developing an accepted framework of the mechanisms of RPV embrittlement. In the latter it will be emphasised how his work ensured a proper debate on the effects of irradiation parameters, such as flux, and material parameters, such as composition and in particular the role of Mn and Ni in the formation of irradiation-induced solute clusters in low Cu RPV steels.

2:40 PM Invited

RPV Embrittlement Models: The Technical Contibution of Professor G. R. (Bob) Odette: *Timothy Williams*[1]; [1]Rolls-Royce plc

In the 1960s and 1970s irradiation embrittlement of RPV steels was predicted using simple trend curves based on statistical fits to the available surveillance data. In general, the models were empirical and the important variables and interactions were deduced by visual or statistical assessment of the data. Bob Odette was responsible for introducing three major advances, which transformed embrittlement modelling and continue to do so. The first was an insistence that model formulation and development be based on sound physical understanding. Second, that important gaps in understanding must be filled by careful, systematic experiments, and the results and insights of these experiments should be used to guide the development of the models fitted to the surveillance data. Third, the continued development of physical models of irradiation damage was also vital to provide insights about the irradiation damage process and to enable better data interpretation.

3:00 PM Invited

Prediction of Irradiation Damage Effects in RPV Steels in the Frame of the PERFECT Project: *Christophe Domain*[1]; Bernard Marini[2]; David Lidbury[3]; Stéphane Bugat[1]; Jean-Paul Massoud[1]; [1]EDF R&D; [2]CEA; [3]Serco Assurance

A multi-scale numerical modelling tools has been developed within the EURATOM project PERFECT to simulate the effects of neutron irradiation on the mechanical, fracture and corrosion properties of the Reactor Pressure Vessel and internal structures in Light Water Reactors (LWR). The models are based on the physics and mechanics at the scale where it can be described with well defined parameters and concepts. They were carefully validated at the relevant scales with dedicated experiments and investigations. PERFECT addresses both the microscopic effects of irradiation, which are manifested by changes in the microstructure and basic properties of the materials, as well as the mechanical properties, which influence the behaviour of reactor components. The simulation tools can be used to predict long-term projections and help the understanding of the phenomena leading to degradations design experimental programs. At the same time the models contribute to explore conditions outside existing experimental databases.

3:20 PM Invited

Characterization of the Early Stages of Solute Clustering in A508 Gr4N Steels and a 1 Ni – 1.3 Mn Weld: *M. Burke*[1]; Jonathan Hyde[2]; R. M. Boothby[2]; Colin English[2]; William Server[2]; [1]Bettis Laboratory; [2]Nexia Solutions

During the last decade there have been significant improvements in microstructural techniques that can be used to characterise the damage caused by neutron irradiation and this has led to improvements in the mechanistic understanding of embrittlement. However, it is clear that no single technique can currently provide a complete description of the microstructural changes that occur as a result of neutron damage. Furthermore, despite advances in techniques and mechanistic understanding, many questions still persist and the detailed interpretation of microstructural data obtained remains open to academic debate. In this paper the authors examine the early stages of irradiation-induced clustering in a low Cu (0.03wt.%), high Ni (~1wt.%) weld and A508 Gr 4N

steels. The materials were irradiated at very high dose rate and then examined by atom probe (Energy-Compensated Optical Position Sensitive Atom Probe-ECOPoSAP) with supporting microstructural information obtained by small angle neutron scattering (SANS).

3:40 PM Break

4:00 PM Invited
Atom Probe Tomography Characterizations of Neutron Irradiated Pressure Vessel Steels: *Michael Miller*[1]; Kaye Russell[1]; [1]Oak Ridge National Laboratory

Atom-probe tomography has provided unique information on the effects of neutron irradiation on the microstructure of reactor pressure vessel steels and related model alloys including the formation and dissolution of ultra-fine copper-enriched precipitates in copper-containing steels and nickel-, manganese-, and silicon-enriched nanoclusters in low copper and copper-free steels; solute segregation to and precipitation on dislocations; and solute segregation to grain boundaries. A review of the quantification of the solute distribution in the microstructure of several different pressure vessel steels after post weld stress relief heat treatments, neutron irradiation, and post irradiation annealing treatments, as determined by atom probe tomography, will be presented. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by Basic Energy Sciences, U.S. Department of Energy and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission, under inter-agency agreement 1886-N695-3W and under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:20 PM Invited
On the Composition of Cu-Mn-Ni Precipitates in Irradiated Reactor Pressure Vessel Steels: *Brian Wirth*[1]; G. Robert Odette[2]; Michael Miller[3]; Takuya Yamamoto[2]; R. D. Klingensmith[2]; J.M. Smith[2]; [1]University of California, Berkeley; [2]University of California, Santa Barbara; [3]Oak Ridge National Laboratory

Irradiation embrittlement of reactor pressure vessel (RPV) steels results from formation of a high density of nm-scale precipitates. In RPV steels with >0.1%Cu the dominant hardening features are copper-rich precipitates (CRPs) alloyed with manganese, nickel and silicon. But as theoretically predicted long ago, manganese-nickel rich precipitates (MNPs) can form in both copper bearing and copper free alloys, containing large amounts of these elements. Large volume fractions of these late blooming MNPs (LBP), cause severe hardening and embrittlement. The presence of LBP-MNPs and large hardening in low copper and copper free alloys has been demonstrated by a variety of techniques. We present results and comparisons from several complementary microanalytical characterization techniques used to investigate the nanoscale precipitates, including small angle neutron scattering, atom probe tomography, positron annihilation spectroscopy, and electrical resistivity – Seebeck coefficient measurements. The results are discussed in context of extended operation of light water reactors.

4:40 PM Invited
Fundamental Modeling of Radiation Effects: *Roger Stoller*[1]; [1]Oak Ridge National Laboratory

Radiation damage in structural materials involves a complex range of phenomena with influence over many time and spatial scales. Considerable research during the past 60 years has provided a good understanding of these phenomena, with the most significant progress obtained when well designed experiments were analyzed on the basis of sound physical models. The computational advances of recent years have provided the opportunity to advance the theory and modeling of radiation effects, taking advantage of the dramatic improvements in the tools and techniques for microstructural and mechanical characterization. The fundamental models used to investigate radiation effects at length scales ranging from the atomistic to the macroscopic will be discussed, with an emphasis on the methods that have been developed and applied by Professor Odette and his co-workers.

5:00 PM Invited
Dose Dependence of Irradiation Hardening of Fe-Cu and Fe-Mn Model Alloys: *Akihiko Kimura*[1]; [1]Kyoto University

The amount of irradiation hardening of iron-copper alloys is significantly larger than those of the other iron base alloys, indicating that copper cluster is a higher potential hardener. The contribution of copper cluster to the hardening is rather large but almost similar to the hardening caused by interstitial dislocation loops. The microvoids are not the high potential hardener. Another high potential hardener is manganese. The iron-manganese model alloys suffer a remarkable hardening as large as those in iron-copper alloys. Since manganese atoms do not cluster like copper atoms and the number density of interstitial loops increases significantly, it is considered that manganese enhances the formation interstitial loops and causes large hardening. The dependence of irradiation hardening on irradiation dose is discussed on the bases of the previous our irradiation data and hardening mechanisms shown above.

5:20 PM Invited
Atomic-Scale Modeling of Hardening Due to Cu-Precipitates in Iron: *Yuri Osetsky*[1]; Roger Stoller[1]; David Bacon[2]; [1]Oak Ridge National Laboratory; [2]University of Liverpool

A vast contribution to mechanical properties depends on the direct interaction between dislocation and obstacles that depends on the particular atomic-scale structure of the both moving dislocation core and obstacle. In this work we present recent progress in large-scale modeling of edge dislocation dynamics in iron containing Cu-precipitates initially coherent with the bcc matrix. Interactions with precipitates of different size were studied over a wide temperature and strain rate ranges. Special attention was paid to structural instability of precipitates, phase transformation during interactions and factors affecting this (temperature, strain rate, vacancy concentration inside precipitates). It was concluded that structural transformation is responsible for the precipitate size dependence of temperature dependent Cu-precipitate hardening.

5:40 PM Invited
Reactor Pressure Vessel Embrittlement and Fracture Mechanics Contributions of Prof. G. Robert Odette: *Randy Nanstad*[1]; [1]Oak Ridge National Laboratory

Current regulations require reactor pressure vessels (RPV) to maintain conservative margins of fracture toughness during both normal operation and accident transients. Neutron irradiation degrades fracture toughness through the evolution of nanoscale features in RPV steels which are linked to key embrittlement variables and how they mediate embrittlement through the micromechanics of the ductile-to-brittle transition temperature shift. Advances have been made in fracture mechanics that permit determination of the transition temperature using relatively small specimens, e.g., the fracture toughness master curve. Professor G. R. Odette and co-workers at UCSB have made significant contributions to many aspects of fracture technology through a combination of experiments, modeling and microstructural studies, including development of models that link fundamental mechanisms of radiation damage with kinetic embrittlement models for prediction of RPV steels frangibility. These studies have provided significant insights into the nature of fracture in RPV steels that enable major advances in RPV embrittlement predictability.

Solar Cell Silicon: Production and Recycling: Session II

Sponsored by: The Minerals, Metals and Materials Society, TMS Extraction and Processing Division, TMS Light Metals Division, TMS: Recycling and Environmental Technologies Committee
Program Organizer: Anne Kvithyld, SINTEF

Wednesday PM — Room: 3004
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Anne Kvithyld, SINTEF

2:00 PM
Recycling of Solar Cell Silicon: An Overview: *Lifeng Zhang*[1]; Xuewei Lv[1]; [1]Missouri University of Science and Technology

The recycling of solar cells include three parts: recycling of the top-cut solar cell silicon, recycling of the sawing slurry generated during wafer cutting, and recycling the used solar cell modules. This paper extensively reviewed the current state-of-the-art of these three kinds of recycling of solar cell silicon. The most recent research on this topic at Missouri S&T was also summarized: a) the new filtration process removed 99% of the inclusions in the top-cut silicon scraps without introducing any pollution to the silicon; b) a design for the recycling of silicon sawing slurry was proposed, and was welcomed by several industries;

c) remelting of waste solar cell modules was also proposed with a new non-contaminative technology.

2:30 PM

Electrorefining of Silicon in Molten Chloride Eelectrolytes: *Geir Haarberg*[1]; Ole Kongstein[2]; Shuihua Tang[1]; Shulan Wang[3]; [1]Norwegian University of Science & Tech; [2]SINTEF Materials and Chemistry; [3]Northeastern University

Electrochemical studies and electrolysis experiments were carried out in molten chloride electrolytes to develop a process for the electrorefining of metallurgical grade silicon to produce solar grade silcon. The molten electrolyte was based on calcium chloride containing some sodium chloride, calcium oxide and dissolved silicon. The experiments were carried out under dry argon at 850°C. Metallurgical grade silicon was mixed with copper and prepared in separate experiments to produce the alloy for the anode (38 - 62 mol% Si - Cu). Molybdenum and high purity silicon were used as cathode substrates, while Ag/AgCl placed in a mullite tube was used as the reference electrode. Electrochemical studies showed that silicon can be anodically dissolved and cathodically deposited from electrolytes based on molten calcium chloride. Analyses of the silicon deposits were made by EDS and ICP. Promising electrorefining results for some key elements were obtained in recent experiments.

3:00 PM

Pre-Electrolysis of Electrolyte for Silicon Electrorefining: Ji-guang Yi[1]; *Yan-qing Lai*[1]; Zhong-liang Tian[1]; Ming Jia[1]; Jian-feng Yan[1]; Ye-xiang Liu[1]; [1]Central South University

The pre-electrolysis of Na_3AlF_6-K_2SiF_6 melt was carried out and the metallurgical silicon was electrorefined in the pre-electrolyzed bath with liquid electrodes. The results show that the appropriate current density of pre-electrolysis is 20 mA/cm^2 at 1000°. The contents of impurities and moisture are reduced after pre-electrolysis process. The purity of the electrolyte is a key factor to obtain high pure silicon, and the silicon of the purity 99.99 % can be obtained by controlling the electrolysis condition.

3:30 PM

Production of High Purity Silicon for Solar Cell with Three-Layer Process Method: Liang Pang[1]; *Huimin Lu*[1]; [1]Beijing University of Aeronautics & Astronautics

At present, polysilicon with 99.9999% purity has been used to solar cell raw. The traditional method of producing polysilicon is improved SIEMENS method. In this paper, the authors explored a new molten salt electrolysis method for purify industrial silicon. Electrolytic process for purify the industrial silicon was called the three-layer process, in which silicon is refined from a lower anodic molten layer of impure silicon, via an intermediate electrolyte layer of molten salts, to a top cathodic layer of pure silicon. The electrolytic process was carried out at temperature 1450 – 1500°C. The industrial silicon fed into the cell may be electrolytically refined to high purity silicon but impurities as Al, Fe, Ca, B, P decreased obviously with ICP-AES test.

State of the NSF Metallic Materials and Nanostructures (MMN) Program: Session I

Sponsored by:
Program Organizer: Alan Ardell, University of California

Wednesday PM — Room: 2002
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: Alan Ardell, University of California

12:30 PM

Materials Research Support at the National Science Foundation: *Alan Ardell*[1]; [1]Program Director, Metallic Materials & Nanostructures Division of Materials Research, Directorate of Mathematical and Physical Sciences, National Science Foundation, Arlington, VA 22230

The NSF perspective on needs and opportunities in materials research and education will be presented. The National Science Foundation invests approximately $400 million annually in this field, supporting people, ideas, and tools primarily through awards to the nation's colleges and universities. A major focus for this activity is NSF's Division of Materials Research (DMR), but there is also substantial support for materials and materials-related research and education from other areas of NSF. Specific new opportunities and directions – including a relatively new program in biomaterials to focus DMR support for the study of biologically related materials and phenomena - will be described. There are also opportunities for collaborative research, nationally and internationally, via different types of block funding programs. For additional information, visit the DMR Web page at <http://www.nsf.gov/mps/divisions/dmr/>. For general inquiries please feel free to contact me, email: aardell@nsf.gov or Dr. Bruce MacDonald, DMR Expert, email: bmacdona@nsf.gov

Surface Structures at Multiple Length Scales: Processing of Novel Surfaces

Sponsored by: The Minerals, Metals and Materials Society, TMS Materials Processing and Manufacturing Division, TMS: Surface Engineering Committee
Program Organizers: Arvind Agarwal, Florida International University; Sudipta Seal, University of Central Florida; Yang-Tse Cheng, University of Kentucky; Narendra Dahotre, University of Tennessee; Graham McCartney, University of Nottingham

Wednesday PM — Room: 3011
February 18, 2009 — Location: Moscone West Convention Center

Session Chair: To Be Announced

2:00 PM Invited

Functional Nanostructures through Nanosecond Laser-Induced Dewetting: *Ramki Kalyanaraman*[1]; [1]University of Tennessee, Knoxville

Techniques of processing nanoscale metallic structures with spatial order and tunable physical characteristics, such as size and microstructure, are paramount to realizing applications in the areas of magnetism, optics and sensing. Here we discuss how pulsed laser melting of ultrathin films can be a powerful but simple and cost-effective technique to fabricate functional nanostructures. Ultrathin metal films (1 to 100 nm) on inert substrates like SiO are generally unstable, with their free energy resembling that of a spinodal system. Such films can spontaneously evolve into predictable nanomorphologies with well-defined length scales. Here we review this laser-based experimental technique and provide examples of resulting robust nanostructures that can have applications in magnetism and optics.

2:30 PM

Novel Infrared-Processed Titanium Composite Coatings for High Temperature Galling Resistance: *Evan Ohriner*[1]; Peter Blau[1]; [1]Oak Ridge National Laboratory

Titanium alloys offer high strength-to-weight ratios for elevated temperature use but are susceptible to wear and galling in applications in which metal to metal contact occurs. A series of titanium composite coatings are being developed that contain significant volume fractions of refractory hard particles, such as carbides and nitrides. The coatings are applied by a novel method of infrared heating using a plasma arc lamp to provide a metallurgical bond to the coating and minimize dilution of the coatings by the substrate. The effects of coating composition and processing parameters on the microstructures of the coatings are discussed.Research sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U. S. Department of Energy.

2:50 PM

Reducing Crack Initiation and Propagation in Coatings Containing Suspended Brittle Particles: Steven Bianculli[1]; *Jack Beuth*[2]; [1]US Steel Research and Technology Center ; [2]Carnegie Mellon University

In this talk, modeling research is presented for determining optimal sizes and shapes of brittle particles suspended in otherwise ductile coatings to increase coating fracture resistance. Although this research is inspired by strain-induced cracking problems in steel coating systems, results are applicable to the general problem of a coating containing brittle particles. In many such systems, coating cracking is linked to the formation of cracks in brittle particles, which then propagate through the coating thickness with additional applied strain. This research considers the separate problems of particle cracking and subsequent propagation of the crack from the particle. This leads to the identification of optimal particle shapes and sizes to reduce crack initiation and propagation, as a function of the ratio of coating matrix and particle stiffnesses.

3:10 PM

Microstructure and Mechanical Properties of 316l/Polymer/316l Sandwich Systems: *Adele Carrado*[1]; Heinz Palkowski[2]; [1]IPCMS; [2]Clausthal University of Technology

Sandwich systems (SMs) - as hybrid materials - offer significant opportunities for enhancement of product performance in terms of strength, stiffness combined with low density. SMs are used in industrial applications such as automotive-, building-, transport-, chemical-, aerospace- and airplane industry. Nevertheless there is a lack of understanding in the interaction between the mono-materials and their behaviour as hybrids, especially in processing. This paper deals with the development of functionally adapted and for customers' use designed SMs produced by press joining rolling process and heating press process. For surface layers austenitic high-grade steel (316L) sheets were used and for the cores different thermoplastic and/or thermoset polymers (also fibre reinforced). Beside the mechanical tests stretch, deep drawing and adhesive tests with different simple geometries were done. Various morphological observations were performed in order to connect them with the mechanical response.

3:30 PM Break

3:40 PM Invited

Synthesis, Elastic Properties and Chemical Stability of MAX Phases: *Jochen Schneider*[1]; [1]RWTH Aachen

A strategy towards knowledge based materials selection for Al_2 O_3 /NiAl based composites for efficient energy conversion is presented. Theoretical and experimental elasticity and phase stability data are considered for a design proposal including a MAX phase interlayer along the Al_2 O_3 /NiAl interface. The major challenges that need to be addressed here are: a) chemical stability at temperatures up to 1300°C and b) stress management during cooling of Al_2 O_3 /NiAl, where NiAl has a 1.6 larger thermal expansion coefficient than Al_2 O_3. Based on bulk modulus, c_44 to bulk modulus ratio, ductility models, and results of our theoretical stress strain analysis, we expect sufficient stiffness and ductility of the MAX phase interlayer to accommodate mechanical loading during operation and cooling down. Our calorimetric data indicate the formation of a stable Al_2 O_3 /V_2 AlC/NiAl composite to be realized at T < 1499°C.

4:10 PM

Microstructure Evolution and Thermal Stability of an Fe-Based Amorphous Alloy Powder and Thermally Sprayed Coatings: K. Chokethawai[1]; P. H. Shipway[1]; *Graham McCartney*[1]; [1]University of Nottingham

High velocity oxy-fuel (HVOF) thermal spraying was used to deposit coatings of a multi-component Fe-based amorphous alloy (Fe43Cr16Mo16C15B10) approximately 300 μm thick onto a steel substrate. The microstructures of the feedstock powder and the coatings before and after heat treatments were investigated by a combination of X-ray diffraction, scanning and transmission electron microscopy and differential scanning calorimetry (DSC). The as-sprayed coatings and gas atomised feedstock powders both had fully amorphous structures. The coatings had layered morphologies due to the deposition and solidification of successive molten or semi-molten slats but only a small amount of oxide formed during the spraying process. The thermal stability and crystallization behaviour of the powders and the coatings were investigated by DSC and XRD techniques. A comparison of the results of XRD and DSC measurements shows that crystallization in the samples is largely a single stage processes. The enthalpy of crystallization and its activation energy were calculated from the DSC data. Broadly, powders and coatings showed identical behaviour with only small differences in crystallization peak shapes.

4:30 PM

Structure-Property Correlation of ZnO Thin Films Grown on Sapphire(0001) by a Two-Step Process by Pulsed Laser Deposition: *Punam Pant*[1]; Jagdish Narayan[1]; [1]North Carolina State University

We have investigated growth of ZnO on sapphire(0001) by a two-step growth process, where the LT buffer grows two-dimensionally via domain-matching epitaxy and therefore it is expected to play a critical role in defect reduction and in turn in improving the device properties of the film. The measured surface roughness(rms) of the films is~1nm. Smooth surface morphology of the film is critical for high performance of LEDs and other optical devices. High-Resolution theta-2theta scans through ZnO(0002) and (10-11) give values of 5.217A and 3.244A for the c and a-lattice parameters. These values of the lattice constants signify that films are fully relaxed as expected from the domain matching paradigm. These results will be supplemented by HRTEM, electrical and optical characterization of the films to further understand the role of the buffer layer in strain relaxation and defect reduction mechanism in the grown film for improved heteroepitaxy of ZnO on sapphire.

4:50 PM

Developing Reversible Surface Structures on Shape Memory Alloys: *Xueling Fei*[1]; Yang-Tse Cheng[2]; David Grummon[1]; [1]Michigan State University; [2]General Motors Corporation

Reversible surface structures are desirable for many applications, ranging from friction control to information storage. In this paper, we present our method of indentation-planarization to create various reversible surface structures on NiTi shape memory alloys with feature sizes ranging from nano to millimeters. Specifically, we show that a shallow indent, made under a spherical tip, can fully recover upon austenitization. Deep spherical indents, made in the martensite, show pronounced two-way cyclic depth changes, for an unlimited number of thermal cycles. Upon planarization, two-way cyclic depth changes are converted to reversible surface protrusions. Various surface protrusions were made using this indentation-planarization method to achieve a variety of basic forms (such as lines and circles). We believe that these "thermo-topodynamic" surfaces at the micro and nano scales have many potential applications for micro-electrical-mechanical systems (MEMS), nano-scale sensors and actuators, variable friction surfaces, information storage devices, and controllable optical devices.

5:10 PM

Formation of Regular Nanostructures on Surfaces of Copper Foils under Electric Discharge: *Sergey Pyachin*[1]; Victor Zavodinsky[1]; Maxim Pugachevsky[1]; Alexander Burkov[1]; [1]Institute of Materials of Khabarovsk Scientific Centre of Far Eastern Branch of the Russian Academy of Sciences

We have studied the regular structure formation on the metal surface under a single electric discharge. 60-120-microns-thick copper foils were used as cathodes. As the anode, a copper sharpened rod was used. The electric discharging current was equal to 10-40 A and the pulse duration was varied in the range of 0.05-1.2 ms. The copper foil is melted by heat, releasing at the electric discharge region. The melting area has a radial symmetry. In regions located approximately 100 microns from the centre of the melting area, a regular cell structure is seen distinctly with periodicity of 0.5–1 micron. The AFM studies have shown that cells consist of small crystallites with lateral size of 50-200 nm. The Fourier analysis of AFM images of the copper surface has shown that the arrangement of cells aspires mainly to the hexagonal packing.

5:30 PM

Structure Formation in Covering Layer at Low-Voltage Electrospark Processing: *Sergey Khimukhin*[1]; Marja Teslina[1]; Tatjana Khimukhina[2]; [1]Institute of Materials of Khabarovsk Scientific Centre of Far Eastern Branch of Russian Academy of Sciences; [2]Pacific National University

Electrospark alloying (ESA) is one of the deposition coating methods with the use of concentrated energy streams. EAS is based on using the electric spark occurring between the anode and the cathode. To investigate the above mentioned basic laws copper and its alloys have been used as the material for the electrodes. Two main mechanisms of covering layer structure formation have been discovered. In one case in the absence of the defects the covering structure consists of a number of basaltiform crystal grains normally oriented towards the layer surface along its whole thickness and equiaxed grains being in the middle. In the other case, the layerwise oriented basaltiform crystal grains are divided by the defects. To reduce or to eliminate the defects and to obtain the required covering layer structure it is necessary to warm up the cathode in the course of electrospark processing to the temperature of 50°C.

Transformations under Extreme Conditions: A New Frontier in Materials: Pressure/Stress-Induced Transformations and In Situ Diagnostics II

Sponsored by: The Minerals, Metals and Materials Society, ASM International, ASM Materials Science Critical Technology Sector, TMS Materials Processing and Manufacturing Division, TMS/ASM: Phase Transformations Committee
Program Organizers: Vijay Vasudevan, University of Cincinnati; Mukul Kumar, Lawrence Livermore National Laboratory; Marc Meyers, University of California-San Diego; George "Rusty" Gray, Los Alamos National Laboratory; Dan Thoma, Los Alamos National Laboratory

Wednesday PM Room: 3001
February 18, 2009 Location: Moscone West Convention Center

Session Chairs: Dan Thoma, Los Alamos National Laboratory; Timothy Weihs, Johns Hopkins University

2:00 PM Invited
Studying Nanoscale Material Processes under Extreme Conditions with High Time Resolution Electron Microscopy: *Thomas LaGrange*[1]; Geoffrey Campbell[1]; Patrice Turchi[1]; Bryan Reed[1]; Nigel Browning[1]; Wayne King[1]; [1]Lawrence Livermore National Laboratory

Often material's macroscopic properties and behavior under external stimuli can be described through observation of its microstructural features and dynamical behavior. Materials models and computer simulations that are used to predict material behavior in different environments, e.g., phase transformation kinetics under high pressure loading, typically require experimental data for validation or interpretation of simulated quantities. However, most materials dynamics are extremely rapid, making it difficult to capture transient, fine-scale features of the material process, especially on the length and time scale relevant for most simulations. In effort to meet the need for studying fast dynamics in material processes, we have constructed a nanosecond dynamic transmission electron microscope (DTEM) at Lawrence Livermore National Laboratory to improve the temporal resolution of in-situ TEM observations. The DTEM consists of a modified JEOL 2000FX transmission electron microscope that provides access for two pulsed laser beams. One laser drives the photocathode (which replaces the standard thermionic cathode) to produce the brief electron pulse. The other strikes the sample, initiating the process to be studied. A series of pump-probe experiments with varying time delays enable, for example, the reconstruction of the typical sequence of events occurring during rapid phase transformations. This presentation will discuss the core aspects of the DTEM instrument with particular focus on how it has been used to study martensitic phase transformations in Ti and "superheated" crystallization processes in amorphous NiTi films. Work was performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory and supported by the Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering, of the U.S. Department of Energy under contract No. DE-AC52-07NA27344.

2:35 PM
Coherent Electromagnetic Radiation Emission from Phase Transformations in Shocked CdSe: A New Ultrafast Materials Diagnostic: *Evan Reed*[1]; [1]Lawrence Livermore National Laboratory

Using molecular dynamics simulations coupled to Maxwell's equations, we show that the ultrafast transformation of wurtzite CdSe to the rocksalt phase under shock compression is accompanied by detectable electromagnetic radiation emission. The wurtzite to rocksalt transition can be accompanied by a change in static macroscopic material polarization which generates electrical currents that radiate. The radiation is in the 100 GHz frequency range, corresponding to the timescale of the onset of the phase transformation. The radiation amplitude is sufficiently large to be detected several mm away from the shocked material using existing THz frequency detection techniques. This work was performed in part under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

2:55 PM
Study of Crystallization of Amorphous Silicon Using Dynamic Transmission Electron Microscopy: *Shona McGowan*[1]; Liliya Nikolova[2]; Federico Rosei[2]; Bradley Siwick[1]; Mitra Taheri[3]; [1]McGill University; [2]Institut National de la Recherche Scientifique; [3]Drexel University

Amorphous silicon is an important material of particular interest in the context of thin film transistors. It also provides an ideal model system for the study of crystallization dynamics, and to this end we have investigated the crystallization of a thin amorphous silicon film in the Dynamic Transmission Electron Microscope (DTEM) at Lawrence Livermore National Laboratory. Crystallization is initiated by a laser pulse that deposits sufficient heat into the system to activate the transition. A variable time delay after this initiation pulse, the sample is probed by a short photoelectron pulse in the TEM, generating a diffraction pattern or image with nanosecond temporal resolution. Thus, using the DTEM we can gain access to the kinetics of crystallization and details of the nucleation mechanism in-situ through time-resolved diffraction patterns and images of the specimen. Results of this study will be presented.

3:15 PM
FIB and Electron Microscopy Movies of Extreme Materials Dynamism: *Warren MoberlyChan*[1]; Bassem El-Dasher[1]; Luis Zepada-Ruiz[1]; Graham Bench[1]; Scott Tumey[1]; Thomas Felter[1]; Alex Gash[1]; Hector Lorenzana[1]; [1]Lawrence Livermore National Lab

FIB and 2-beam-tools enable site-specific serial-sectioning at nanometer-scale. Movies and 3D-image-collection are versatile, but collection remains slower than sub-microsecond-timescales for extreme experiments. Piecing together nanostructural changes in 3D illuminates how nanosecond events occurred. The analogy is quenching series capturing thermal reactions by metallography. What happens when loading is so fast and so hard that <1 nanosecond is insufficient time for deformation mechanisms to engage? How can self-sustaining reactions, propagating >nanometer/nanosecond in an energetic material, be "stopped"? The reverse reaction always present on a free energy diagram, is statistically irrelevant at the macroscale but paramount at the picoscale, so what happens when the reverse reaction of deposition catches up to the primary reaction of erosion? Or the reverse? To understand and control processing at the nanoscale, materials metrology needs picosecond and picometer resolution. But until we achieve extreme in situ capabilities, FIB/EM movies help answer these extreme questions. (DOE-LLNL Contract-#-DE-AC52-07NA27344-UCRL-ABS-405723).

3:35 PM Break

3:50 PM Invited
Interface Mobility for Ti Alpha to Omega Transformation: *Dallas Trinkle*[1]; [1]University Illinois, Urbana-Champaign

While the shock-induced Ti alpha (hcp) to omega martensitic transformation has been studied extensively experimentally, atomistic-scale simulations of the transformation mechanism have only recently added new insight. Ab initio work generated and sorted through homogeneous transformations to find the TAO-1 pathway with an energy barrier more than four times lower than other transformations. Now, a new atomistic potential for Ti accurately describes phonons, surface and stacking fault energies for the alpha, omega, and beta (bcc) phases, as well as homogeneous transformation barriers for alpha to omega. The new potential determines the structure and mobility of glissile disconnections (interfacial line defects) in the alpha/omega interface for different transformation pathways under pressure. This provides crucial insight into the dynamics of the shock-induced transformation, and the important role of interfacial kinetics in strongly driven transformations. The transformation of alpha interstitial sites to omega helps elucidate the atomistic-scale behavior of oxygen on the transformation.

4:25 PM
Omega Phase Transition in Zirconium Based Alloys under High Pressure: *Raghvendra Tewari*[1]; Jyoti Gyanchandani[1]; Dinesh Srivastava[1]; Srikumar Banerjee[1]; Gautam Dey[1]; [1]Bhabha Atomic Research Centre

The ω phase under high pressure exists as an equilibrium phase in Group 6 metals. However, with pressures exceeding 30 GPa the ω-phase transforms to the β-phase in pure-Zr. This observation establishes the transformation sequence α-β-ω with increasing pressure. This paper reports that the formation of the ω phase in a β stabilized zirconium alloy under shock pressure condition. The plate shape morphology of the ω phase in the β matrix is akin to the martensitic phase. The mechanism for the formation of the plate shaped ω phase is explained on the

basis of a shear on <112> planes of the bcc lattice and the mechanical instability of the β phase. Similar platelet morphology of the ω phase was observed when pure-Zr was also subjected to shock pressure whereas the granular morphology was observed under static pressurization. Different mechanisms have been proposed to explain differences in the morphology.

4:45 PM

Dislocation Patterning and Formation of Omega Phase in Shocked Tantalum: *Luke Hsiung*[1]; Geoffrey Campbell[1]; [1]Lawrence Livermore National Lab

We present the results obtained from transmission electron microscopy studies of shocked tantalum, a group V transition metal that exhibits no clear solid-state phase transformation under static-pressure conditions, to investigate and verify the occurrence of shock-induced phase transformation under peak pressures above 30 GPa. Since the omega phase domains are frequently found in regions containing high-density dislocations with no cell-wall formation, we suggest that the shock-induced phase transformation in tantalum is nucleated through the catalysis of dislocations with a unique patterning configuration when the dynamic recovery process for cell-wall formation becomes suppressed under dynamic conditions. A strain-rate dependent dislocation mechanism based upon the clustering of closely spaced dislocation loops generated from a jogged screw dislocation is proposed to rationalize the shock-induced phase transformation. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

5:05 PM

The Influence of the Shape Memory Transformation on the Shock Dynamic Behavior and Phase Stability of U-6Nb: *Dan Thoma*[1]; Robert Field[1]; Ellen Cerreta[1]; Darcie Dennis-Koller[1]; George Gray[1]; Heather Volz[1]; Ann Kelly[1]; Robert Hackenberg[1]; Jason Lashley[1]; [1]Los Alamos National Laboratory

U-6wt%Nb is a disordered alloy that exhibits a thermoelastic phase transition at approximately 100°C during cooling and associated shape-memory behavior. Shock loading of the material using a variety of techniques, including explosive charges with water capture and gas gun testing, results in two distinct effects: 1) the high temperature austenitic phase is stabilized, even after soft capture, and 2) the material does not readily display a Hugoniot elastic limit. Dynamic gas gun testing has been performed at different temperatures to reveal the characteristics of the phase transition under various loading conditions. The phase stability in the virgin and recovered samples has been investigated through x-ray diffraction, TEM, and thermal analysis techniques. The results suggest that reversion of the martensite masks the Hugoniot elastic limit and that the high temperature phase is stabilized by residual strains under extreme (explosive) loading conditions.

5:25 PM

Phase Transformation and Microstructural Evolution of a NiTi Shape Memory Alloy at High Strain Rates: *Xiuhua Zheng*[1]; Shukui Li[1]; Benqiang Zhu[1]; [1]Beijing Institute of Technology

To get a better understanding of phase transformation and microstructural evolution of NiTi SMAs occurring during dynamic loading, an experimental investigation into the stress–strain response of a NiTi shape memory alloy with different specimen geometry was conducted using Split Hopkinson Pressure Bar (SHPB). The microstructures of tested specimens were studied using SEM, TEM and XRD. The influence of strain rate and specimen geometry on phase transformation behavior of the material at high strain rate was discussed and compared with the behavior at quasi-static strain rates.

5:45 PM

Stress-Induced Phase Transformation in Nanocrystalline UO2: *Tapan Desai*[1]; Blas Uberuaga[2]; Paul Millett[1]; Dieter Wolf[1]; [1]Idaho National Laboratory; [2]Los Alamos National Laboratory

We have performed Molecular Dynamics (MD) simulations using an empirical potential to study stress-induced phase transformation in nanocrystalline UO2 at T=1000K. The columnar UO2 microstructure consists of 6 grains of identical hexagonal shape and diameter (d=20 nm) in a three-dimensional periodic simulation cell. Under constant-stress tensile loading conditions, we found a phase transformation from the fluorite to a-PbO2 structure. The heterogeneous nucleation process of this new phase (a-PbO2) occurs at the grain boundaries and the new phase then grows toward the interior of the grain. To verify that this phase transformation seen in MD simulations is physically reasonable, density functional theory (DFT) calculations were performed. The DFT calculations agree that the a-PbO2 structure is energetically favored over the fluorite structure under certain tensile conditions. According to our knowledge, experimental validation of this phase transformation is not yet available. This work was supported by the DOE-BES Computational Materials Science Network.