# 2002 TMS Fall Meeting
## October 6-10, 2002

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# Technical Program
# 2002 TMS FALL MEETING
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## Corrosion in Reactors and Nuclear Power Systems: Corrosion in Reactors and Related Topics

*Sponsored by:* Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), SMD-Nuclear Materials Committee
*Program Organizers:* Robert J. Hanrahan, Los Alamos National Laboratory, Los Alamos, NM 87544 USA; David J. Senor, Pacific Northwest National Laboratory, Richland, WA 99352 USA

Monday AM — Room: 213
October 7, 2002 — Location: Columbus Convention Center

*Session Chair:* Robert J. Hanrahan, Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

**8:30 AM Invited**
**Corrosion Resistant Ceramic Coatings on Zircaloy Cladding**: *Darryl Butt*[1]; Michael Nastasi[2]; Ronald Baney[1]; James Tulenko[2]; Gerhard Fuchs[1]; Sung Rok Bang[1]; Yousif Al-Olayyan[1]; [1]University of Florida, Matls. Sci. & Eng., Gainesville, FL 32611 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS K765, MST-CMS, Eniwetok Rd., Los Alamos 87545 USA

An important materials issue in the operational lifetime of light water reactor (LWR) fuel elements is the waterside corrosion of the zirconium alloy cladding that encases the nuclear fuel. In particular, waterside corrosion currently limits the high burnup of nuclear fuel. The corrosion of Zircaloy is currently controlled in part through careful of LWR water chemistry and other environmental variables. In this presentation we will discuss in general, the corrosion behavior of Zircaloy under ambient and hydrothermal conditions, and the potential for using thin ceramic coatings for protection. In this presentation we will discuss the breakdown mechanisms of several coating systems including alumina, diamond like carbon, and silicon carbide. Factors that significantly affect the adhesion and breakdown mechanism include the roughness and pretreatment of the metal/ceramic interface, pre-existing defects in the coating, and coating thickness and residual stress. To date, silicon carbide proved to be the most effective coating for corrosion mitigation.

**9:00 AM**
**Microbially Influenced Corrosion (MIC) in Nuclear Power Facilities**: *James C. Earthman*[1]; [1]University of California-Irvine, Cheml. Eng. & Matls. Sci., ET916, Irvine, CA 92697-2575 USA

Microbially influenced corrosion (MIC) in nuclear power facilities will be reviewed. MIC refers to changes in rates and types of electrochemical reactions involved in corrosion processes due to the presence and activities of microorganisms in biofilms on metal surfaces. MIC can be manifest by changes in concentration of electrolytes, pH, and oxygen resulting in a broad range of outcomes from differential cell pitting and crevice corrosion to significant reductions in corrosion rate. Practically all engineering alloys are susceptible to MIC including those used in nuclear facilities. Mechanisms and case histories of deleterious MIC at facilities such as Three Mile Island Nuclear Power Plant will be discussed. Field studies of corrosion control using regenerative biofilms (CCURB) will also be reviewed.

**9:25 AM**
**Development of an Electrochemical Test Procedure for Detecting Susceptibility of Alloy 22 to Intergranular Corrosion**: *K. S. Raja*[1]; Denny Jones[1]; [1]University of Nevada-Reno, Metallurgl. & Matls. Eng., Reno, NV 89557 USA

Electrochemical Potentiokinetic Reactivation (EPR) is a rapid electrochemical test method (ASTM G108) for detecting susceptibility to intergranular corrosion (IGC) of type 304 and 304L stainless steels. For nickel base alloys, only cumbersome and time-consuming weight-loss measurements are available following aggressive chemical exposures (ASTM G28). The objective of this investigation has been to develop an EPR procedure to better quantify the IGC susceptibility of Alloy 22 (UNS N06022), a candidate corrosion-resistant alloy for high-level nuclear waste storage containers. Double loop EPR measurements are being applied to sensitized Alloy 22, using alternate activators such as thioacetamide (CH3CSNH2), aminothiourea (H2NHNCSNH2), and thiourea (H2C=CHCH2HNCSNH2) in place of conventional potassium thiocyanate (KSCN) used for the stainless steels. EPR results will be correlated with the ASTM G28 test results to develop a rapid electrochemical testing procedure for detecting IGC susceptibility of Alloy 22.

**9:50 AM**
**Experimental Observations of Stress Corrosion Cracking Behavior of Alloy 690 in High Temperature Hydrogen Supersaturated Steam**: *Mehboob M. Ali*[1]; Hugo F. Lopez[1]; [1]University of Wisconsin-Milwaukee, Matls. Dept., Milwaukee, WI 53201 USA

Nickel base alloys 600 and 690 have found widespread applications in nuclear reactor steam generators. In particular, alloy 690 with a major element composition of typically Ni-30Cr-9Fe, has shown an apparent performance when compared with alloy 600 in both primary water and caustic environments. Nevertheless, alloy 690 can also be susceptible of undergoing hydrogen induced stress corrosion cracking (SCC). However, the role of the microstructural state is not well defined, such as the effect of the presence or absence of carbide precipitation along grain boundaries on the overall susceptibility. In this work, self-loaded linear elastic fracture mechanics M-WOL specimens made of alloy 690 were tested in both, the solution annealed condition (minimal grain boundary carbide precipitation), and after a grain boundary carbide precipitation treatment. Tests were carried out at 320-400°C in an instrumented autoclave used to "in-situ" monitor crack growth rates. The environment used was high-pressure supersaturated hydrogen steam. The tested M-WOL specimens were loaded under various applied KI values (between 50-80 MPam1/2). This work discusses the kinetic and microstructural results found on the SCC susceptibility of alloy 690.

**10:15 AM Break**

**10:25 AM Invited**
**The Corrosion Kinetics of Structural Materials in Lead-Bismuth Coolant Systems**: *R. Scott Lillard*[1]; Pallas A. Papin[1]; Mary Ann Hill[1]; Christophe Valot[2]; Michael Paciotti[3]; Robert J. Hanrahan[1]; Patricia O. Dickerson[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-6, Los Alamos, NM 87545 USA; [2]University Bourgogne, Lab. Rech. Reactivite Solides, UMR 5613, Dijon F-21078 France; [3]Los Alamos National Laboratory, Physics, P-25, Los Alamos, NM 87545 USA

The dielectric properties (capacitive and resistive) of the oxide films formed on steels 316L (austenite), HT-9, and 440A (both ferric/martensitic steels) were examined in a static liquid lead-bismuth eutectic (LBE) bath. In some cases samples were pre-oxidized in air while others were oxidixed in LBE. In addition to these experiments, the dielectric properties of the oxide were monitored as a function of time during 800 MeV proton irradiation. In the absence of proton irradiation the capacitance of the pre-oxide formed on all samples in air decreased with exposure time while the real component of the impedance increased indicating the layer formed by the pre-oxidation process passivated the surface to some extent. However the protective nature of these oxides was found to be inversely related to temperature. In comparison, for samples freshly abraded and subsequently preoxidixed in LBE, dielectric measurements indicated that the oxide film formed on these surfaces was non-protecting at all temperatures studied. Irradiation of the SS 316L sample (after exposure to LBE for 2 hrs.) at approximately 80 nA showed a small increasing trend in oxide capacitance indicating a change in the transport properties during proton irradiation and, thus, a decrease in corrosion resistance. However, no catastrophic change in the oxide properties occurred and, hence, the ability of this oxide to protect the metal substrate from corrosion does not appear to fail catastrophically. Preliminary metallography of the samples revealed that the oxide on both SS 316L and HT-9 was a thick, dual layer film enriched in chromium with respect to the alloy composition.

**10:55 AM**
**Localized Corrosion and Thermal Stability Behavior of Alloy 22 Welds**: *Raúl B. Rebak*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., 7000 E. Ave., L-631, Livermore, CA 94550 USA

Nickel-based Alloy 22 was selected to fabricate the corrosion resistant barrier of the nuclear waste containers for the potential repository site at Yucca Mountain. Alloy 22 is nickel based and contains by weight–22% chromium, 13% molybdenum, 3% tungsten and 3% iron. As-welded Alloy 22 is less resistant to localized corrosion than the mill annealed wrought structure. In the wrought condition, this alloy is a single austenitic phase; however, if Alloy 22 is aged for more than 1 h at temperatures higher than approximately 600°C, second phases may precipitate in the austenitic matrix. This type of treatment could be representative of non-recommended welding practices. The resulting microstructure after high temperature aging is brittle and has a lower corrosion resistance in aggressive environments. Therefore, the second part of the work was to characterize the effect of aging temperature and aging time on the mechanical properties and corrosion resistance of aged materials. Changes in mechanical properties were measured according to ASTM E 8 and changes in the corrosion resistance were measured according to ASTM G 28, G 31 and G 61. Changes in corrosion behavior were measured both in aggressive environments and in multi-ionic aqueous environments that could be more representative of underground water.

**11:20 AM**
**Pellet Cladding Interaction Failures of Fuel Rods Due to Stress Corrosion Cracking and Their Mitigation in Water Reactors**: *Sheikh T. Mahmood*[1]; K. L. Murty[2]; [1]Global Nuclear Fuel, GE Vallecitos Nucl. Ctr., M/C V03, 6705 Vallecitos Rd., Sunol, CA 94586 USA; [2]North Carolina State University, Dept. of Nucl. Eng., PO Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloys are commonly used as cladding tubes in water reactors and are exposed to various aggressive environments while being under complex biaxial loading. One of the major problems in late 70s and early 80s has been the pellet cladding mechanical interaction (PCI) in the presence of fission gases such as iodine, cadmium, etc., which lead to premature failures of the fuel rods due to pin-hole type localized breach. The severity of the problem, in particular in boiling water reactors, led to the development of barrier cladding by coextrusion of Zircaloy-2 with an inner iodide zirconium that essentially eliminated the PCI-related failures. However, the substantially lower corrosion resistance of zirconium layer led to clad breach and failures by hydrogen uptake in lieu of PCI due to stress corrosion cracking. This resulted in the development of tubing with 4-layers of Zircaloy-2 and pure Zr, the so-called Triclad\@ with the various layers of Zircaloy-2 resistant to nodular and general corrosion. The paper briefly summarizes the various underlying factors of PCI-SCC that led to the development of PCI-resistant fuel cladding.

**11:45 AM**
**Physical Electrochemistry at 304 SS in 288°C Water**: *Young-Jin Kim*[1]; [1]GE Global Research Center, 1 Rsrch. Cir., Schenectady, NY 12309 USA

Characteristics of the oxide film formed on 304 SS have been the subject of investigations to understand corrosion processes in boiling water reactor (BWR) environments. The mechanism and kinetics of the corrosion processes can be altered by the chemical and physical properties of oxide films. This paper will describe the oxide structure and chemistry and the electrochemical polarization behavior of oxygen and hydrogen reactions on 304 stainless steel (SS) in high temperature to shed further light on the possible correlation of the intergranular stress corrosion cracking (IGSCC) and radioactive buildup on the oxide surface.

## General Abstracts - Chemistry & Physics of Materials

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Monday AM — Room: 224/225
October 7, 2002 — Location: Columbus Convention Center

*Session Chairs:* William A. Soffa, University of Pittsburgh, Matls. Sci. & Eng., Pittsburgh, PA 15261 USA; Ian Baker, Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA

**8:30 AM**
**Topological Model of 2-D Grain Growth–Evolution of the Distribution of Edges Per Grain**: *Burton R. Patterson*[1]; Alan P. Sprague[2]; Xin Deng[1]; Margarita M. Estrada[1]; [1]University of Alabama at Birmingham, Dept. of Matls. Sci. & Eng., 1530 3rd Ave. S., Rm. 254, BEC, Birmingham, AL 35294 USA; [2]University of Alabama at Birmingham, Dept. of Compu. & Info. Scis., 1530 3rd Ave. S., CH 129, Birmingham, AL 35294 USA

A curvature driven computer simulation of two-dimensional grain growth that monitors the number of different types of topological events and the corresponding grain areas swept by them has been developed. The numbers of grain switching events resulting in gain or loss of an edge on grains with different numbers of edges and the ratios of these events to the number of three-edged grains disappearing have been computed. These ratios have been used in a numerical simulation that predicts the form of the grain edge distribution. The overall ratio of the number of grain switching events to grain disappearances was found to be 1.3. The relative areas swept by grain boundaries due to grain disappearances, grain switching events, and general motion were approximately 2, 8 and 90%, respectively.

**8:55 AM**
**Excitons of the Structure in In(x)Ga(1-x)N and Their Properties**: *Dimiter G. Alexandrov*[1]; [1]Lakehead University, Dept. of Electl. Eng., 955 Oliver Rd., Thunder Bay, Ontario P7B5E1 Canada

Existence of new type of exciton called exciton of the structure is reported in this paper. Author uses new approach for determination of the electron band structures of both types In(x)Ga(1-x)N–wurtzite and zinc-blende. These electron band structures are basis for explanation of observed spectral shift in structures on In(x)Ga(1-x)N. It is found on this basis that the new exciton exists in In(x)Ga(1-x)N. The binding energy and hydrogen like energy levels of the exciton of the structure are determined. The photon-stimulated emission of these excitons is investigated. It is found that the current of these excitons is small and that they are localized quasi-particles having long lifetime. It is found that destroying of the excitons of the structure has place in their interactions with hetero-junction and these results are used as basis for successful explanation of the observed spectral shift in quantum well structures on In(x)Ga(1-x)N.

**9:20 AM**
**The Microstructural Location of Impurities in Polar Ice**: *I. Baker*[1]; D. Cullen[1]; R. Obbard[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Recently, we developed a technique to determine the microstructural location of impurities in ice that involves controlled preferential sublimation of uncoated ice in a low-vacuum SEM. This leaves behind impurities, in both the grain boundaries and the grain interiors, which can be identified using X-ray microanalysis. Ice specimens from GISP2 and Byrd Station were examined in a JEOL 5310LV LVSEM, operated at 10 kV, equipped with a PGT IMIX X-ray microanalysis system with a pure germanium, ultrathin-window detector. The ice specimens were kept at 160 K using a home-built cold stage. The ice cores showed impurities such as Cl, S and less-commonly Na, Ca and Mg throughout the ice. In the GISP2 ice, filaments consisting chiefly of NaCl, were observed in the grain boundaries. Several experiments were performed which showed that the filaments formed by coalescence of the impurities that were located in the grain boundary plane. This research was supported by US National Science Foundation grant OPP-9980379 and Army Research Office grant DAAD 19-00-1-0444.

**9:45 AM**
**Segregation, Transition and Drag Force at Grain Boundaries**: *Ning Ma*[1]; [1]Ohio State University, Matl. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

We investigate impurity segregation and transition at grain boundaries and the corresponding drag effect on grain boundary migration. A discrete lattice model of grain boundary segregation and its continuum counterpart (i.e., the gradient thermodynamic approach) are formulated for a prototype planar grain boundary in a regular solution. The model takes into account several physically distinctive terms, including concentration gradient, spatial variation of the gradient-energy coefficient and concentration dependence of the impurity-grain boundary interactions. Their effects on the equilibrium and steady-state impurity concentration profiles across the grain boundary, the low-high-segregation transition temperature and the corresponding drag forces are characterized. It is found that omission of these terms could result in a significant overestimate or underestimate (depending on the boundary velocity) of the enhancement of impurity segregation and drag force for systems of a positive mixing energy. Without considering these terms, much higher transition temperatures are predicted and the critical point is displaced towards much higher bulk solute concentration and temperature. The model predicts a sharp transition of grain boundary mobility as a function of temperature, which is related to the sharp transition of solute concentration of grain boundary as a function of temperature. The transition temperatures obtained during heating and cooling are different from each other, leading to a hysteresis loop in both the concentration-temperature plot and the mobility-temperature plot. These predictions agree well with experimental observations. The low-high-segregation transition behavior is also analyzed using Cahn's variational approach to critical point wetting.

**10:10 AM Break**

**10:35 AM**
**Mechanisms of Twinning in the Ferromagnetic Phase in Manganese-Aluminum Alloys**: *William A. Soffa*[1]; Cagatay Yanar[1]; Eric A. Stach[1]; Velemir Radmilovic[1]; Jorg M.K. Wiezorek[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The ferromagnetic L10-ordered intermetallic phase tau-MnAl appears near equiatomic compositions after appropriate heat treatment. This metastable phase, exhibiting high uniaxial magnetocrystalline anisotropy, is the basis for attractive magnetic properties in these materials. The technologically important properties of these alloys are highly structure-sensitive, depending strongly on the microstructure and defect structure produced by transformation from the high-temperature disordered epsilon-phase to the ordered tau-phase. The tau-phase product characteristically a plethora of crystal defects resulting from the phase transformation, including a profusion of twin boundaries and twin-related variants. Recently the nature of the transformation mode has been elucidated and as part of this study the mechanism of twin formation has been investigated in detail, using CTEM, HREM and in-situ hot-stage TEM. The results of this analysis will be discussed and support from the National Science Foundation (DMR) is gratefully acknowledged.

**11:00 AM**

**Diffusional and Displacive Modes of Transformation in the Formation of the Tau-Phase in Ferromagnetic Manganese-Aluminum Alloys**: *William A. Soffa*[1]; Cagatay Yanar[2]; Velemir Radmilovic[3]; Eric A. Stach[3]; Jorg M.K. Wiezorek[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA; [2]Carnegie Mellon University, Matls. Sci. & Eng., Roberts Hall, Pittsburgh, PA, USA; [3]Lawrence Berkeley National Laboratory, Natl. Ctr. of Electron Microscopy, Cyclotron Rd., Berkeley, CA, USA

The ferromagnetic L1o-ordered intermetallic phase tau-MnAl is the basis for attractive magnetic properties of these materials for potential advanced permanent magnet and thin film applications. The hysteresis behavior of this metastable phase is highly structure-sensitive, depending on the extraordinary microstructure and defect structure resulting from transformation of a high-temperature disordered hexagonal-phase to the ordered tetragonal tau-phase. Recent studies have shown that the isothermal transformation in bulk materials occurs predominantly via a diffusional akin to a "massive" transformation and that atomic attachment at the interphase-interfaces plays a key role in generation of the characteristic defect structure of resultant tau-phase. However, the previously reported displacive mode of tau-phase formation has been observed as a competing mode of transformation and the nature of this transformation will be discussed as well as implication in thermomechanical processing of these materials.

**11:25 AM**

**Characteristics of Some Defects in Lanthanum Hexaboride Crystal**: Shuqi Zheng[1]; [1]Shandong University, Sch. of Matls. Sci. & Eng., 73 Jingshi Rd., Jinan, Shandong 250061 China

In this paper, the inclusions and defects in lanthanum hexaboride (LaB6) were examined by transmission electron microscopy (TEM). It was shown that there exist defects such as inclusions, such as SiO2 and La5Si3, dislocation pileup and dislocation networks in LaB6 crystal obtained by sintering at the temperature of 2273K for holding time of 120min in Ar pressure of 800Torr under the pressure of 0.4-0.5GPa. The formation mechanism and characteristics of those defects were analyzed.

**11:50 AM**

**Synchrotron X-Ray Study of Phase Transformations in Superelastic NiTi**: *Raj Vaidyanathan*[1]; Chandrasen R. Rathod[1]; David C. Dunand[2]; [1]University of Central Florida, AMPAC/MMAE, Engr.-1 Rm. 381, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA; [2]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The stress-induced transformation of NiTi from an austenitic cubic phase to a martensitic monoclinic phase can result in tensile strains as high as 8% under applied stress. On unloading, the monoclinic phase becomes unstable and transforms back to the parent austenitic phase, with a concomitant macroscopic strain recovery that constitutes the superelastic effect. By recording diffraction spectra during mechanical loading, such reversible stress-induced transformations can be investigated as they occur. Previously, we have used neutron diffraction to investigate such behavior in centimeter-scaled, polycrystalline NiTi specimens. In this study, we use synchrotron x-rays at the Advanced Photon Source, Argonne National Laboratory to investigate millimeter-scaled, polycrystalline NiTi during external loading. The texture, strain and phase fraction evolution are described. Such measurements, while requiring shorter count times (order of minutes for synchrotron x-ray compared to hours for neutrons), link macroscopic and microscopic phenomena in superelastic alloys and are applicable to biomedical stent geometries.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Overviews

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Monday AM — Room: 216
October 7, 2002 — Location: Columbus Convention Center

*Session Chairs:* Ali Argon, Massachusetts Institute of Technology, Cambridge, MA 02139 USA; Wole Soboyejo, Princeton University, Dept. of Mechl. Aeros. Eng., Princeton, NJ 08544 USA

**8:30 AM**

**Percolation of Cleavage Through a Field of Grains at the Lower Shelf: Experiments and Models**: *A. S. Argon*[1]; Y. Qiao[1]; [1]Massachusetts Institute of Technology, Cambridge, MA 02139 USA

Experiments were carried out on the modes of advance of cleavage cracks through a field of randomly oriented grains at -20C, in the pure cleavage range of the lower shelf, in very large grain size Fe-2%Si alloy, permitting detailed observations of the chronology of percolation of a cleavage crack front across a field of grains. Utilizing previously developed model-based information on the peak resistance to fracture of high angle grain boundaries, with given tilt and twist misorientation across them, and detailed measurements of orientations of individual grains in the crack path of the samples, it was possible to develop percolation maps of cleavage through the field of grains that paralleled well the experimental observations. On the basis of these experiments and modeling, a simple expression is proposed for the effect of grain boundaries on the overall cleavage fracture resistance of low carbon steels in the lower shelf region. These finding indicate that high angle grain boundaries should increase the overall cleavage cracking resistance of polycrystalline steels in the lower shelf region by about a factor of 3, roughly independent of grain size.

**8:55 AM**

**Discrete Dislocation Modeling of Fatigue Crack Growth**: *Alan Needleman*[1]; Vikram S. Deshpande[2]; Erik Van der Giessen[3]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02912 USA; [2]Cambridge University, Dept. of Eng., Trumpington St., Cambridge CB2 1PZ UK; [3]University of Groningen, Dept. of Appl. Physics, Nyenborgh 4, 9747 AG Groningen, The Netherlands

A framework for analysis of crack growth under cyclic loading conditions is discussed where plastic flow arises from the motion of large numbers of discrete dislocations and the fracture properties are embedded in a cohesive surface constitutive relation. The formulation is the same as used to analyze crack growth under monotonic loading conditions, differing only in the remote loading being a cyclic function of time. Fatigue, i.e. crack growth in cyclic loading at a driving force for which the crack would have arrested under monotonic loading, emerges in the simulations as a consequence of the evolution of internal stresses associated with the irreversibility of the dislocation motion. Predictions for the fatigue threshold, for Paris law exponents and for short crack effects obtained within this framework are discussed.

**9:20 AM**

**Cracks and Notches: An Idea of John's that I'm Still Working On**: *David Taylor*[1]; [1]Trinity College Dublin, Mechl. Eng. Dept., Dublin 2 Ireland

Sitting in The Eagle pub in Cambridge in 1982, John Knott suggested to me an idea for a final-year project. By carrying out fatigue tests on cracks which had been previously blunted by applying a high load, we could investigate the transition from crack-like to notch-like behaviour. The project went well, but left a few unanswered questions, and twenty years later I'm still working on it! This paper summarises the subsequent work on high-cycle fatigue behaviour of stress concentrators, including size effects, complex three-dimensional shapes and welded joints. Some age-old critical-distance methods are brought up to date with the help of fracture mechanics, and it is argued that a critical-distance approach produces a theoretical model capable of predicting behavour in both cracks and notches, which is thus more generally applicable than LEFM.

**9:45 AM**

**Dimple Cleavage Transitions for Pre-Cracked Charpy-Type Specimens from Slip Line Fracture Mechanics**: *Frank A. McClintock*[1]; [1]Consultant, Rm. 1-304, 77 Massachusetts Ave., Cambridge, MA 02139 USA

Although transitions from dimple to cleavage with decreasing temperature in steel Charpy specimens are often associated with the crack initiation stage, such transitions are also seen in the crack growth stage at a given temperature. Furthermore, in Charpy specimens during crack growth there is often a reverse transition from cleavage back to dimple fracture. A model of such behavior is derived from a stressed-based deterministic cleavage criterion, along with plane strain slip line fracture mechanics. In practice, the statistics of desirably rare dimple to cleavage transitions during growth are also of concern. A prior statistical model for crack growth in a constant stress field is re-examined. It has a high coefficient of variation, as has sometimes been observed. Despite simplifications there does not yet appear to be a feasible way of deriving the probabilities of rare cleavage transitions in large structures from experimentally accessible data.

**10:10 AM**

**Effective Dislocation Free Zone and Effective Stress Intensity Factor Revisited for Mode III Crack**: *Johannes Weertman*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Camous Dr., Cook Hall, Evanston, IL 60208 USA

The non-redundant (geometrically necessary) dislocations that exist within the plastic zone that surrounds a crack tip in small scaling yielding shield the crack tip perfectly[1]. Perfect shielding occurs if the non-redundant screw dislocation density $\rho_N$ is given everywhere by the non-redundant dislocation density equation[2] in which $\rho_N$ is proportional to stress gradients. With perfect shielding the effective crack tip K is reduced to zero. Close to the crack tip $\rho_N$ is orders of magnitude larger than the redundant dislocation density $\rho_R$ ($\rho_R \approx$ (stress magnitude)$^2$). We have argued (most recently in Acta Mater, 2002) that where the non-redundant dislocations swamp in numbers the redundant dislocations the latter dislocations cannot pin in place the former dislocations. Thus near the tip $\rho_N$ is reduced to the level $\rho_N \approx \rho_R$, producing an effective dislocation free zone at the tip (R Thomson, J. Mat. Sci.1978; J. Weertman Acta Met. 1978). The shielding produced by these lost non-redundant dislocations is lost too and the crack tip no longer is perfectly shielded. Explicit expressions are found for $K_{eff}$. ([1]Chp 6; [2]Chp 2 of Dislocation Based Fracture Mechanics, J. Weertman)

**10:35 AM**

**Fatigue and Fracture of Polysilicon Micromechanical Systems**: *R. O. Ritchie*[1]; C. L. Muhlstein[1]; E. A. Stach[2]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA 94720 USA

Over the past few years, microelectromechanical systems (MEMS) have found extensive application in safety-critical applications, such as airbag accelerometers and inertial guidance systems. Brittle, silicon-based films are the dominant material system for these micron-scale mechanical components. In contrast to conventional aerospace and power system applications, the use of damage intolerant materials such as silicon to micromechanical applications have not been halted by concerns over durability and reliability. Due to the real potential for premature failure, the long-term use of brittle structural films such as silicon mandates the investigation of subcritical crack growth phenomena such as cyclic fatigue and stress-corrosion cracking. Recent studies using an electrostatically actuated (on-chip) fatigue characterization structure, have clearly demonstrated that thin (~2 to 20 m) films of silicon are susceptible to premature failure under cyclic loading conditions. Specifically, tests on notched cantilever beam specimens in both laboratory air and controlled environments at ~40 kHz have shown "metal-like" stress-life (S-N) curves for both mono and polycrystalline micron-scale silicon films, with the stress to cause failure after 109 to 1010 cycles being ~50% of the single cycle strength. Mechanisms for such behavior are presented based on the concept of a reaction-layer fatigue process, which are confirmed using direct high-voltage transmission electron microscopy. Methods to suppress such "thin-film silicon fatigue" are described using self-assembled monolayer coatings that restrict the formation of the native oxide on silicon.

**11:00 AM Break**

**11:15 AM**

**Applications of Quantitative Fractography to Spectrum-Load Fatigue Life Analysis of Aircraft Structures**: *Sami M. El-Soudani*[1]; [1]The Boeing Company, Phantom Works, 2401 E. Wardlow Rd., MC C078-0533, Long Beach, CA 90807-5309 USA

Methodology for pattern recognition of spectrum-load fatigue-striation fracture markings and correlation of such features with specific elements of a mission-mix flight-load block sequence is demonstrated for a baseline, aircraft wing, aluminum alloy 2219-T851. The developed methodology has been successfully used for validation and rigorous analysis in support of structural design development tests with fatigue fractures emanating from fastener holes in a wing lower cover rib attachment. Using a complete montage of scanning electron microscope images taken along the entire fracture path in the subject component simulation test and correlation of fatigue crack growth rates with intermittent (statistical) spot sampled rates deduced from local flight block spacing, it is shown that statistical sampling of flight-block crack-growth increments can accurately predict spectrum-load fatigue crack propagation life. This approach to test validation would considerably reduce the laborious nature of quantitative fractographic analyses in support of structural life demonstration tests. The investigation reported herein also shows other beneficial aspects of using quantitative fractography in support of structural design development testing, namely: (1) determination of fatigue crack propagation life of component-simulating test specimens with complex configurations, (2) quantification under spectrum load conditions of the effect of assumed initial flaw sizes on crack propagation life for non-destructive evaluation and inspection plan recommendation purposes, (3) deducing the associated fatigue crack initiation life, especially where surface processing (anodizing, plating, shot peening, etc.), or suspect surface conditions (assembly damage, load asymmetry, etc.) may prevail, (4) quantification of the effect of bolt hole pre-load on fatigue crack initiation life. Despite all these benefits there remains considerable uncertainties in spectrum load fatigue life prediction in other high strength materials (e.g. a typical landing gear alloy 300M steel), where unlike the aircraft wing lower cover, normally experiencing predominantly–tension spectrum, these materials may experience tension-compression, or even predominantly compression load spectra. The uncertainty is further complicated by the fact that often such high strength steels do not mark fatigue fracture surfaces as well as aluminum alloy 2219. A brief review of the sources of error in analytical model predictions of spectrum-load fatigue life is made focusing on: (a) Accumulative damage model assumptions with respect to relative contributions of tensile versus compressive portions of the spectrum and miner's linear damage rule (b) adopted cycle counting algorithms (peak counting, simple range, rainflow, range pair (or Loopin) codes), (c) whether the critical area for fatigue crack initiation experiences a strain-controlled or load-controlled stress singularity, (d) complexities associated with mean stress and dynamic load transfer effects, (e) material data base used, (f) surface processing and environmental complications (plating, shot peening, electrochemical interactions), and last but not least (g) the fatigue initiation mechanism (contact versus non-contact stress fatigue). Recommendations are made outlining areas in need of further research for improved spectrum-load fatigue life predictions.

**11:40 AM**

**Fracture Mechanics of Blunt Notches**: *Ted Smith*[1]; Douglas A. Scarth[2]; [1]Manchester University–UMIST, Matls. Sci. Ctr., Grosvenor St., Manchester M1 7HS UK; [2]Kinectrics, Inc., 800 Kipling Ave., Toronto, Ontario, M8Z 6C4 Canada

Linear elastic fracture mechanics of cracks is well established and is based on the stress field near a crack tip being described by the stress intensity factor, with crack extension occurring when the stress intensity factor is equal to a critical value, which is referred to as the fracture toughness of the material. This methodology has been applied to a wide range of materials and structures, with the fracture toughness being related to the micro-mechanistic fracture processes, often via the cohesive process zone representation of these fracture processes. The objective of our wide-ranging research programme is to extend the fracture mechanics methodology to blunt notches, so as to take credit for the blunt notch geometry, the strategy being to parallel, as far as possible, the methods that have been developed for cracks. It is shown that an appropriate characterizing parameter, analogous to the stress intensity factor for a crack, is the elastic peak notch-tip stress, with fracture initiating when the peak stress attains a critical value, which is related to the notch root radius and material parameters. This paper highlights some of the key features of the authors' current position regarding their work in this area.

**12:05 PM**

**Probabilistic Aspects of Brittle Fracture**: *J. F. Knott*[1]; [1]The University of Birmingham, Sch. of Eng. (Metall. & Matls.), Edgebaston, Birmingham, England B15 2TT UK

Statistical treatments of local brittle fracture stress and fracture toughness are treated at three levels: the micro-scale, the macro-scale and the meso-scale. Developments of the RKR/Curry-Knott models and other forms of "local approaches" are set in the context of a need to estimate credible lower-bound properties for safety assessments. A comparison is made between Weibull analysis of data and the use of a Normal distribution. The dangers in using non-physically-based extrapolation of data for spatially "heterogeneous" materials are explained and guidelines for analyzing such data are proposed. Application to the brittle fracture of silicon carbide and of optical fibres helps to clarify effects of variations in fracture stress, fracture toughness and "inherent" defect population.

## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Characterization - I

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Monday AM Room: 214
October 7, 2002 Location: Columbus Convention Center

*Session Chair:* Robert O. Ritchie, University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA

**8:30 AM**
**Characterization: The Key to Materials**: *Ronald Gronsky*[1]; [1]University of California, Matls. Sci. & Eng., 569 Evans Hall, Berkeley, CA 94720-1760 USA

Materials selection and design in intelligent engineering practice has always been intimately linked to microstructure. The more demanding the application, the more strict the microstructural requirements must be in order to assure successful in-service performance. This tenet of materials engineering continues to challenge the scientific community and to drive the development of new characterization methodologies with greater utility. Spatial resolution at the atomic level is now routinely claimed in both electron-optical and scanning-probe techniques, while energy resolution and temporal resolution are also advancing to support compositional profiling and dynamic studies with superior precision. This presentation highlights the recent historical development of microstructural and microchemical characterization methodologies applied to metallic, ceramic, and semiconducting materials used in modern engineering. In each case, the key attributes driving new materials developments are identified.

**9:00 AM**
**Microchemistry-Property Relationships in Metals and Alloys**: *David B. Williams*[1]; [1]Lehigh University, Matls. Sci & Eng., Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18015-3195 USA

Gareth Thomas' principal contribution to metallurgy and materials engineering has been his demonstration of the power of transmission electron microscopy (TEM) and related techniques to elucidate the relationships between the microstructure and properties of engineering materials. Through more than 40 years of such research, he has advanced the use of TEM techniques to the point where, today, any self-respecting physical metallurgy study requires a serious TEM component. While structure-property relationships remain at the core of Gareth's legacy, he has always been quick to leap on new TEM technologies to strengthen our understanding of materials properties. He was among the pioneers of the use of X-ray and electron spectroscopy in the TEM. This paper illustrates the use of such techniques to understand the mechanical failure of complex steels and other alloys, thus demonstrating that microchemistry-property relationships are as important as the classical microstructure-property relationships pioneered by Gareth.

**9:30 AM**
**Transmission Electron Microscopy Study of Precipitates and Solute Clusters in Al Alloys**: *Henny W. Zandbergen*[1]; Jianghua Chen[2]; [1]Delft University of Technology, Rotterdamseweg 137, Delft 2628 AL Netherlands; [2]Delft University of Technology, Natl. Ctr. for HREM, Rotterdamseweg 137, Delft 2628 AL Netherlands

The precipitation sequence for 6xxx alloys is: Super Saturated Solid Solution >> solute atomic clusters >> GP zones >> b? >> b?/B?-phase-> b (stable). Some authors consider the GP zones as GP1 zones while the b? is called GP2 zones. The structures of the b? phase and a phase occurring before b?, the pre-b? phase, were determined by a combination of electron diffraction and through focus exit wave reconstruction. The structure for pre-b? resembles that of the b? but with different positions of some of the Mg atoms along the needle direction. The space group of the new phase is C2/m, as for b?. We have also studied the solute atomic clusters in Al-Mg-Si-Cu alloys. The common feature of these clusters is that they do not have a well-developed three-dimensional crystalline structure. Atomic imaging by means of through focus exit wave reconstruction is therefore used to fulfill the tasks.

**10:00 AM**
**Temporal Evolution of Microstructures on a Nanoscale: Experiments and Simulations**: *David N. Seidman*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Cook Hall, Evanston, IL 60208-3108 USA

The temporal evolution of microstructures created by solid-state decomposition in multicomponent and multiphase systems is studied on an atomic scale employing three-dimensional atom-probe microscopy (3DAP) and kinetic Monte Carlo (MC) simulations. The confluence of these two techniques allows us to compare directly the experimental 3DAP results with the computational results of the kinetic MC simulations for similar numbers of atoms. Both techniques give a detailed atomistic picture of the temporal evolution of partitioning of elements between phases, segregation at the heterophase interfaces created as a result of decomposition, compositions of the matrix and precipitate phases, and precipitate dimensions and morphologies. Supplementary experimental studies are performed employing conventional and high-resolution electron microscopies for examining the microstructures on coarser length scales. Experimental 3DAP results are presented for both metal/ceramic and metal/metal systems. Experimental 3DAP and kinetic Monte Carlo results are given for model nickel-based superalloys (Ni-Al-Cr) with low and high supersaturations of aluminum and they are compared with one another in detail. In addition, the roles played by quaternary and quinary additions (Ta, Re, W) on the temporal evolution of the nickel-based superalloys are discussed. Research supported by the National Science Foundation and Department of Energy.

**10:30 AM Break**

**10:45 AM**
**Any Unresolved Metallurgical Mysteries?**: *Robert M. Fisher*[1]; [1]University of Washington, Matls. Sci. & Eng., Roberts Hall, Box 352120, Seattle, WA 98195 USA

Virtually all of the materials science topics that drew much attention in the mid-fifties, when Gareth Thomas was just embarking on a long and illustrious career, are now well understood or thought to be understood, and are not vogue in this century. The focus in this presentation will be on modern "text-book" renderings of several of the subjects that were of particular interest to this author. These include the pearlite transformation, yield points, dislocation pile-ups and stress localization, boron-enhanced hardenability, the "885" embrittlement phenomenon, and stree-accelerated growth of metal whiskers.

**11:15 AM**
**Design of Structural Alloys Using Second Phases**: *Nack J. Kim*[1]; [1]POSTECH, Ctr. for Adv. Aeros. Matls., San 31, Hyojadong, Pohang, Gyungbuk 790-784 Korea

In recent years, there have been ever increasing demands for energy conservation and environmental protection. Such demands place a great burden for materials scientists to develop high performance structural alloys which have better mechanical properties than conventional alloys. One obstacle is that the improvement in one property (e.g., strength) often results in the degradation of other properties (e.g., ductility, toughness). One of the ways of overcoming the obstacle is to utilize the beneficial effects of second phases. There are several factors to consider when one develops structural alloys using second phases; properties (hardness, ductility, elastic modulus, thermal stability), coherency (crystal structure, lattice spacing, orientation relationship), morphology (size, shape, distribution) and volume fraction of second phases. In this paper, fundamental aspects of the alloy design using second phases will be discussed along with examples of Fe-, Al-, Mg- and Ti-based alloys.

**11:45 AM**
**Laser Surface Modification of the Carbon Plain Steel with Chromium**: *Jan Piotr Kusinski*[1]; Agnieszka Elzbieta Woldan[1]; [1]Academy of Mining and Metallurgy, Metall. & Matls. Sci., 30 Mickiewicza Ave., Krakow, Malopolska 30-059 Poland

Laser surface alloying is a process of growing interest because it permits local surface modification that improves corrosive and wear resistance properties of the alloyed layer, whose depend on the surface quality, penetration depth, structure, chemical composition and homogeneity as well as its adhesion to the substrate. The paper describes the microstructure and properties (phase and chemical composition, microhardness, corrosive and wear resistance) of the laser alloyed with chromium surface layer. The microstructure and mechanical properties of the carbon plain steel and Cr coatings were investigated experimentally by using optical, SEM and TEM electron microscopy, quantitative analysis by X-ray spectroscopy and phase composition (XRD) and microhardness Vickers measurements. The surface alloyed layers varied in microstructure, zones depth and width, as well as Cr content according to the thickness of the predeposited Cr powder layer, bonding paint type and the process parameters (power and scanning velocity). The microprobe analysis showed that higher chromium content in alloyed zone resulted from the thicker power pre-coated layer as well as higher scanning velocity. Electron microscopy examinations show the dendritic structure of the melted zone or typically martensitic in dependence on applicated bonding paint type.

## Third Workshop on the Effects of Alloying Elements on the Gamma to Alpha Transformation in Steels: Session I

*Sponsored by:* McMaster Centre for Steel Research, TMS Materials Science & Manufacturing Division, ASM International, Jt. Phase Transformations Committee
*Program Organizer:* Gary R. Purdy, McMaster University, Department of Materials Science and Engineering, Hamilton, Ontario L8S 4L7 Canada

Monday AM — Room: 220/221
October 7, 2002 — Location: Columbus Convention Center

*Session Chair:* Gary R. Purdy, McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

**8:30 AM**
**Ferrite Transformation Below the TTT-Diagram Bay in an Fe-C-Mo Alloy**: K. M. Wu[1]; M. Kagayama[2]; *M. Enomoto*[2]; N. Maruyama[1]; T. Tarui[3]; [1]Formerly of Ibaraki University, now at University of Science and Technology, Beijing, China; [2]Ibaraki University, Dept. of Matls. Sci., Nakanarusawa-cho, Hitachi 316 Japan; [3]Formerly of Ibaraki University, now at Nippon Steel Corporation, Steel Rsrch. Labs., 20-1 Shintomi, Futtsu 293-8511 Japan

The Mo concentration at ferrite/austenite boundaries during the ferrite transformation below the bay temperature was measured by STEM-EDX in an Fe-0.28Mo-3Mo alloy. There was an increasing tendency of Mo concentration with the isothermal holding time. Under the assumption that the transformation stasis occurs when the carbon diffusion controlled fast growth of ferrite is terminated (followed by the alloying element diffusion controlled slow growth), the experimental fraction transformed at the stasis (including the data reported in other alloys) were compared with the calculation that incorporated the solute drag.

**9:15 AM**
**Reaction Time and Temperature Dependence of Mo Accumulation at Austenite:Ferrite Boundaries in an Fe-0.24% C-0.93% Mo Alloy**: *E. Humphreys*[1]; H. A. Fletcher[2]; A. J. Garratt-Read[1]; W. T. Reynolds[3]; G. R. Purdy[4]; H. I. Aaronson[5]; J. D. Hutchins[6]; G. D.W. Smith[6]; [1]Massachusetts Institute of Technology, Ctr. for Matls. Sci. & Eng., Cambridge, MA 02139 USA; [2]Corus Steel; [3]Virginia Polytechnic Institute and State University, Dept. of Matls. Sci. & Eng., Blacksburg, VA 24061 USA; [4]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada; [5]Monash University, Dept. of Physics & Matls. Eng. at Monash Univ. & Dept. of Matls. Sci. & Eng. at Carnegie Mellon Univ., Victoria 3800 Australia; [6]Oxford University, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

For the first time in any alloy steel, the time and temperature dependence of X accumulation (here Mo) at austenite:ferrite boundaries has been measured. A special STEM technique (Ikeda et al, 1991; Garratt-Reed et al., 1997) was used. The bay temperature, $T_b$, in this alloy lies just above 650°C. At 650°C, a moderate, time-independent accumulation, with little scatter in the data, was found. At both 630° and 610°C, on the other hand, the scatter was large and there appears to have been an increase in Mo accumulation with reaction time, apparently greater at 630°C. These results suggest that Mo accumulation at $T_b$ occurs mainly at immobile areas of austenite:ferrite boundaries, whereas below this temperature additional Mo accumulation occurs preferentially at kinks on risers of growth ledges. Extensive data scatter at the lower temperatures may derive from the long distance between growth ledges relative to the length of the STEM scan.

**10:00 AM Break**

**10:20 AM**
**Comparisons of Coupled-Solute Drag Effects in Various Fe-C-X Systems**: *W. T. Reynolds*[1]; H. I. Aaronson[2]; G. R. Purdy[3]; [1]Virginia Polytechnic Institute and State University, Dept. of Matls. Sci. & Eng., VA, USA; [2]Monash University, Sch. of Physics & Matls. Eng. at Monash Univ. & Dept. of Matls. Sci. & Eng. at Carnegie Mellon Univ., Victoria 3800 Australia; [3]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

An attempt to identify phenomena occurring during the proeutectoid ferrite/bainite reaction in Fe-C-X alloys that may be explained by the coupled-solute drag effect (C-SDE) has been made. These phenomena are listed in approximate order of increasing strength of this effect needed to produce them. (i) Ferrite growth kinetics are changed without altering ferrite morphology. (ii) A bay develops in the TTT-diagram for initiation of transformation, accompanied by a minimum in plate lengthening and allotriomorph thickening kinetics. (iii) Widmanstatten morphologies increasingly yield to thickened allotriomorphs. (iv) Allotriomorphs develop non-parabolic thickening kinetics; when the C-SDE increases allotriomorph thickening exhibits two or three stages. (v) Twin boundary allotriomorphs and "wrinkled ferrite" form in the bay region. (vi) Incomplete transformation appears at sub-bay temperatures. (vii) Degenerate ferrite forms below the bay. The C-SDE tends to increase with the Wagner interaction parameter, but is stronger when $\varepsilon_C^{\gamma} < 1$ than when it is > 1.

**11:05 AM**
**Decarburization of Alloy Steels: Modeling and Experiment**: A. Phillion[1]; J. Nakano[1]; D. V. Malakhov[1]; *H. S. Zurob*[1]; Hui Guo[1]; G. R. Purdy[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

Decarburization offers the possibility of the study of a "single-interface" transformation of austenite to ferrite. The austenite phase can be considered semi-infinite; the kinetic information so obtained is highly reproducible. The process is modelled for alloy steels under the limiting assumptions of interfacial paraequilibrium and full unpartitioned local equilibrium. Experimental results are presented for simple Fe-C-Ni alloys. The rate of thickening of a surface ferrite layer is closely parabolic, allowing the comparison of experimental kinetic results with model predictions.

## Corrosion in Reactors and Nuclear Power Systems: Corrosion Issues in Yucca Mountain

*Sponsored by:* Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), SMD-Nuclear Materials Committee
*Program Organizers:* Robert J. Hanrahan, Los Alamos National Laboratory, Los Alamos, NM 87544 USA; David J. Senor, Pacific Northwest National Laboratory, Richland, WA 99352 USA

Monday PM — Room: 213
October 7, 2002 — Location: Columbus Convention Center

*Session Chair:* Raul B. Rebak, Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Livermore, CA 94550 USA

**2:00 PM**
**Temperature and Ionic Concentration Effects on Corrosion Behavior of Medium Carbon Steels in Yucca Mountain Waters**: *Ahmet Yilmaz*[1]; Dhanesh Chandra[1]; Jaak Daemen[1]; Denny A. Jones[1]; Venugopal Arjunan[1]; Vinay Deodeshmukh[1]; [1]University of Nevada-Reno, Metallurgl. & Matls. Eng., MS 388, Reno, NV 89557 USA

Corrosion behavior of medium carbon steel rock bolts has been investigated by various methods such as potentiodynamic polarization, electrochemical impedance spectroscopy, and laboratory-immersion tests in simulated Yucca Mountain (YM) waters. Corrosion rates were determined for different concentrations (1x, 10x, 100x, and 500x) of dissolved ions under aerated and de-aerated aqueous environments at the temperatures in the range of 25 to 85°C. In aerated conditions, corrosion rates increased as the concentration changed from 1x to 10x. However, the corrosion behavior was altered for 100x and 500x, as the rates decreased with increasing temperatures. The decrease in corrosion rates at these higher concentrations was due to build-up of oxide precipitates on the specimen surface. At lower concentrations (up to 10x), uniform corrosion was observed for both aerated and deaerated environments, whereas localized corrosion and pitting for the higher ionic concentrations. Pitting potentials decreased with increasing concentrations and temperatures for both aerated and deaerated waters. In deaerated conditions, the rates increased slightly with increasing temperatures. The corrosion rates obtained by the three methods will be compared. This research is sponsored by the US Department of Energy, Yucca Mountain Project- Task 18 DE-FC08-98NV12081.

**2:25 PM**
**Review and Comparison of SCC Models Utilized for Long-Term Predictions at Yucca Mountain**: *Fengmei (Frank) Song*[1]; D. A. Jones[1]; [1]University of Nevada, Metallurgl. & Matls. Eng., Reno, NV 89557 USA

Boiling and evaporation in the near-field environment are expected to produce hot concentrated chloride solutions around the nuclear waste containers (NWC) for use in the Yucca Mountain Project (YMP). Under tensile stress in this potentially aggressive environment, the NWC alloys may suffer brittle stress corrosion cracking (SCC), which is a significant threat to the projected 10,000-year lifetime of the containers. Experimental work sponsored by the YMP on the SCC resistance of candidate alloys immersed in aggressive aqueous environments is limited to comparatively short times, and cannot predict container lifetimes beyond the experimental ranges. Fundamental deterministic modeling, derived from fundamental principles, allows the long-term prediction of SCC beyond the short-term data used for calibration. Modeling also can resolve complex simultaneous interactions between numerous SCC variables related to material, environment, and stress, which often cause discrepancies among experimental data obtained under nominally identical conditions. The Ford/Andresen SCC model, adopted for use by the YMP, is based on a film-rupture slip-dissolution mechanism. It was developed to explain, and has been correlated empirically with, extensive SCC data for austenitic stainless steels used in boiling-water nuclear reactors operating up to 300C. The YMP has requested development of "alternative" SCC models to confirm the F/A model. To this end, we have undertaken a comparison of the F/A model with the deterministic one proposed by MacDonald, which also is based on a film rupture mechanism with resultant anodic currents at the crack tip. Neither one in its present configuration considers the known anodic-current effects on the plasticity and consequent embrittlement of the substrate alloy. Efforts to resolve conflicts in these models and develop an improved deterministic SCC model for the YMP will be described in this presentation.

**2:50 PM**

**Peer Review Panel Assessment of the Planned Waste Package Materials for Disposal of High Level Nuclear Waste at Yucca Mountain**: Joe H. Payer[2]; John A. Beavers[3]; Thomas M. Devine[4]; *Gerald S. Frankel*[1]; Russell H. Jones[5]; Robert G. Kelly[6]; Ronald M. Latanision[7]; [1]The Ohio State University, Fontana Corrosion Ctr., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Case Western Reserve University, 10900 Euclid Ave., Cleveland, OH 44106-7204 USA; [3]CC Technologies Laboratories, Inc., 6141 Avery Rd., Dublin, OH 43016-8761 USA; [4]University of California-Berkeley, Dept. of Matls. Sci. & Eng., 11 Teodora Ct., Berkeley, CA 94720-1760 USA; [5]Pacific Northwest National Laboratory, 902 Batelle Blvd., PO Box 999, Richland, WA 99352 USA; [6]University of Virginia, Dept. of Matls. Sci. & Eng., Thornton Hall, Charlottesville, VA 22903 USA; [7]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 8-202, Cambridge, MA 02139-4307 USA

The US Department of Energy has recommended Yucca Mountain, Nevada as a geologic repository site for the disposal of high-level radioactive waste and spent nuclear fuel. Although the waste packages will not be immersed in water because the repository is well above the water table, water does permeate the mountain and corrosion of the waste packages will be possible. The current design utilizes a Ti Grade 7 drip shield and a corrosion-resistant outer canister fabricated from Alloy 22. This presentation will summarize the findings of a peer review panel that was convened to evaluate the technical basis for long-term performance of these materials exposed to the conditions of the proposed repository. The issues evaluated include the evolution of the environment on the waste package, uniform corrosion, localized corrosion, stress corrosion cracking, metallurgical stability, hydrogen embrittlement, and fabrication. The Panel concluded that the current waste package design is likely to meet the performance criteria for the repository if some technical issues are favorably resolved.

**3:15 PM**

**Continuous Passive-Current Measurements in Simulated Open-Circuit Conditions Using an Open Circuit Reference Electrode**: *K. S. Raja*[1]; Denny Jones[1]; [1]University of Nevada-Reno, Metallurgl. & Matls. Eng., Reno, NV 89557 USA

An open-circuit reference electrode (OCRE), passivated by dissolved oxygen in a reference half cell, replaces the conventional reference electrode in a potentiostatic circuit. The potentiostat controls the working electrode (WE) potential in the deoxygenated solution of the polarization cell at that of the passive corrosion potential of the metallurgically identical OCRE. Reference-cell and polarization-cell solutions are of identical electrolyte composition. The normally active WE in the deoxygenated solution is anodically polarized to the corrosion potential of the passive OCRE. The resultant anodic current measures the growth of the passive film on the WE during the noble drift of the passive OCRE corrosion potential. Corrosion potentials of the OCRE in the presence of dissolved oxygen and the required anodic passive-corrosion currents necessary to polarize the WE to the OCRE potentials have been measured simultaneously and recorded vs. time for type 304 austenitic stainless steel and nickel base Alloy22 (UNS N06022) in 1-N sulfuric acid. Current levels and decay kinetics were similar to those obtained from conventional constant-potential potentiostatic tests. Mott-Schottky measurements showed very similar passive film semiconducting behavior whether the film was formed by dissolved oxygen or by the potentiostat for both alloys. We conclude that constant potentiostatic passive-potential tests in deoxygenated solutions give a valid representation of passive corrosion behavior in aerated solutions.

**3:40 PM Break**

**3:55 PM**

**Retention of Selected Properties of Candidate Yucca Mountain Waste Container Alloys During Long-Term Aging**: *Michael Gustav Fahrmann*[1]; James R. Crum[1]; [1]Special Metals Corporation, Tech. Dept., 3200 Riverside Dr., Huntington, WV 25705 USA

Several material families have been proposed and are still being evaluated for corrosion-resistant containers for the final disposal of radioactive waste at Yucca Mountain, NV. In this work, the retention of critical properties of several candidate Ni-base alloys after long-term exposure to temperatures anticipated to exist at the storage site is being addressed. Specifically, retention of impact strength and corrosion resistance as evaluated per ASTM G28A upon aging is presented. Any microstructural changes were monitored by differential thermal analysis. This test program encompasses both base metals as well as some recommended weld metals, and is ongoing.

**4:20 PM**

**Environmentally Assisted Cracking Resistance of Alloy 22**: *Raúl B. Rebak*[1]; Kenneth J. King[2]; John C. Estill[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., 7000 E. Ave., L-631, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, 7000 E. Ave., L-532, Livermore, CA 94550 USA

The proposed nuclear waste container for the potential repository site in Yucca Mountain would contain an external layer of Alloy 22. Nickel-based Alloy 22 was selected due to its excellent resistance to general and localized corrosion in most industrial applications. Alloy 22 is also highly resistant to environmentally assisted cracking (EAC) or stress corrosion cracking (SCC), especially in chloride containing environments. The purpose of this research work was to characterize the EAC behavior of both welded and wrought Alloy 22 in a variety of aqueous electrolyte solutions, temperatures and applied electrochemical potentials. The resistance of Alloy 22 to EAC was evaluated using the slow strain rate technique (SSRT), constant deformation technique (U-bend) and the reversing DC potential drop technique (Compact specimens). Alloy 22 was found extremely resistance to cracking both in multi-ionic solutions and in concentrated chloride-containing solutions. Using SSRT, Alloy 22 was susceptible to EAC in one multi-ionic solution (SCW) at a potential of 0.3-0.4 V [SSC] and at temperatures higher than 70°C.

**4:45 PM**

**Environmental Effects on Hydrogen Permeation in Low Carbon Steel Sets Proposed for the Yucca Mountain Repository**: *Vinay Deodeshmukh*[1]; Venugopal Arjunan[1]; Dhanesh Chandra[1]; Jaak Daemen[1]; Denny A. Jones[1]; Ahmet Yilmaz[1]; [1]University of Nevada-Reno, Metallurgl. & Matls. Eng., MS 388, Reno, NV 89557 USA

Electrochemical studies have been performed on candidate steel sets to be used for supporting the Yucca Mountain (YM) emplacement drifts in which high-level nuclear waste will be stored. These steel sets may encounter corrosive environment due to dissolved ionic salts and the pH may vary depending upon the concentration of salts. To understand the hydrogen permeation behavior of carbon steels, experiments were conducted using Devanathan and Stachurski cell as per ASTM G-148. The anodic side of the Devanathan cell contained 0.1N NaOH, and the cathodic side contained YM waters. Environmental effects of change in electrolyte concentration in simulated YM waters (1x, 10x, 100x, and 500x) were investigated using this permeation method. It was found that the maximum permeation current (iP) decreased with the increase in ionic concentration from 1x to 500x indicating that these ions form a barrier film that inhibits the hydrogen permeation. As this environment contains bicarbonate and silicate as the major passivating species, experiments were performed to understand the effect of these ions in 3.5% NaCl solution. The hydrogen diffusion coefficients of these steels were determined in 0.1N NaOH solution. The effect of irreversible and reversible hydrogen trapping in as-received and cold worked samples was also investigated. The effects of environmental variables and trapping on permeation current (iP) in YM steel sets will be presented. This research is sponsored by the US Department of Energy, Yucca Mountain Project- Task 18 DE-FC08-98NV12081.

**5:10 PM**

**Effect of Bicarbonate and Silicate Ions on Passivation Behavior of Medium Carbon Rock Bolt Steels Proposed for the Yucca Mountain Repository**: *Vinay Deodeshmukh*[1]; Venugopal Arjunan[1]; Dhanesh Chandra[1]; Jaak Daemen[1]; Denny A. Jones[1]; Ahmet Yilmaz[1]; Scott Lea[2]; Mark Engelhard[2]; [1]University of Nevada-Reno, Metallurgl. & Matls. Eng., MS 388, Reno, NV 89557 USA; [2]Pacific Northwest National Laboratory, Environml. Molecular Scis. Lab., Richland, WA 99352 USA

The effect of bicarbonate and silicate on the passivation behavior of Carbon Steel in 3.5% NaCl solutions have been evaluated for candidate rock bolts for Yucca Mountain (YM) emplacement drifts in which high-level nuclear waste will be stored. These rock bolts may encounter corrosive environment due to the above mentioned dissolved ionic species present in the YM waters in different concentrations (1x, 10x, 100x, 500x). Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) studies were performed to evaluate combined effects of bicarbonate and silicate ions on corrosion inhibition of carbon steels in YM repository waters. Results show that these ions act synergistically in such a way that bicarbonate with silicate enhances the passivation tendency of carbon steel in chloride medium. The passivation current density (ip) decreased considerably in combined solutions as compared to individual ionic effects in the chloride solutions. Complementary impedance studies also showed that the combined effect of bicarbonate and silicate ions exhibit better corrosion resistance. Scanning electron microscopy and energy dispersive X-ray analyses and X-ray photoelectron spectroscopy were performed on passivated and corroded samples. The combined effect of bicarbonate and silicate ions on carbon steels will be compared with varying ionic concentration in YM waters will be presented. This research is sponsored by the US Department of Energy, Yucca Mountain Project- Task 18 DE-FC08-98NV12081.

## Forming and Shaping of Light Weight Automotive Structures: Materials & Evaluations - I

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

Monday PM — Room: 210
October 7, 2002 — Location: Columbus Convention Center

*Session Chair:* Dan Zhao, Johnson Controls Inc., Plymouth, MI 48170 USA

**2:00 PM Invited**
**Production and Properties of Advanced High Strength Sheet Steels for Automotive Applications**: *Kevin T. Bilkey*[1]; [1]Ispat Inland, Inc., R&D, 3001 E. Columbus Dr., E. Chicago, IN 46312 USA

Applications for Advanced High Strength Steels (AHSS) have been steadily increasing due to the need for reduced costs, increased safety, and reduced mass. AHSS grades have tensile strengths of 500-1500MPa, and include both dual phase and fully martensitic microstructures. This presentation will describe the variety of production techniques employed by Ispat Inland for the production of these grades as well as the properties of Advanced High Strength Steels.

**2:30 PM**
**Stamping Evaluation of Continuous Cast 5xxx Aluminum**: *Sooho Kim*[1]; Paul E. Krajewski[1]; Marc A. Cwik[2]; [1]General Motors Company, R&D, MC 480-106-212, 30500 Mound Rd., Warren, MI 48090 USA; [2]General Motors Corporation, Metal Fabrication Div., USA

The press formability of continuous cast (CC) 5xxx aluminum sheets was evaluated. The stamping comparisons of direct chill (DC) ingot cast and CC AA5754 and AA5182 aluminum sheets were performed using a rectangular pan die. No significant differences were observed in formability between the CC and DC cast materials. The dash panel for the General Motors EV1 electric vehicle was successfully stamped using AA5754 aluminum sheet produced from CC slab. Four different 5754-type materials were tested, each with a different thermomechanical processing history and/or composition. The successful parts were stamped from the material that had been cast to 20 mm thick slabs and hot rolled to final gage (2.0 mm). These trials indicate that CC 5xxx series alloys should be able to replace DC cast material without sacrificing press formability. This project was a part of the development program on Low-Cost Aluminum-Alloy Sheet Production (CRADA NO. LA95C10245-A001) under the Light Metals Division (LMD) of the US Automotive Materials Partnership (USAMP).

**2:55 PM**
**Effect of Annealing on the Mechanical Behavior of Ultra-High Strength Martensitic Sheet Steels**: *Sriram Sadagopan*[1]; Nassos Lazaridis[1]; Kevin Bilkey[1]; [1]Ispat Inland, Inc., R&D, 3001 E. Columbus Dr., E. Chicago, IN 46312 USA

Applications of tempered martensitic sheet steels in the automotive industry have been steadily increasing. During manufacturing processes, it is possible to selectively modify the properties of the steel product at specific locations, to locally improve its formability, without negatively affecting structural performance. This paper presents results from tensile and formability testing for fully tempered martensitic steels of tensile strengths ranging from 1300-1500 MPa as a function of annealing temperature for two different quench rates. The results show some interesting effects of the imposed thermal treatment on ductility and offer some guidelines on the use of these materials.

**3:20 PM**
**The Influence of Stress Relaxation and Anelastic Recovery on Springback**: *Richard E. Ricker*[1]; David J. Pitchure[1]; David Dayan[1]; [1]National Institute of Standards and Technology, Matls. Sci. & Eng. Lab., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899 USA

Recent advances in finite element modeling of springback has dramatically reduced the uncertainty in springback predictions. Further improvements in these predictions may result from developing a better understanding of the contributions of factors that appear to alter the elastic modulus of deformed parts such as texture changes, stress relaxation, and anelasticity. This paper reports on a study into the contributions of stress relaxation and anelasticity in springback as measured by a variety of techniques. The results indicate that stress relaxation and anelastic processes represent a significant portion of the observed springback and that accounting for these effects could reduce the uncertainty in springback predictions. The measurement techniques and metallurgical factors influencing the results will be presented and discussed.

**3:45 PM Break**

**3:55 PM**
**Superplasticity and Microstructural Evolution of Coarse-Grained AZ31 Magnesium Alloy**: *Yi Liu*[1]; Xin Wu[2]; [1]Wayne State University, Dept. of Mechl. Eng., 2100 Eng. Bldg., 5050 Anthony Wayne Dr., Detroit, MI 48202 USA; [2]Wayne State University, Dept. of Mechl. Eng., 2144 Eng. Bldg., 5050 Anthony Wayne Dr., Detroit, MI 48202 USA

Superplasticity of coarse-grained AZ31 magnesium alloy has been investigated at the temperature between 350-500°C with the strain rate 1´10-3 s-1-1 s-1. The maximum elongation of 320% has been obtained at 773K and a strain rate of 1´10-3 s-1. At the temperature above 400°C, elongation of near or above 100% can be obtained at highest strain rate within tested conditions. This elongation seems to be enough for some engineering use. During deformation, the initial coarse grains were refined from initial 300mm to 25mm when the stress reached its maximum. The grain size maintained 25-30mm dynamically in the proceeding deformation. EBSD technique was used to study the grain distribution during deformation. Observations on the surface of the samples deformed at different stage indicated that both dislocation slip in the grains and grain boundary sliding contributed to the deformation.

**4:20 PM**
**Weld Ductility in Aluminum Alloys–Comparison of TIG and Friction Stir Welded Sheets**: *Michael P. Miles*[1]; Brian J. Decker[1]; Tracy W. Nelson[1]; [1]Brigham Young University, Col. of Eng. & Tech., 265 CTB, Provo, UT 84602 USA

The use of stamped tailored blanks in the automotive industry is now well-established for steel, but is not common for aluminum alloys. While weld ductility in steel tailored blanks has been shown to be very good, recent work has shown that weld ductility can be quite poor for aluminum tailored blanks. In this study a series of experiments are performed, comparing tungsten inert gas (TIG) welded and friction stir welded blanks of 6061-T6 aluminum, in the thickness range of 0.050" to 0.100". Weld ductility will be measured using tensile tests, with welds in different orientations with respect to the rolling and welding directions of the material. Process parameters for both the TIG and friction stir welded specimens will be varied to obtain optimal weld ductility.

**4:45 PM** ***Cancelled***
**Effect of Large Shear Deformation on the Microstructure and Properties of Two As Cast 6000 Alloys**: *Mingdong Cai*[1]; Gordon Lorimer[1]; Nick Parson[2]; [1]University of Manchester/UMIST, Manchester M1 7HS UK; [2]Alcan International, Ltd., Kingston, Ontario K7L 5L9 Canada

**5:10 PM**
**High Strain Rate Superplasticity in TiN Particulate Reinforced Aluminum Matrix Composites**: *Shangli Dong*[1]; *Tsunemichi Imai*[1]; Liang Zhen[2]; Makoto Takagi[3]; [1]National Institute of Advanced Industrial Science & Technology, Inst. for Structl. & Eng. Matls., 2266-98 Shimoshidami, Moriyama-ku, Nagoya 463-8560 Japan; [2]Harbin Institute of Technology, Sch. of Matls. Sci. & Eng., 92 W. Dazhi St., Harbin 150001 China; [3]Aichi Institute of Technology, Toyota, Japan

The superplastic behavior of TiN particulate reinforced Al-Mg, 2014, 2017 and 1N90 (pure Al) composites were investigated at temperatures ranging from 773K to 923K and at strain rates from $10^{-3}$ to $10^{-1}$ $S^{-1}$. The TiN/Al composites were prepared by a powder metallurgy route as hot pressing at 773K and followed by hot extrusion at 773K. Further secondary processing techniques were also applied to produce fine grain structures within matrix of the composites. The influence of alloying elements and effect of the secondary processing on superplastic behavior of the TiN/Al composites were examined, and the optimized conditions to obtain superplasticity, especially the high strain rate superplasticity (HSRS) in the composites have been determined. The potential to perform near-net forming for TiN/Al components using HSRS mechanism is also discussed.

## General Abstracts – Aluminum & Magnesium Alloys and Phase Transformations

*Sponsored by:* TMS

*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Monday PM — Room: 224/225
October 7, 2002 — Location: Columbus Convention Center

*Session Chairs:* Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA; Jorg M. Wiezorek, University of Pittsburgh, Matls. Sci. & Eng., Pittsburgh, PA 15261 USA

**2:00 PM**
**Investigations and Evaluation of Cluster-Hardening Phenomena in Al(Zn-Mg-Cu) Alloys**: Jorg M. Wiezorek[1]; *Amal A. Al-Ghaferi*[1]; *Anirudha R. Deshpande*[1]; Huiping Xu[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 864 Benedum Hall, Pittsburgh, PA 15261 USA

Isothermal ageing of precipitation-strengthened Al-alloys (e.g. 2xxx Al(Cu-Mg) and 7xxx Al (Zn-Mg))at temperatures of about 130-150°C is characterized by two stages of hardness increase. The first stage of hardening occurs very rapidly (complete within ~60s), whereas the second stage occurs after much longer aging times (~100h). For Al(Cu-Mg) alloys previous studies have attributed the first hardening phenomenon to fine scale clusters of solute atoms that form rapidly, i.e. to a cluster-hardening phenomenon. The second stage hardening process was related to G.P. zones. The thermal signatures of the anisothermal aging behavior of 7xxx-Al (Zn-Mg-Cu) obtained by differential scanning calorimetry (DSC) exhibit features virtually identical to those of 2xxx-Al(Cu-Mg). Hence, it appears feasible that cluster hardening phenomena also occurs in 7xxx alloys. A series of systematic interrupted isothermal ageing treatments has been combined with DSC experiments and microstructural characterization by Transmission Electron Microscopy and Atom Probe Field Ion Microscopy. Hence, the dominant solid state mechanism responsible for hardening in each of the aging stages are determined and the applicability of the cluster hardening mechanism to 7xxx-alloys is ascertained.

**2:25 PM**
**Recrystallization Kinetics and Texture Evolution in Aluminum Alloy 1050**: *Mohammed Haroon Alvi*[1]; Bassem Samy El-Dasher[1]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The recrystallization kinetics and texture evolution of Hot-worked Aluminum Alloy 1050 (AA 1050) have been studied. Isothermally annealed samples of AA 1050 at 623 K were analyzed using micro-hardness and Electron Back Scatter Diffraction (EBSD) in an SEM. Recrystallization kinetics were obtained from the micro-hardness data using classical JMAK analysis and also from the EBSD data. A semi-automatic procedure for extracting fraction recrystallized from EBSD maps was tested. Texture evolution was studied by analyzing the EBSD data and measuring volume fractions for various texture components of interest as a function of time and fraction recrystallized. The variation in recrystallization kinetics as a function of the parent (deformed) orientation was examined to determine whether systematic variations occur.

**2:50 PM**
**In-Situ Quantification of the Effect of Solutes on Grain Boundary Mobility in Aluminum Alloys**: *Mitra L. Taheri*[1]; Hasso Weiland[2]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15217 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA

The effect of solute elements on grain boundary mobility and energy is examined in a group of specialty aluminum alloys through a combination of in-situ annealing and electron backscattered diffraction (EBSD) within a scanning electron microscope. Grain boundary energy is extracted from dihedral angles and the crystallographic boundary type. Mobilities are extracted from in-situ measurement of boundary migration during recrystallization. The experiments are motivated by the limitations of early solute drag theories and attempt to quantify the effect via measurement of boundary velocities and driving pressure (stored energy), ultimately obtaining an activation energy. This value is supported by complimentary measurements of stored energy using a differential scanning calorimeter. High-purity Al with Zr, Commercial-purity Al with Zr, and AlMg alloys with Zr are investigated and compared with results from recent publications. The mobility results are compared with the accepted peak of mobility for boundaries with a 40º<111> disorientation.

**3:15 PM Break**

**3:40 PM**
**Kinetics of Grain Shrinkage and Grain Boundary Roughness**: *K.-Y. Jung*[1]; A. D. Rollett[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Monte-Carlo simulation of recrystallization and grain growth was performed to study the evolution of grain area and grain boundary roughness as a function of grain boundary mobility, grain boundary energy, annealing temperature and time (Monte-Carlo Steps). The initial structure consisted of two grains with different orientation and varying grain boundary properties. The grain boundary roughness and the rate of area change of two shrinking grains were determined. The kinetics of grain shrinkage was analyzed in the context of Mullins' former work to ascertain that the simulated microstructure evolved based on heoretical prediction. Preliminary results confirmed that areas of grains decreased linearly and the rate of change of area was proportional to the mobility and the grain boundary energy. Further, the grain boundary roughness was calculated as the microstructure evolved during simulation. It was found that the roughness increased with increasing annealing time and annealing temperature. The influence of grain boundary mobility and grain boundary energy on roughness is currently under investigation.

**4:05 PM**
**Simulation of Kinetics and Morphology of Dendritic Growth and Application to Al-Mg and Al-Cu Alloys**: *Lazaro Beltran-Sanchez*[1]; Doru M. Stefanescu[1]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487 USA

A model based on the Cellular Automaton (CA) technique using first principles for the simulation of dendritic growth controlled by solutal effects in the low Péclet number regime was developed. The model does not use an analytical solution to determine the velocity of the solid-liquid (SL) interface as common in other models but solves the solute conservation equation subjected to the boundary conditions at the interface. Using this approach the model does not need to use the concept of marginal stability and stability parameter to uniquely define the steady state velocity and radius of the dendrite tip. The model indeed contains an expression for the stability parameter but the process determines its value. It is found that the stability parameter is not a constant, but rather changes with time and angular position during dendrite formation. The model proposes a solution for the evaluation of local curvature, which eliminates mesh dependency of calculations. The model is able to reproduce qualitatively most of the dendritic features observed experimentally, such as secondary and tertiary branching, parabolic tip, arms generation, selection and coarsening, etc. Computation results are validated in two ways. First, the simulated secondary dendrite arm spacing (SDAS) is compared with literature values for an Al-4wt%Cu alloy. Then, the predictions of the classic Lipton-Glicksman-Kurz (LGK) theory for steady state tip velocity, are compared with simulated values as function of melt undercooling for an Al-Mg alloy. Both comparisons are found to be in very good agreement.

**4:30 PM**
**Phase Changes in Shock-Loaded Ni-Ti**: *R. E. Hackenberg*[1]; D. C. Swift[3]; J. C. Cooley[1]; K. C. Chen[2]; D. J. Thoma[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G770, Los Alamos, NM 87545 USA; [2]California Polytechnic State University, Dept. of Matls. Eng., San Luis Obispo, CA 93407 USA; [3]Los Alamos National Laboratory, Physics Div., MS E526, Los Alamos, NM 87545 USA

Near-equiatomic Ni-Ti alloys have been shock-loaded to better understand the phase transformations in dynamically-loaded shape-memory alloys. The laser-driven method employed in this study generates shocks of nanosecond-scale duration, an important factor in being able to discern the mechanism of stress-assisted martensite formation. The resulting microstructural changes are characterized using x-ray diffraction, SEM and TEM. The formation (or dissolution) or martensite and intermetallic phases from the B2 austenite matrix, caused by the dynamically imposed stress and temperature states is of particular interest. Preliminary work has demonstrated the apparent dominance of thermal effects when the initial pressure is in the 10-20 GPa range. To better resolve the competition between stress and thermal effects, smaller peak stresses were employed, which gives a smaller temperature rise in the shocked region than previously. Additionally, the effect of different loading paths (direct laser shock vs. laser-driven flyer plate impact) is examined.

**4:55 PM**
**The Effect of Solute on the Anisotropy of Crystal-Melt Interfacial Free Energy**: *Ralph E. Napolitano*[1]; Shan Liu[2]; Rohit Trivedi[1]; [1]Iowa State University, Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA; [2]Iowa State University, Inst. for Physl. Rsrch. & Tech., Ames, IA 50011 USA

The anisotropy of solid-liquid interfacial free energy plays a critical role in morphological stability and the dynamic selection of solidification microstructures. Recent theoretical and experimental gains have been made toward the quantification of the orientation dependence of interfacial energy in single-component metals and dilute alloys. In this work, we examine the composition dependence of anisotropy in binary crystal-melt systems. Equilibrium shape measurements are presented for several binary alloys, and theoretical descriptions for solute-interface interactions are discussed. This work was supported by the Office of Basic Energy Science, Division of Materials Science, US Dept. of Energy, under contract No. W7405-Eng-82.

**5:20 PM**
**Liquation Cracking in Ultra-Light Magnesium Alloys**: *Wei Zhou*[1]; Tianzhong Long[2]; [1]Nanyang Technological University, Sch. of Mechl. & Production Eng., 50 Nanyang Ave., Singapore 639798 Singapore; [2]University of South Carolina, Mechl. Eng. Dept., Columbia, SC, USA

Magnesium alloys have high strength-to-weight ratio compared with other metals and excellent recyclability compared with plastics. Because of the increasingly stringent environment regulation, restricted natural resources and the demand for energy saving, the use of the "ultra-light" magnesium alloys is steadily gaining importance. This in turn has resulted in more demands on welding technology for solving various problems in joining magnesium components. Thin plates of magnesium alloys AZ91D and AM60B were butt-welded using TIG welding method. The TIG arc was also used to deposit welding beads on some of the thin plates. No cracking was found in the butt joints. However, cracking was always observed to propagate from the heat affected zone under the welding bead into the weld metal right after a welding bead was deposited on the thin plate. Metallographic and fractographic evidence was obtained to show that the cracking is liquation cracking in the partially melted zone under the high thermal stresses.

**5:45 PM**
**Corrosion Behavior of Near-Nanocrystalline 5053 Al and Al-7.5%Mg**: C.-C. Wu[1]; Enrique J. Lavernia[1]; *James Calvin Earthman*[1]; [1]University of California, Irvine, Cheml. Eng. & Matls. Sci., Eng. Tower 916, Irvine, CA 92697-2575 USA

Corrosion experiments were performed on conventional and near-nanocrystalline (NNC) 5053 Al and Al-7.5%Mg alloys produced by cryomilling. The samples were tested in simulated seawater using electrochemical impedance spectroscopy (EIS). Two time constants were detected, one of which is governed by the integrity of passive film on the surface. The shape of the curves for the alloys tested are similar to that for conventional 5083 Al in terms of the number of time constants present. However, it appears that the polarization resistance for NNC Al-7.5Mg is over a factor of two greater than that for the conventional and NNC 5083 Al samples. DC polarization and electrochemical noise data will also be compared between the conventional and near-nanocrystalline alloys.

## General Abstracts – Mechanical Metallurgy and Composite Materials

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

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|---|---|
| Monday PM | Room: 222/223 |
| October 7, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Raymond Buchanan, University of Tennessee-Knoxville, Knoxville, TN 37996 USA; Raghavan Srinivasan, Wright State University, Mechl. & Matls. Eng. Dept., Dayton, OH 45324 USA

**2:00 PM**
**Microsample Tensile and Fatigue Testing of LIGA-Ni MEMS Materials**: Cristian Cionea[1]; Kevin J. Hemker[1]; [1]Johns Hopkins University, Dept. of Mechl. Eng., 3400 N. Charles St., Latrobe 200, Baltimore, MD 21218 USA

Process optimization and long-term reliability of MEMS devices can best be modeled when the microstructure-property relations of the materials employed in these devices are well understood. Room-temperature indentation and micro-tensile measurements of LIGA Ni structures have been reported, but a full balance of mechanical properties have not been forthcoming. The present study was undertaken to characterize the fatigue performance of these materials. Preliminary fatigue results indicate that fatigue life decreases with increased stress amplitude above a fatigue limit of approximately 200 MPa. However, the samples in these preliminary tests all fractured at a stress concentration associated with the shoulder of the specimen, indicating that specimen geometry plays an important role in determining fatigue strength. New microsample fatigue specimens have been designed and fabricated to follow the ASTM standard (E466-96) in order to minimize the importance of stress concentrations. The fatigue behavior of these ASTM specimens will be reported, and the importance of both specimen geometry and internal microstructure on the fatigue resistance of LIGA Ni structures will be discussed.

**2:25 PM**
**A New Testing Method for Evaluating Die Wear During Manufacturing Operations that Involve Punching of Sheet Materials**: *Roopa Narayan*[1]; Raghavan Srinivasan[1]; [1]Wright State University, Mechl. & Matls. Eng. Dept., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA

Manufacturing operations such as piercing, cutting or slitting on sheet materials can result in the wear of blades and dies. A method for measuring the wear is proposed. This method involves penetrating a sheet sample multiple times with a needle. The force required to make the punches depends upon the geometry of the needle. Changes in the geometry of the needle due to wear changes the punching force. The trend of the force is used to measure the potential wear that a sheet material will cause to the manufacturing tools. Several different materials including metal foils, polymer sheets and paper were tested using this method. Results from these tests as well as the wear observed in the punching needles will be presented.

**2:50 PM**
**Characterizing the Mechanical Behavior of MEMS Serpentine Springs**: *Carolina L. Elmufdi*[1]; K. J. Hemker[1]; [1]Johns Hopkins University, Dept. of Mechl. Eng., Baltimore, MD 21218 USA

Electroplated LIGA Ni structures offer higher aspect ratios and greater functionality than traditional vapor deposited thin films. The performance of LIGA Ni thermal actuators and springs are dependent on the elastic stiffness and strength of the material in its as-deposited state. The reduced size, development of texture and importance of crystallographic orientation may be expected to play an important role in determining the mechanical performance of these devices. The current study combines microsample tensile experiments with finite element (FE) modeling of the elastic response of LIGA Ni serpentine springs with lateral dimensions on the order of 10 microns. Measured and predicted values of spring constants and failure strengths will be presented and the importance of factors such as: spring geometry, grain size and crystallographic texture will be discussed.

**2:50 PM *Cancelled***
**High Cycle Fatigue Corrosion Investigation of a Nickel-Based Alloy**: *Rejanah V. Steward*[1]; Raymond A. Buchanan[1]; Peter K. Liaw[1]; Doug Fielden[1]; D. Klarstrom[2]; [1]University of Tennessee-Knoxville, Knoxville, TN 37996 USA; [2]Haynes International, Inc., Kokomo, IN 46904-9013 USA

**3:15 PM Break**

**3:40 PM**
**Thermal Stir Welding–A New Solid State Welding Process**: *R. Jeffrey Ding*[1]; [1]NASA, Marshall Space Flight Ctr., ED33, Huntsville, AL 35812 USA

Thermal stir welding is a new welding process developed at NASA's Marshall Space Flight Center in Huntsville, AL. Thermal stir welding is similar to friction stir welding in that it joins similar or dissimilar materials without melting the parent material. However, unlike friction stir welding, the heating, stirring and forging elements of the process are all independent of each other and are separately controlled. Furthermore, the heating element of the process can be either a solid-state process (such as a thermal blanket, induction type process, etc), or, a fusion process (YG laser, plasma torch, etc.) The separation of the heating, stirring, forging elements of the process allows more degrees of freedom for greater process control. This paper introduces the mechanics of the thermal stir welding process. In addition, weld mechanical property data is presented for selected alloys as well as metallurgical analysis.

**4:05 PM**
**Thermal Fatigue Resistances of the HSLA Cast Steels with Variations of Alloy Elements**: *Jai-Hyun Park*[1]; Young-Sub Kim[1]; [1]RIST, Reliability Assessment, PO Box 135, Pohang 790-600 Korea

Thermal fatigue resistances of the HSLA(High Strength Low Alloy) cast steels affected by additions of alloying elements have been investigated. The thermal fatigue resistances of the HSLA cast steels were superior to those of C-Mn(SC42) cast steels, and which were mainly caused by high thermal conductivity, elastic modulus and tensile strength. In case of the HSLA cast steels, the steels with both Nb and V had more excellent thermal fatigue life than those with Nb or V individually. Increment of C contents gave a harmful effect on thermal fatigue resistance. In case of Mn contents, HSLA cast steels with 1.2% Mn content had the highest thermal fatigue life among them and that with 1.5% Mn had lower thermal fatigue life than that with 1.2% Mn content. The optimum composition of HSLA cast steels to obtain the highest thermal fatigue resistance was 0.1%C-1.2%Mn-0.05%Nb-0.05%V.

**4:30 PM**
**3D Modeling of Alumina-Based Composite Structures with Finite Element Technique**: *Maksim V. Kireitseu*[1]; [1]Institute of Machine Reliability, Mech. of Composites, Lesnoe 19-62, Minsk, Belarus

A finite element method is used for the two-dimensional dynamic analysis of structural systems based on alumina-chrome carbide composite coating. The governing equation for the motion is derived from conservation of energy relations, while the eigen problem is derived from free vibration theories. The solution to the eigen problem is obtained through subspace iteration, resulting in a reduced set of natural frequencies and mode shapes for the system. The time response is found using modal analysis and superposition; the overall response is obtained by exploiting the piecewise continuity of the system over its elements. A brief discussion of error and proper finite element discretization is included. In the paper, the basic concepts of vibration and the finite element method are reviewed, and subsequently a solution method is derived in an explicit, step-by-step fashion. Wherever possible, the derivation steps are accompanied by an intuitive, plain explanation of their physical basis (i.e. the effect on the system). Once the solution method is obtained, a suitable transformation to computer code will be discussed in brief, and an outline of the included solver program presented. Lastly, the application of the solver to various dynamic systems will be considered, including the limitations of the program, assumptions made, and interpretation of output. Sources of error are explored, and possible accuracy improvements are suggested. A brief discussion of acceptable finite element convention, concerning error reduction, is also given.

**4:55 PM**
**Study of Wear Resistant MoSi2-SiC Composites Fabricated by Self-Propagating High Temperature Synthesis Casting**: *La Pei Qing*[1]; [1]Chinese Academy of Sciences, Lanzhou State Key Lab. of Solid Lubrication, Lanzhou Inst. of Cheml. Physics, Lanzhou 730000 China

MoSi2 composites with 10, 15 and 20 wt.% SiC as wear resistant materials were fabricated by self-propagating high temperature synthesis (SHS) casting route and analyzed with X-ray diffraction (XRD) and scanning electron microscopy (SEM) with X-ray energy dispersive spectroscopy (EDS). Hardness, toughness and wear rate of the composites were measured. The results showed the composites mainly consisted of MoSi2, SiC and Al2O3 phases and were dense. Most of SiC phase in the composite with 10 wt.% SiC were large particles, some SiC phase in the composite with 15 wt.% SiC were large particles and the others were groups of short fibers and all of SiC phase in the composite with 20 wt.% SiC were short fibers. Bonding interfaces between the SiC phase and MoSi2 matrix were well in the composites. There were small cracks in the matrix near interface between the large SiC particles and the matrix in the composites with 10 and 15 wt.% SiC but no small cracks were observed in the matrix of the composite with 20 wt.% SiC. Content of Al2O3 phase was about 3 wt.% in the composites. Hardness and toughness of the composites increased with SiC content and those of the composites with 15 and 20 wt.% SiC were higher than that of the monolithic MoSi2. Wear rate of the composites dramatically decreased with SiC content and those of the composite with 15 and 20 wt.% SiC were lower than that of the monolithic MoSi2 and the composite with 20 wt.% SiC had the best wear properties.

the order of 10 microns. Measured and predicted values of spring constants and failure strengths will be presented and the importance of factors such as: spring geometry, grain size and crystallographic texture will be discussed.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Fracture and Fatigue of Materials

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Monday PM — Room: 216
October 7, 2002 — Location: Columbus Convention Center

*Session Chairs:* John Lewandowski, Case Western Reserve University, Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA; Alfred Soboyejo, The Ohio State University, Dept. of Food, Columbus, OH 43210 USA

**2:00 PM**
**Diffusion-Controlled Brittle Fracture**: *Charles J. McMahon*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

In contrast with the usually discussed forms of brittle fracture in metals, either transcrystalline or intercrystalline, which propagate at rates approaching the speed of sound, there exists a form in which cracks propagate quasi-statically at rates that are orders of magnitude lower. In this case, surface-adsorbed embrittling elements are induced to diffuse inward, usually along grain boundaries, and decohesion can occur if the applied stress is high enough. Examples include stress-relief cracking in steels, in which surface-adsorbed sulfur is to blame, and oxygen-induced cracking in nickel-based superalloys. Research in this area, known as dynamic embrittlement, over the past several years will be reviewed, and several methods for retarding the cracking will be discussed.

**2:25 PM**
**Simulating Void Growth and Coalescence During Ductile Fracture**: *Donald A. Koss*[1]; James Bandstra[2]; [1]Pennsylvania State University, University Park, PA 16802 USA; [2]University of Pittsburgh at Johnstown, Johnstown, PA 15904 USA

Tensile fracture of ductile metals typically occurs by microvoid growth and coalescence. In this study, we utilize a novel specimen geometry to model the growth and coalescence among clusters of two and three neighboring spherical cavities. Experimentally, we have employed tensile specimens containing either two or three blind-end holes with hemispherical ends to determine the ligament-thinning behavior between the cavities formed by the hole ends. The central idea is that the strain-induced thinning behavior of the inter-cavity ligaments is similar to that of the ligament between neighboring microvoids located a similar relative distance apart. The blind-end hole results were used to validate a three-dimensional finite element analysis, which in turn was then extended to examine the strain-induced growth behavior within a cluster of three spherical cavities. Those results will be compared to the strain-induced growth of isolated voids, and implications to ductile fracture will be discussed.

**2:50 PM**
**Environmental Effects on Fatigue Behavior of Type 316 Stainless Steel for the Application of Spallation Neutron Source**: *Hongbo Tian*[1]; Peter K. Liaw[1]; Joe P. Strizak[2]; Lou K. Mansur[2]; [1]The University of Tennessee-Knoxville, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

The high-cycle fatigue behavior of Type 316 stainless steel (SS), the prime candidate target-container material for the Spallation Neutron Source (SNS), was investigated in air and mercury at frequencies of 0.2 and 10 Hz with an R ratio of -1, and 10 and 700 Hz with an R ratio of 0.1. Here R equals the ratio of the applied minimum load to maximum load during fatigue experiments. A decrease in the fatigue life of 316 LN SS in mercury was observed, relative to that in air, at 0.2 Hz. The decrease in the fatigue life is interpreted as due to liquid metal embrittlement (LME) in mercury. Correspondingly, intergranular fracture was found on the fracture surface for specimens tested in mercury at 0.2 Hz, which is a typical fracture mode caused by LME. In the fatigue tests at 700 Hz, the fatigue lives of 316 SS in air and mercury were comparable, indicating no effect of the mercury. Heating by mechanical working was observed during fatigue tests at 10 Hz and R of -1, which resulted in great increases in temperature for large stress amplitudes. Theoretical modeling of temperature evolution during fatigue is provided. The provided and measured temperature evolutions were found to be in good agreement.

**3:15 PM**

**Ambient Temperature Time-Dependent Sub-Critical Crack Growth in Aluminum Alloys**: *Sharon L. Johnson*[2]; John J. Lewandowski[1]; Terry Evans[3]; Henry Holroyd[2]; [1]Case Western Reserve University, Dept. Matls. Sci. & Eng., Cleveland, OH 44106 USA; [2]Luxfer, USA, Riverside, CA, USA; [3]University of Newcastle, Newcastle, UK

Aluminum alloys can suffer a 'spectrum' of time-dependent sub-critical crack growth mechanisms at ambient temperatures, ranging from stress-strain controlled processes independent of the environmental conditions (e.g. Sustained load cracking in Al-Mg-Si alloys) through to processes controlled by the environmental conditions and independent of the local stress-strain conditions (e.g. intergranular corrosion). Relative susceptibilities and temper dependencies for these crack growth mechanisms are generally alloy system and alloy temper dependent. This paper critically reviews these dependencies in terms of proposed crack growth mechanisms, identifying commonalities and differences. Specific instances are highlighted where a 'conventionally' deemed highly resistant or highly susceptible alloy system/temper can behave unexpectedly, e.g. a commercial Al-Mg-Si alloy (AA6061) suffering stress corrosion cracking or a high strength commercial Al-Zn-Mg-Cu alloy (AA7032) being 'practically' immune to stress corrosion cracking.

**3:40 PM**

**Nanoindentation of Alumina-Chrome Carbide and Alumina-Ultra Dispersed Diamonds Composite Coatings**: *Maksim Kireitseu*[1]; Sergey Yerakhavets[1]; [1]National Academy of Sciences, Dept. of Mech. & Trib., Lesnoe 19-62, NAMATEX Sys. Div., Inst. of Machine Reliability, Minsk 223052 Belarus

Nanoindentation experiments have been performed on electrophoretic-deposited films of alumina-chrome carbide and alumina-ultra dispersed diamonds composite coating on different substrates like steel, aluminum and corundum. Films with thicknesses between 60 and 300 µm prepared at various current intensities were indented with spherical indenters with nominal radii of 10, 50, and 150 µm. Force-displacement data were analyzed to determine contact pressure and elastic modulus versus depth results. The modulus and contact pressure behavior with depth exhibited opposite trends with indenter radius: the modulus increase was least for the 10 µm and greatest for the 150 µm, whereas, the contact pressure was the inverse. The results may be rationalized by plotting modulus normalized to the ratio "contact radius/film thickness" (a/t), whereas the contact pressure results at small alt could be normalized when plotted versus contact strain, i.e., contact radius divided by indenter radius (a/R). These approaches enabled the properties of the variously deposited films to be compared. Additional interesting microstructural and cracking behavior patterns are also discussed. The influence of deposition current and drying conditions were investigated using SEM. The suitability of this technique to determine morphology and the use of small spherical-tipped indenters to evaluate the mechanical properties of powder compacts was established. The revealed results may be summarized as follows: (1) The structure, grain size and morphology strongly depend on deposition current although the film density does not. At low current intensity, the grain size is found to be close to the initial particle size, whereas at higher current intensity an apparent coarser grain size occurs that, however, also contains pores and internal voids. (2) It is expected that the higher over potential results in the coarser grain size and formation of pores at higher current intensity that caused the onset of electrolysis of the aqueous medium. The localized electrolysis and oxygen evolution near the anode, resulting in a localized change in pH of the aqueous suspension, produced particle agglomeration as well as bubble formation. (3) Spherical indentation technique is found to be effectively measured contact pressure and effective elastic modulus as a function of penetration depth. The difference in contact pressure and elastic modulus vs. the ratio of the contact radius to the film thickness. The errors in the data could also be associated with radial cracks within the contact area. (4) The influence of deposition current indicates that higher current intensities and lower (10%) humidity resulted in higher contact pressure at constant contact strain. The influence of humidity was associated with denser films resulting from higher compressive stresses during faster drying. (5) Evidence of residual tensile stresses within the film manifested itself in the form of radial cracks from pores. The thicker films showed a greater influence of the cracks.

**4:05 PM Break**

**4:20 PM**

**Ductile to Brittle Transition in C-Mn Weld Metals**: *Milorad Novovic*[1]; [1]University of Birmingham, Structl. Matls. Rsrch. Ctr., Sch. of Eng., Birmingham B15 2TT UK

The paper presents the results of an extensive experimental programme on Charpy impact energy of a carbon-manganese (C-Mn) pressure vessel steel. Charpy specimens were machined from multipass submerged arc weld metal with various notch tip locations and orientations relative to the weld metal and tested in as-received and prestrained conditions. The impact energy was evaluated over a range of temperatures from -196 to 150°C. It has been found that the significant contribution to the impact energy variability at a given test temperature is due to the microstructural inhomogeneity of a multipass weld. A definite trend has been established that impact toughness for specimens with notch root location in the reheated microstructure is greater than the impact toughness of specimens with notches located in the as-deposited microstructure. This trend is observed in both transition and upper shelf regions and is also valid for both as-received and prestrained material conditions.

**4:45 PM**

**Evaluation of Tensile Properties of Cast Al-7%Si-Mg Alloys Via Toughness and the Use of Work Hardening Characteristics**: *Murat Tiryakioglu*[1]; John Campbell[2]; James T. Staley[3]; [1]Robert Morris University, Dept. of Eng., John Jay Eng. Lab., Pittsburgh, PA 15108 USA; [2]University of Birmingham, IRC in Matls. for High Perf. Applications, Edgbaston, Birmingham B15 2TT UK; [3]Retired, Durham, NC, USA

Microstructure-property relationships in cast Al-7%Si-Mg alloys are very sensitive to the presence of discontinuities introduced into parts during processing. These discontinuities, namely oxide bifilms and porosity, reduce the tensile strength and especially elongation of these alloys. Tensile strength and elongation have been used to evaluate the structural quality of casting poured from these alloys. The authors have shown recently that toughness (or energy to fracture) can be used effectively to evaluate structural quality. Using work hardening characteristics following the Kocks-Mecking Work hardening model, critical toughness can be calculated for those specimens that fracture prematurely due to the presence of major discontinuities in the structure. A new quality index based on comparison of the actual and critical toughness will be introduced. The statistical validity of this technique will be verified by Weibull plots of toughness and quality index.

**5:10 PM**

**Effects of Microstructure and Stress Triaxiality on Ductility in Alpha/Beta Titanium Alloys**: *Seiji Kanamori*[1]; *Takashi Miyata*[1]; [1]Nagoya University, Dept. of Matls. Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

The effects of microstructure and stress triaxiality on the process of micro-void coalescence type of fracture and ductility of Alpha/Beta titanium alloys were investigated. Materials tested were 3 types of titanium alloys and two or three different heat treatments were conducted for these three alloys in order to obtain different grain size.It was shown that the most of micro-voids in Alpha/Beta titanium alloys were nucleated at Alpha/Beta interfaces, and the microstructural parameter defined as the average distance between Alpha/Beta interfaces was closely related to the dimple size on the fracture surface. The refinement of microstructure leads to the reduction of ductility, though near-Beta titanium alloys with ultra-fine particle of Alpha have exceptionally a different tendency from other Alpha/Beta titanium alloys. The dependency of triaxiality on fracture strain obtained from the round bar specimen were also investigated. Significant reduction of strain with the increase of triaxiality is observed, and it is shown that the void growth strain in higher strength materials is relatively small.

**5:35 PM**

**An Investigation of the Effects of Aging on the Compressive Deformation Behavior of Open Cell 6101 Al Foams**: *Jikou Zhou*[1]; S. Allameh[1]; W. O. Soboyejo[1]; [1]Princeton University, Princeton Matls. Inst. & The Dept. of Mechl. & Aeros. Eng., Olden St., Princeton, NJ 08544 USA

This paper presents the results of a combined experimental and theoretical study of the effects of aging on the compressive deformation behavior of Duocel open cell 6101 aluminum foams. Following the microstructural characterization of struts subjected to under-aging, peak aging and over-aging, micro-tensile properties and foam deformation characteristics are elucidated for these three microstructural conditions. Mechanism-based models are then used to predict the foam strengths as function of porosity and strut strength. The predicted strength levels are compared with the measured strengths before presenting some guidelines for the design of metallic foams.

## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Characterization - II

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Monday PM Room: 214
October 7, 2002 Location: Columbus Convention Center

*Session Chair:* David B. Williams, Lehigh University, Matls. Sci & Eng., Bethlehem, PA 18015-3195 USA

**2:00 PM**
**In-Situ TEM Observation of Alloying Process in Isolated Nanometer-Sized Particles**: *Hirotaro Mori*[1]; Jung Goo Lee[1]; [1]Osaka University, Rsrch. Ctr. for Ultra-High Voltage Electron Microscopy, Yamadaoka 2-1, Suita, Osaka 565-0871 Japan

Alloy phase formation in isolated nanometer-sized alloy particles has been studied by in-situ TEM. It is revealed that the finite size effect on the eutectic point, Teu, in binary alloy systems is so strong that in nm-sized alloy particles Teu can be lowered down to a temperature even below the glass transition temperature, Tg. As a result of this, in a particular system of the Au-Sn system where room temperature (RT) at which observations were carried out, lies in such an order as Tg>RT> Teu, a crystalline-to-amorphous transition has been observed by simply adding Sn atoms onto nm-sized crystalline particles of pure Au, whereas in the Sn-In system where RT lies in such an order as RT>Tg>Teu, a crystalline-to-liquid transition has been observed by simply adding In atoms onto nm-sized crystalline particles of pure Sn. It should be noted here that both the amorphous phase in the former system and the liquid phase in the latter system can be present at RT as phases more stable than crystalline counterpart(s) only when the size of the system is in the nanometer range.

**2:30 PM**
**Characterization of Bioactive Glass Coatings for Ti Implant Alloys**: *Antoni P. Tomsia*[1]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., One Cyclotron Rd., MS 62-203, Berkeley, CA 94720 USA

A new family of bioactive glasses has been developed with thermal expansion coefficients closely matching those of Ti-based implant alloys while maintaining bioactivity in vitro. These new glasses are intended for use as bonding agents between Ti-based implants and bone; in fact, adherent, mechanically sound graded coatings of these glasses have been deposited on titanium alloys. A firing schedule was developed that minimizes reactions and produces coatings with no cracks and good adhesion to the substrate. During firing a thin TiO layer formed on the Ti, preventing interfacial reactions. Adhesion tests indicate that the glass and titanium form a bond with a high enough crack growth resistance to drive the crack out of the interface into the bioactive glass coating. Support is provided by the National Institute of Health under grant number NIH/NIDCR 1R01DE11289.

**3:00 PM**
**Is Segregation-Induced Intergranular Embrittlement a Kinetically-Constrained Polymorphous Melting Process?**: *Paul R. Okamoto*[1]; Jonas K. Heuer[2]; Nghi Q. Lam[1]; James F. Stubbins[3]; [1]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Bldg. 212, E-214, Argonne, IL 60439 USA; [2]Bechtel Bettis, Inc., PO Box 79, W. Miffin, PA 15122 USA; [3]University of Illinois, Dept. of Nucl., Plasma & Radiologl. Eng., Urbana-Champaign, IL 61801 USA

Why and how S-segregation leads to intergranular embrittlement of Ni has been investigated by a combination of AES, slow-strain-rate tensile tests, and ion-implantation and RBS studies. Grain-boundary sulfur concentrations in dilute Ni-S alloys were varied by time-controlled annealing of specimens at 625°C. The critical S-concentration for 50% intergranular fracture of 15.53 ± 3.4 at. % S was found to be equal, within experimental error, to the critical implant concentration of 14.23 ± 3.3 at. % S required to induce 50% amorphization of single-crystal nickel during S implantation at liquid nitrogen temperature. These critical S concentrations are very close to the maximum possible solubility limit of 18 at. % S in Ni defined by the polymorphous melting curve on the Ni-S phase diagram. This suggests that segregation-induced intergranular embrittlement, like implantation-induced amorphization, may be a kinetically-constrained polymorphous melting process, albeit one occurring locally at grain boundaries. This interpretation is supported by the fact that the % intergranular fracture versus S grain boundary concentration can be described by the kinetic model of implantation-induced amorphization used to calculate the % amorphous volume fraction versus S-implant concentration. That the two curves are nearly identical implies that the distribution of implanted S-atoms and those at grain boundaries obey the same Poisson statistic and that the threshold S-concentration for the onset of amorphization and grain boundary decohesion are the same as that for polymorphous melting. The polymorphous melting concept for grain boundary decohesion also provides a simple explanation for well-known the synergistic effects of hydrogen-sulfur co-segregation on embrittlement.

**3:30 PM Break**

**3:45 PM**
**Development of Advanced Materials by Aqueous Metal Injection Molding**: *Santosh K. Das*[1]; Jerry LaSalle[1]; Jimmy Lu[2]; Mel Goldenberg[1]; [1]Polymer Technologies, Inc., 35 Monhegan St., Clifton, NJ 07013 USA; [2]Honeywell International, Tempe, AZ, USA

Metal Injection Molding is a well known technique for the cost effective production of complex multidimensional components. In general, such components are small, less than 25 grams, and often made in high production volumes. They are employed in a variety of industry sectors, for example, automotive and firearms. Application to the aerospace industry has been very limited due to demanding specifications, relatively low production runs, and difficulty in working with relatively advanced materials such as nickel-based superalloys. The development of a water based agar metal injection molding binder has allowed us to mold and sinter large superalloy components with mechanical properties exceeding those of investment cast superalloys. Tensile and fatigue properties of nickel-based superalloy IN718 exceed cast properties and meet aerospace specifications.

**4:15 PM**
**Microstructure and Chemistry of Commercial Boron Carbides**: *M. W. Chen*[1]; *Kevin J. Hemker*[1]; [1]Johns Hopkins University, Mechl. Eng., 3400 N. Charles St., Baltimore, MD 21218 USA

Due to its ultra high hardness and low density, boron carbides are being considered as a candidate material for military armor applications. Traditionally, bulk boron carbides are synthesized by hot pressing with various additions, such as: Al, V, Fe, Si, Mo, Ti, Fe2O3, nitrides, silicate, and etc. These additives are engineered into the boron carbide to improve densification and microstructural refinement, but the detailed mechanisms related to the distribution and effect of these dopants are not clear. In this study, the microstructure and chemistry of two commercial bulk boron carbides has been characterized with analytical electron microscopy (AEM) and scanning transmission electron microscopy (STEM). Grain boundary phases and intra-granular precipitates were characterized by energy-filtered imaging (EFI) and micro-beam electron diffraction. The distributions of Fe2O3, Al2O3 and other doped compounds were also investigated by STEM and found to exist as micron sized inclusions.

**4:35 PM**
**Microstructure Evolution of a Thermal Barrier Coating Platinum Aluminide Bond Coat During Thermal Cycling**: M. W. Chen[1]; *Kevin J. Hemker*[1]; [1]Johns Hopkins University, Mechl. Eng., 3400 N. Charles St., Baltimore, MD 21218 USA

Thermal barrier coatings, successfully used in rotating turbine blades in aircraft turbine engines, are designed as multilayered multi-functional systems. The bond coat layer is a key component because it provides both oxidation resistance and a foundation for the top coat. Microstructural and chemical investigations of as-deposited and thermally cycled platinum modified diffusion aluminide bond coats were performed with TEM and XRD. The structure of the as-received bond coat was confirmed to be an ordered B2 phase, but fine scale modulations were observed. At various stages of thermal cycling, the compositional development assisted by chemical inter-diffusion has been related to the transformation of the bond coat from its original B2 structure to a Ni-rich L10 martensite. The transformations, martensite ?\B2 phase, were confirmed to be reversible and to occur on heating and cooling in each cycle. Quantitative high temperature XRD measurements have been conducted to show that the volume of the B2 phase is approximately 2% larger than that of the martensite, and thereby ~0.7% transformation strain is produced on the phase transformation.

**4:55 PM**
**Iron Substitution in FeAl Alloy**: *Simon Dorfman*[1]; David Fuks[2]; Vlad Liubich[2]; Larisa Kutsenko[2]; [1]Technion, Phys., Haifa 32000 Israel; [2]Ben-Gurion University of the Negev, Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel

The aim of our paper is to study the bonding in FeAl alloyed by Ni, Ti and Cr and to estimate the preferable positions of these transition metals substituting for iron in D0_3 phase. We compare the results of our calculations with the experimental data and discuss the links between the bonding and site preference for studied Fe_3Al alloys.



## Pb-Free and Pb-Bearing Solders - I

*Sponsored by:* Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA

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*Session Chairs:* K. N. Subramanian, Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA; Linga Murty, National Science Foundation, Prog. Dir. Metals Rsrch. DMR, Arlington, VA 22230 USA

**2:00 PM Opening Remarks**

**2:05 PM Invited**
**Pb-Containing Fatigue-Resistant Solder Alloys for High Temperature Applications**: *Tsung-Yu Pan*[1]; Frank W. Gayle[2]; Duane Napp[3]; Alan Gickler[4]; James Slattery[5]; Jerry L. Badgett[6]; Gordon Whitten[6]; Gary Becka[7]; Brian Bauer[8]; Angela Grusd[8]; Christopher G. Olson[9]; Iver Anderson[10]; Jim Foley[10]; Ahmer Syed[11]; [1]Ford Motor Company, Ford Rsrch. Lab., 2101 Village Rd., Bldg. R, MD 3135, Dearborn, MI 48124-2053 USA; [2]NIST, Metlgcl. Div., 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA; [3]NCMS, 111 Hazeltine Dr., Georgetown, TX 78628 USA; [4]Johnson Manufacturing, 114 Lost Grove Rd., PO Box 96, Princeton, IA 52768-0096 USA; [5]Indium Corporation of America, PO Box 269, 1676 Lincoln Ave., Utica, NY, USA; [6]Delphi Delco Electronic Systems, One Corporate Ctr., MS 8186, PO Box 9005, Kokomo, IN 46904-9005 USA; [7]AlliedSignal, Inc, FM&T, PO Box 419159, D/836, MS 2C43, Kansas City, MO 64141-6159 USA; [8]Heraeus, 24 Union Hill Rd., W. Conshohocken, PA 19428 USA; [9]Rockwell Collins, Inc., 400 Collins Rd. NE, MS 107-110, Cedar Rapids, IA 52498 USA; [10]Ames Laboratory, 126 Metals Dvpt., Ames, IA 50011 USA; [11]Amkor Technology, Inc., 1900 S. Price Rd., Chandler, AZ 85248-1604 USA

In the High Temperature Fatigue-Resistant Solder Project, sponsored by National Center for Manufacturing Science, several lead- (Pb-) containing solders were evaluated for potential applications for up to 205°C operating temperature. Extensive phase diagram survey was conducted. Certain criteria were applied: alloys systems with very complex phase (>15 atoms/unit cell) and eutectics with >50% brittle intermetallic phases were eliminated. Twenty-two solder alloys, in the systems of Pb-X binary eutectics, Pb-Ag-X, Pb-Sb-X, and Pb-X-Y ternary alloys, were chosen. Wetting characteristic and intermetallic compound growth during thermal aging were measured. The effect on the stability of microstructure by the small additions of rare earth elements and other strong oxide formers including Te, Se, Ce, and Mg was examined. The addition of Li was found to improve the wetting characteristics of pure Pb to be comparable with Pb-10Sn.

**2:35 PM Invited**
**Development of a Thermodynamic Tool and its Application in Design of Micro-Soldering Materials in Electronic Packaging**: *Xing Jun Liu*[1]; Ikuo Ohnuma[1]; Ryosuke Kainuma[1]; Shuanglin Chen[2]; Y. Austin Chang[3]; Kiyohito Ishida[1]; [1]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Aoba-yama 02, Sendai, Miyagi 980-8579 Japan; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Madison, WI 53719 USA; [3]University of Wisconsin, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA

A user-friendly thermodynamic tool, ADAMIS/Pandat, has been developed for designing alloys for micro-soldering. ADAMIS/Pandat is a Window-based program, which combines a thermodynamic database including Ag, Au, Bi, Cu, In, Sb, Sn, Zn and Pb elements with phase diagram calculation software. The thermodynamic parameters describing Gibbs energies of the different phases in the database have been evaluated by optimizing experimental data for phase equilibria and thermodynamic properties. Using this tool, a user can easily calculate the liquidus projection, isothermal and vertical sectional diagrams and phase fraction. In addition, the tool provides other related information such as the surface tension, viscosity of the liquid phase and solidification simulation. This tool is expected to play an important role in design of the micro-solders used in the electronic packaging.

**3:05 PM**
**Thermodynamic Database of the Phase Diagrams in Copper Base Alloy Systems**: *C. P. Wang*[1]; X. J. Liu[1]; I. Ohnuma[1]; R. Kainuma[1]; K. Ishida[1]; [1]Tohoku University, Dept. of Matls. Sci., Sendai 980-8579 Japan

The development of substrate materials as well as Pb-free solders is important for the devices of electronic packaging technology. The present paper shows the construction of the thermodynamic database on the phase equilibria of the Cu-base alloys, including Cu-X binary system and Cu-Fe, Cu-Ni, Cu-Cr base ternary systems by the CALPHAD (Calculation of Phase Diagrams) method. The thermodynamic parameters describing Gibbs energies of the different phases have been evaluated by optimizing experimental data for phase equilibria and thermodynamic properties. Based on the database, the liquidus projection, isothermal and vertical sectional diagrams and phase fraction can be easily calculated. This database is expected to play an important role in design of the substrate materials in the electronic packaging and to present some information on interfacial reactions between Cu substrate and solders.

**3:30 PM**
**A Comprehensive and Quantitative Characterization Study on Lead-Free and Lead-Bearing Solder Materials**: *Pei-Lin Wu*[1]; Chiapyng Lee[2]; [1]ASUSTek Computer, Inc., Taipei Prototype Testing Lab./Mfg. Dept., 150 Li-Te Rd., Taipei, Taiwan; [2]National Taiwan University of Science and Technology, Dept. of Cheml. Eng., Taipei, Taiwan

The purpose of this investigation is to provide quantitative information to develop lead-free processes. The need to understand the distinguishing characteristics of lead-free solders and their impacts on both practical applications and solder joints reliability was the driving force behind this study. Two kinds of reliability evaluations, including electrical and mechanical strength tests, were implemented in this study. More aggressive flux systems might be needed for lead-free alloys in order to achieve better wetting performances which can be comparable with those of SnPb systems. However, certain aggressive flux chemistries might cause electromigration and form dendritic filaments. Therefore, surface insulation resistance(SIR) tests should be included in electrical reliability evaluations. In order to understand the mechanical properties of lead-free solder alloys, mechanical strength tests were implemented and solder joints were cross-sectioned for OEM studies.

**3:55 PM Break**

**4:15 PM Invited**
**Development of Tin Whiskers on Solder Joints Formed with Sn-3.8Ag-0.7Cu on Sn, Sn-Cu, and Sn-Bi Plated Lead Frames**: *V. Schroeder*[1]; G. Henshall[1]; P. Roubaud[1]; T. Campbell[1]; Swaminath Prasad[2]; Flynn Carson[2]; [1]Hewlett Packard, 1501 Page Mill Rd., MS 1222, Palo Alto, CA 94304-1126 USA; [2]ChipPAC, 47400 Kato Rd., Fremont, CA 94538 USA

Matte Sn is the primary plating candidate for lead frames on Pb-free components. Although matte Sn has been used in production for years, failures persist due to solid-state growth of Sn whiskers on Sn-plated lead surfaces. To retard this whisker growth, component manufacturers have been investigating dilute alloys of Sn, including Sn-Cu and Sn-Bi. This study evaluates whisker growth on or near joints formed with Sn-3.8Ag-0.7Cu solder on component leads plated with matte Sn, Sn-1%Cu, and Sn-XBi (X=1, 3, and 6%). In earlier work, it has been hypothesized that Sn whiskers do not develop after surface mount reflow because the reflow process relaxes the compressive stresses necessary for whisker growth. The current study investigates the validity of this hypothesis. The appearance and growth of Sn whiskers on solder joints and plated components, reflowed to a peak temperature of 260°C, are examined with SEM. These examinations are carried out on joints following reflow and again following three separate environmental exposures: (1) 500 thermal cycles from -55°C to 85°C, (2) 4 weeks at a constant temperature of 60°C with 90% relative humidity, and (3) 500 thermal cycles followed by 4 weeks at 60°C with 90% relative humidity. The data are presented in terms of the influence of plating composition and environmental exposure on the length, morphology, and distribution of Sn whiskers.

**4:45 PM**
**COST531: A New European Initiative on Lead-Free Solders**: *William John Plumbridge*[1]; [1]The Open University, Matls. Eng., Walton Hall, Milton Keynes, Buckinghamshire, UK

Under its system for Co-operation in the field of Scientific and Technical Research (COST) the European Community has just commenced an action on lead-free solders. The principal objective is to explore new alloy systems from a scientific basis and to identify those that may be used as lead-free solders. Additionally, potential products of on-board interactions will be investigated. Six working are envisaged, ranging from prediction and phase diagrams of possible systems, through mechanical, chemical and physical properties to reliability and performance of joints in service. A Memorandum of Understanding has been signed by more than ten countries, although participation is open. A request for Expressions of Interest and a Call for Proposals have recently been issued.

**5:10 PM**

**Microstructural Effects on the Behavior of Sn-Ag Solder Joints Under Repeated Reverse Stressing**: *K. J. Ferguson*[1]; A. Telang[1]; J. G. Lee[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Double shear lap joints made with eutectic Sn-Ag solder were cooled from 270°C at different rates ranging from air-cooling to iced-brine quenching. These joints were subjected to reversed stressing with constant shear strain amplitude at room temperature. Shear strain amplitudes between 0.75 and 1 were imposed during the course of this investigation. Role of microstructural features on the surface damage accumulation and stress-strain behavior was investigated as a function of shear strain amplitude and number of cycles of repeated stressing. Shear banding was found to be along Sn dendrites in air-cooled specimens, while it cut across the small Sn grains present in the quenched specimens. Acknowledgment: Project funded by National Science Foundation under grant number NSF-DMR-0081796.

**5:35 PM**

**Spalling Prevention on Thin Film UBM for Lead-Free Solders by Self-Formed Reaction Barrier Layer**: *C. Y. Liu*[1]; S. J. Wang[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

Spalling phenomenon is the one of key reliability issues in flip chip solder joints, since it will deteriorate the mechanical strength of solder joints. In next few years, lead-free solder will start to be implemented on electronic packaging industries. Fast reactions between Sn-rich lead-free solders and UBM will consume metal pad in UBM quickly and enhance spalling effect. To solve this problem, we report a novel technique to prevent the spallation of metal bond pad during the lead-free soldering reaction. We simply introduced a Cu reservoir into the structure of C4 (Controlled Collapse Chip Connections) solder joints. Cu atoms, which were contributed from the Cu reservoir, reacted with Sn atoms and formed a layer of Cu-Sn compound on the surface of UBM (Under Bump Metallization). This layer of Cu-Sn compound served as a reaction barrier to retard the Ni consumption from the soldering reaction. Therefore, the spalling effect is prevented.

## Rapid Prototyping of Materials: Rapid Prototyping Overview Presentations

*Sponsored by:* Materials Processing & Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector; MPMD-Powder Materials Committee, SMD-Composite Materials Committee-(Jt. ASM-MSCTS)

*Program Organizers:* Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3901 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Meisha L. Shofner, Rice University, MEMS, Houston, TX 77005 USA

Monday PM Room: 212
October 7, 2002 Location: Columbus Convention Center

*Session Chair:* Fernand D.S. Marquis, South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Eng., Rapid City, SD 57701 USA

**2:00 PM Opening: Fernand Marquis**

**2:10 PM Keynote**

**Freeform Fabrication–History and Current Processes**: *David L. Bourell*[1]; [1]University of Texas at Austin, Mechl. Eng., MC C2200, Austin, TX 78712 USA

Freeform fabrication as a technology is 20 years old, although its antecedents date back about 100 years. A brief historical context for freeform fabrication will be presented. Included is an introduction to current techniques and overview of processes. The presentation will close with a review of current applications and future possibilities.

**2:55 PM Keynote**

**Gas Phase Solid Freeform Fabrication**: *Harris L. Marcus*[1]; James Crocker[1]; Erik Geiss[1]; Haoyan Wei[1]; Leon L. Shaw[1]; [1]University of Connecticut, IMS/M&ME, U3136, Storrs, CT 06269-3136 USA

This paper will review research in the solid freeform areas of selective area laser depositon(SALD) and selective area laser deposition vapor infiltration (SALDVI). The topics covered will be the experimental approaches and a wide range of materials, composites and fabricated devices that have been processed using the SALD and SALDVI approaches. Advantages and disadvantages of the approaches will be discussed.

**3:40 PM Break**

**4:00 PM Invited**

**The Effect of Process Variables on Residual Stress and Melt Pool Size in Laser-Based Solid Freeform Fabrication**: *Jack L. Beuth*[1]; Aditad Vasinonta[1]; [1]Carnegie Mellon University, Dept. of Mechl. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

In this research, control of melt pool size and residual stress in laser-based solid freeform fabrication processes is studied for both thin-walled and bulky structures. Numerical simulations are used to construct non-dimensional plots (termed process maps) that quantify the effects of changes in part height, laser power, deposition speed and part preheating on melt pool size and maximum stress magnitudes. Maximum stress reductions due to the manipulation of process variables are quantified for both geometries. Although melt pool size and stress results are different for the two extreme geometries considered, broad strategies for the control of melt pool size and stress are similar. Two mechanisms for stress reduction are identified and are shown to be identical for both geometries. This leads to a strategy for simultaneous control of melt pool size and residual stress valid for both geometries. This modeling work is being performed in tandem with process development research underway on the LENS™ process at Sandia National Laboratories.

**4:45 PM Invited**

**Finite Element Analysis of Distortion Minimization in Layer-by-Layer Laser-Processed Components**: Kun Dai[1]; *Leon L. Shaw*[1]; [1]University of Connecticut, Dept. of Metall. & Matls. Eng., Storrs, CT 06269 USA

Residual thermal stresses and distortion are frequently present in the part built using layer-by-layer solid freeform fabrication techniques assisted with a moving laser source. In this study 3D finite element analysis is used to investigate effects of the laser scan pattern and the size of the part to be processed on residual thermal stresses and distortion. It is found that the out-of-plane distortion of a layer processed by a moving laser beam can be minimized with a proper selection of the laser scan pattern. A scan pattern having a change in its scan direction by 90° at every turn can lead to the cancellation of concave upward and downward distortions. It is also found that distortion and residual thermal stresses also depend on the relative size of the part with respect to the laser beam size. When the size of the part is relatively small with respect to the laser beam size, distortion is very small. However, as the size of the part becomes relatively large, distortion increases and is mainly caused by asymmetrical plastic deformation driven by transient thermal stresses rather than residual thermal stresses.

**5:30 PM Invited**

**Metal Powder Deposition: From Microns to Meters**: *James Sears*[1]; [1]South Dakota School of Mines & Technology, Adv. Matls. Procg. Ctr., Rapid City, SD, USA

This presentation briefly highlights aspects of metal powder deposition that have led up to the current state-of-the-art. Metal powder deposition has come along way since Schoop received his patent on "Metal-Spray (Metallizing)" in 1910. Since then the concept of using metal spraying for direct fabrication and repair of components has continued to mature, for example molten metal sprayforming commercialized by Osprey Ltd. and arc spray techniques developed by Singer. Today, however, the combination of a very controllable heat source–The Laser–and CAD/CAM interface developed by the rapid prototyping industry has led to a new rapid manufacturing technology "Laser Powder Deposition". Currently this technology is being applied at over six orders of magnitude, from microns to meters, fabricating components as large as aircraft parts and mining equipment to as small as electronic circuitry. This presentation will mainly focus on Laser Powder Deposition. Topics discussed include material property response, repair applications, cladding, hardfacing, preform fabrication, additive fabrication of structural components, meso-scale fabrication, functionally gradient materials, nano-materials, and fabrication of containers for hot isostatic pressing (HIP) of powders.

## Third Workshop on the Effects of Alloying Elements on the Gamma to Alpha Transformation in Steels: Session II

*Sponsored by:* McMaster Centre for Steel Research, TMS-ASM Phase Transformations Committee
*Program Organizer:* Gary R. Purdy, McMaster University, Department of Materials Science and Engineering, Hamilton, Ontario L8S 4L7 Canada

Monday PM Room: 220/221
October 7, 2002 Location: Columbus Convention Center

*Session Chair:* Gary R. Purdy, McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

**2:00 PM**
**Kinetic Transitions in Proeutectoid Ferrite Growth in Fe-C-Ni Alloys**: *C. R. Hutchinson*[1]; A. Fuchsmann[1]; H. S. Zurob[2]; Y. Brechet[1]; [1]Domaine Universitaire, Lab. de Thermodynamique et Physico-Chimie Métallurgiques, St. Martin d'Hères, Cedex, France; [2]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

In our work, binary Fe-Ni diffusion couples have been constructed from high purity Fe-1Ni (wt. %) and Fe-5Ni (wt. %) alloys and subsequently carburised to provide, in a single sample of constant C activity, a spectrum of Fe-0.1C-Ni alloy compositions. The kinetic transitions in ferrite growth have been examined, as a function of Ni content and temperature, by isothermally transforming these diffusion couples at several temperatures. On the basis of these experimental observations, new alloys of selected compositions have been cast and the isothermal growth kinetics of ferrite growth examined in the conventional manner. Our experimental observations and preliminary interpretations will be discussed.

**2:45 PM**
**Mn Diffusion and Partitioning During the Austenite/Ferrite Transformation in Fe-C-Mn and Fe-C-Mn-Si Alloys**: *Hui Guo*[1]; G. R. Purdy[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

The partitioning of Mn near the kinetic transition from slow to fast ferrite growth has been studied by STEM microanalysis in Fe-2.78 mass % Mn-1.81 mass % Si alloys and in Fe-2 mass % Mn alloys with different levels of carbon. In some cases it was possible to retain austenite in the immediate vicinity of the quenched $\alpha$:$\gamma$ interface by means of a second, low temperature, heat treatment. The observed Mn diffusion profiles in the ferrite and austenite phases are compared with the predictions of models for the transformation.

**3:30 PM Break**

**3:50 PM**
**Three-Dimensional and Electron Backscatter Pattern Analyses of Proeutectoid Ferrite**: *G. Spanos*[1]; R. O. Rosenburg[2]; M. V. Kral[3]; A. W. Wilson[4]; M. Enomoto[5]; [1]Naval Research Laboratory, Physl. Metall. Branch, Code 6324, Washington, DC 20375-5000 USA; [2]Naval Research Laboratory, Info. Tech. Div., Code 5594, Washington, DC 20375-5000 USA; [3]Formerly at Naval Research Laboratory, now at University of Canterbury, Dept. of Mechl. Eng., PO Box 4800, Christchurch, New Zealand; [4]Formerly at Naval Research Laboratory, now at Boeing Satellite Systems, El Segundo, CA 90009 USA; [5]Ibaraki University, Dept. of Matls. Sci., Nakanarusawa-cho, Hitachi 316 Japan

Despite the importance of understanding the true three-dimensional (3-D) morphology, distribution, and interconnectivity of solid-state precipitates, microstructural characterization is usually accomplished by methods that enable only two-dimensional (2-D) observations. This talk will review research in our group which has resulted in new findings about the true 3-D nature of ferrite precipitates (mostly Widmanstatten) in hypoeutectoid ferrous alloys. This talk will also review some recent work on application of Electron Backscatter Pattern Diffraction/Orientation Imaging Microscopy (EBSP/OIM) techniques to study the morphology, crystallography, and quantification of the proeutectoid ferrite transformation. In particular, pattern quality mapping techniques (also referred to as image quality mapping techniques) and various ways of crystallographic OIM representation have been applied to better understand and characterize the proeutectoid ferrite transformation. The results presented here provide useful insight into the morphology, interconnectivity, distribution, crystallography, and quantification of proeutectoid ferrite precipitates; this insight should be taken into account when considering alloying element effects on the ferrite reaction.

**4:35 PM**
**General Discussion and Summary**: *G. R. Purdy*[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

## Characterization and Representation of Material Microstructures in 3-D: Serial Sectioning & 3D Representation

*Sponsored by:* Structural Materials Division,
*Program Organizers:* Mike Uchic, Air Force Research Laboratory, Dayton, OH 45433-7817 USA; Dennis M. Dimiduk, Air Force Research Laboratory, Materials and Manufacturing Directorate, WPAFB, OH 45433 USA; Milo V. Kral, University of Canterbury, Department of Mechanical Engineering, Christchurch, New Zealand; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375 USA

Tuesday AM Room: 222/223
October 8, 2002 Location: Columbus Convention Center

*Session Chairs:* Michael D. Uchic, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA; Peter W. Voorhees, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA

**8:30 AM Opening Remarks**

**8:35 AM**
**A Historical Review of 3-D Reconstruction of Experimental Microstructures in Materials Science**: *George Spanos*[1]; Gary J. Shiflet[2]; Milo V. Kral[3]; [1]Naval Research Laboratory, Washington, DC 20375-5000 USA; [2]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904 USA; [3]University of Canterbury, Dept. of Mechl. Eng., New Zealand

Most direct observations of microstructural features in materials involve either optical or electron microscopy techniques. Until fairly recently, with a few exceptions (e.g., deep etching or stereo microscopy), optical and scanning electron microscopy (SEM) have typically been restricted to observations of a single plane of polish, while transmission electron microscopy (TEM) is limited to thin foils (approximately 100 nm) of material. This usually involves viewing a two-dimensional slice of material in order to deduce often complex microstructural features such as morphology, interconnectivity, and precipitate and/or grain distributions. For many microstructures of interest, this often results in serious errors in microstructural characterization. Although serial sectioning studies have been used by materials scientists as far back as nearly a century ago, not until about a decade ago, with the introduction to the materials science community of computer hardware and software capable of easily reconstructing, visualizing, and measuring 3-D microstructures, has 3-D analysis become such a very powerful (in many cases required) technique. On the other hand, sophisticated 3-D hardware and software has been in use for a longer time by other fields such as the medical industry, the defense department, and the entertainment industry. This talk will review the history of 3-D analysis of microstructures in materials science, from crude serial sectioning studies, to "movies" of serial sections, to various investigations which have more recently employed computer reconstruction, visualization and measurement techniques. The talk will conclude with some discussion of how far we have come, and how far we have to go.

**9:00 AM Invited**
**From Dendrites to Superalloys: New Views of Microstructural Evolution Using Three Dimensional Reconstructions**: R. Mendoza[1]; A. Lund[1]; J. Alkemper[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

We investigate the three-dimensional microstructure of a model $\gamma$-$\gamma$' alloy and dendritic solid-liquid mixtures during coarsening using digital reconstructions. The reconstructions allow quantitative measures of the microstructure to be obtained that have heretofore been impossible. In the dendritic coarsening studies we have determined the mean and Gaussian curvatures of the solid-liquid interfaces, the spatial anisotropy of the microstructure as measured by the spatial variation in the normals to the interfaces, and the genus of the microstructure, all as a function of time during coarsening. In the work on $\gamma$-$\gamma$' alloys we have examined the three-dimensional spatial distribution of the precipitates, the aspect ratios of the shape of the individual particles, and the anisotropy of the microstructure again using the normals to the interfaces. Results from each study and the insights provided by these measurements on microstructural evolution will be presented.

**9:30 AM Invited**
**Three Dimensional Microstructure Modeling in Phase Transformations**: *Gary J. Shiflet*[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904 USA

This talk will focus on using three dimensional modeling to understand phase transformations in metal alloys. Since about 1990 we have developed a number of techniques applied to serial sectioning, registering, and reassembling a metallurgical structure in a computer workstation for further interrogation. Besides the insight that comes from viewing a phase transformation product in its three dimensional entirety, useful quantitative information can be ascertained. For example, early on we coupled electron back scattered diffraction with our serial sectioning of a cellular transformation in Cu-Ti alloys and in ferrous pearlite. This allowed crystallographic information, such as, orientation relationships, to be included with phase transformation nucleation sites in 3-D structures. More recently in a Cu-In alloy we combined sectioning in a focused ion beam (FIB) instrument and SIMS obtaining serial sectioning and serial chemical mapping to examine diffusion fields in three dimensions of a growing cellular colony. The presentation will explore these and other useful techniques, including obtaining stereological information from digital structures. NSF has supported this work for the past 12 years as part of a DMR grant.

**10:00 AM**
**Serial Section and 3D Reconstruction of Engineering Materials**: *Milo V. Kral*[1]; [1]University of Canterbury, Dept. of Mechl. Eng., New Zealand

Several engineering materials have been examined via serial sectioning and 3D reconstruction to study: Pore size, shape and strut connectivity in a porous tantalum biomaterial; Creep void distribution in a Fe-Ni-Cr heat-resistant alloy; Connectivity of a pearlite colony in a carbon steel; Morphological evolution in a 'Hadfield' steel. The different experimental approaches and outcomes of these efforts will be discussed.

**10:25 AM Break**

**10:40 AM Invited**
**Characterization of Three-Dimensional Grain Structure in Polycrystalline Iron by Serial Sectioning**: C. Zhang[1]; T. Ishimaru[1]; *M. Enomoto*[1]; [1]Ibaraki University, Dept. of Matls. Sci., Hitachi 316-8511 Japan

The grain structure during grain growth of polycrystalline alpha-iron (containing ~60ppm C and ~100ppm O) was studied by multiple sectioning and three-dimensional reconstruction. The square plate specimens, cold rolled 90%, 10 mm in the side length and 0.8 mm in thickness, were annealed at 750°C for 30 min to produce a microstructure of the average grain size ~20 mm. In combination with quantitative metallograhic analysis on each section the distributions of the volume, the surface area and the number of faces of individual grains are measured. The results will be compared with theories or empirical rules, e.g. Aboav-Weaire rule in three dimensions.

**11:10 AM Invited**
**3D Analysis of Bainite Morphologies in Fe-C-M Alloys**: *R. E. Hackenberg*[1]; G. J. Shiflet[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div. (MST-6), MS G770, Los Alamos, NM 87545 USA; [2]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

Serial sectioning and 3D reconstruction of austenite decomposition products were undertaken in bayforming ternary steels to better understand their true morphologies in the bay region of their TTT diagrams. Jagged growth interfaces are revealed in allotriomorphic bainite formed at the bay in Fe-0.24C-4Mo, contrasting with the idealized geometries often assumed when formulating growth models. This also has implications for experimental thickening kinetics measurements. Examination of the so-called "degenerate" ferrite formed below the bay in Fe-0.3C-6.3W reveals that it is not degenerate at all, but rather has a Widmanstatten rod morphology which gives the appearance of degeneracy due to the multiplicity of ways that they can intersect a randomly-oriented plane of polish. Furthermore, these rods are grouped in packets possessing a common elongation direction, highlighting the crystallographic nature of their formation. The impact of these findings on the understanding of austenite decomposition in bayforming steels will be discussed.

**11:40 AM**
**Robo-Met.3D: The Design and Construction of a Fully-Automated Serial Sectioning Device for 3-D Microstructural Investigations**: *Jonathan E. Spowart*[2]; Herbert M. Mullens[3]; Daniel B. Miracle[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Southern Ohio Council of Higher Education, 3171 Research Blvd., Ste. 141, Dayton, OH 45420 USA

In order to provide the materials science community with accurate 3-dimensional microstructural information, ML is currently constructing a fully-automated (robotic) metallographic serial sectioning machine, Robo-Met.3D. While the technique of metallographic serial sectioning has been useful in the past, manual sectioning of specimens is extremely labor intensive, and therefore represents significant research risk. Robo-Met.3D aims to reduce the section preparation time by at least a factor of 10 by offering automated polishing, imaging, data storage and image manipulation, all in a fully-scripted "lights off" operation. In order to increase the utility of the machine, significant amounts of developmental effort have been devoted to the "front end" of the instrument, with state of the art data reduction and manipulation techniques being employed for 3-D visualization and processing of the collected data. Real-time iso-surface renderings are utilized for visualization. In addition, user-specified sections through the captured volumetric data sets are also available, via a suite of customizable IDL software routines. As well as improving speed, the utilization of a high-precision parallel-polishing machine and the removal of the human operator lead to improvements in precision over current manual techniques. Rapid serial sectioning over large fields of view (100-200 µm) with section thicknesses around 0.5 µm now becomes routine, allowing for the first time the possibility of obtaining real-time 3-D microstructural data as part of a process stream or rapid materials development program such as DARPA-AIM. Examples of 3-D microstructures obtained via the serial sectioning of a Discontinuously-Reinforced Aluminum (DRA) material are also presented. These materials are currently being used in high specific stiffness AF aero-structural applications (F-16 fighter ventral fin replacement). It is believed that the 3-D spatial arrangement of the reinforcement phases is key to understanding the fracture properties of this class of materials, and so they form an ideal candidate material system for demonstrating the capabilities of Robo-Met.3D.

**12:05 PM**
**Comparison of Methods for Recovering the Distribution of Grain Boundaries Over All Five Macroscopic Degrees of Freedom**: *David M. Saylor*[1]; Bassem S. El-Dasher[2]; Gregory S. Rohrer[2]; [1]National Institute of Standards and Technology, Ceram./Matls., 100 Bureau Dr., Stop 8521, Gaithersburg, MD 20899-8521 USA; [2]Carnegie Mellon University, Matls. Sci. & Eng. Dept., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Two procedures for recovering the distribution of grain boundary configurations in polycrystalline materials are described. The configuration of a boundary is defined by the combination of three parameters specifying the lattice misorientation and two parameters specifying the crystallographic orientation of the grain boundary plane. The first procedure for recovering the boundary distribution involves using an automated EBSD based mapping technique in concert with serial sectioning to directly observe of the geometric and crystallographic configuration of the grain boundary network in three-dimensions. The second is a stereological approach which allows the boundary distribution to be determined by characterizing the distribution of boundary traces on an observation plane. Both techniques were applied to a polycrystalline STO specimen. The results from the two techniques are compared, and the potential benefits and drawbacks of both are discussed.

## Forming and Shaping of Light Weight Automotive Structures: Materials & Evaluations - II

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

Tuesday AM — Room: 210
October 8, 2002 — Location: Columbus Convention Center

*Session Chair:* Dan Zhao, Johnson Controls Inc., Plymouth, MI 48170 USA

**8:30 AM Invited**
**Microstructural Evolution in High Strength Pearlitic Steel Wires**: *Michael G. Zelin*[1]; Amit Prakash[1]; [1]Goodyear Tire & Rubber Company, Techl. Ctr., PO Box 3531, Akron, OH 44309 USA

Recent increases in the strength of steel wire to 4000 MPa range provide new opportunities for weight reduction of products utilizing steel cord, particularly, automotive tires. Processing of this pearlitic steel requires control of microstructure yielding a high strength combined with a good fatigue and toughness. Major processes of microstructure evolution during wire drawing, including ferrite/cementite lamellae thinning, changes in inter-phase interfaces, texture development, and non-uniformity of plastic flow have been characterized. Effect of these processes on steel wire strength and ductility is discussed.

**9:00 AM**
**Microstructural Evolution at the Contact Interface of Automotive Aluminum Alloys Subject to Oscillatory Linear Relative Motion**: *Ray Jahn*[1]; Larry Reatherford[1]; Dan Wilkosz[1]; [1]Ford Motor Company, Ford Rsrch. Lab., 2101 Village Rd., MD 3135, SRL 2327, Dearborn, MI 48124 USA

Due to regulatory CAFÉ requirements on fuel efficiency, aluminum alloys have been employed increasingly in automotive applications in order to reduce component weight. While the tribological behavior of aluminum has enjoyed significant attention in the functional performance of powertrain products (wear, fatigue, etc.) and manufacturing processes (tool die, etc.) in automotive industry, it has received limited investigation in body construction applications. In this study aluminum sheets in overlap configurations were subjected to oscillatory linear relative vibration under compressive pressure normal to the sheet surfaces. The material reaction taking place at the junction interface under pressure was investigated using optical and electron microscopy, and energy dispersive X-ray spectroscopy. In particular, the microstructural evolution at both the junction interface and in the adjacent substrate resulting from the stress wave dynamics excited in the system was examined as a function of vibration time. At appropriate vibration conditions, wake features similar to those characteristic of the Rayleigh-Taylor instability commonly found in fluid systems were observed in solid state aluminum alloys. The implications of this result on mechanistic hypotheses and its potential similarity to the Rayleigh-Taylor instability in solid aluminum recently reported in nuclear fusion research will be discussed.

**9:20 AM**
**Investigation of the Mechanical Properties of Extrusion Welds in Al6082-T4**: Ghatu Subhash[1]; *Adam R. Loukus*[1]; [1]Michigan Technological University, Mechl. Eng.-Eng. Mechanics, 1400 Townsend Dr., Houghton, MI 49931 USA

Aluminum extrusions are increasingly used in automotive structures with intent to reduce overall weight and gain fuel efficiency. However, many extruded sections inevitably contain extrusion welds (also called seam welds). These welds are found to be the sources for premature failure during subsequent forming operations as well as of the parts in service. Therefore, a better understanding of the weld influences on the overall mechanical behavior of an extruded part is essential for proper design and fabrication. In this investigation, the microstructural details of the weld-affected regions of an A-pillar made of Al6082-T4 were systematically investigated. To determine the mechanical properties of the welds, tensile specimens were prepared with the weld located at 0°, 45°, and 90° to the tensile axis. For comparison purposes specimens from non-weld regions were also tested. The results of the microstructural investigations, the tensile response, and the failure modes will be presented.

**9:40 AM**
**Microstructure Evolution in AA 6061 Subject to Severe Plastic Deformation**: *Yogesh Bhambri*[1]; Shravan Indrakanti[1]; Raghavan Srinivasan[1]; [1]Wright State University, Mechl. & Matls. Eng. Dept., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA

It has been well established that severe plastic deformation to large strains can lead to a considerable refinement of the grain size of a metal, leading to potentially high strain rate sensitivity and improved formability at elevated temperatures. In this study, samples of over-aged AA 6061 were subject to severe plastic deformation at room temperature by ECAE (Equal Channel Angular Extrusion) by multiple passes through a 120° die. The changes in microstructure, including grain refinement, fracture and redistribution of precipitates in this alloy were studied. The deformed specimens were also subject to thermal aging treatments at temperatures up to 500°C to study the stability of the microstructure. Initial results indicate the precipitates break during the deformation process and are more uniformly distributed. Thermal aging studies will confirm whether the microstructure is sufficiently stable to retain the fine grain size up to the forging temperatures for this alloy.

**10:00 AM Break**

**10:10 AM** ***Cancelled***
**Microstructural Development During Processing of Strip Cast AA 5754 Aluminum Alloy**: Hamid Najjar-Azari[1]; *David S. Wilkinson*[1]; [1]McMaster University, Hamilton, Ontario L8S 4L7 Canada

**10:30 AM**
**Flow Behavior of AA 6061 after Severe Plastic Deformation**: *Shravan Indrakanti*[1]; Yogesh Bhambri[1]; Raghavan Srinivasan[1]; [1]Wright State University, Mechl. & Matls. Eng. Dept., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA

Over-aging of AA-6061 results in the formation of a relatively soft material. Subjecting this material to severe plastic deformation by ECAE (Equal Channel Angular Extrusion) results in the break up of the precipitate particles. Subsequent deformation behavior of the material is influenced by several dynamic phenomena, such as recovery, recrystallization and dissolution of the precipitates, that can occur. In this study, samples of over-aged AA 6061 were subject to severe plastic deformation by multiple passes through a 120° ECAE die at room temperature. Samples thus processed were tested in compression at constant strain rates at temperatures up to 400°C. Initial test results indicate the flow curves (true stress versus true strain curves) of this material show fluctuations, the periodicity and amplitude of which changes with deformation conditions. These flow curves will be compared with the flow behavior of as-received AA-6061-T6 deformed under the same conditions.

**10:50 AM**
**A Comparison of Microstructures and Refinement Mechanisms of Al-Ti-C and Al-5Ti-1B Refiners**: *Xiangfa Liu*[1]; Zhenqing Wang[1]; Yanhui Liu[1]; Zuogui Zhang[1]; Xiufang Bian[1]; [1]Shandong University, Key Lab. of Liquid Structure & Heredity of Matls., Jinan 250062 China

The differences of Al-5Ti-1B and Al-Ti-C refiner's microstructures, refining characteristics and refinement mechanisms were discussed in this article. Both of the refiners have TiAl3 compounds, they tend to present in blocky morphologies in Al-5Ti-1B, which are closely related to refining behavior; while TiAl3 compounds in Al-Ti-C refiner usually exists in needle-like or broken blocky morphologies, which have no relation with grain refinement efficiency. TiB2 particles in Al-5Ti-1B are strip-like which are closely agglomerated in the region of a-Al grain boundary. Their morphologies and sizes don't depend on the production parameters; TiC particles in Al-Ti-C refiners are equiaxed and near round, their surfaces are clear and regular, their sizes are from larger than 3?Êm to below 0.1mm, which is related to the melting temperature, Ti/C ratio and holding time. TiC particles distribute around the boundaries in interdendritic regions or inside and around the needle-like TiAl3 compounds, they seem to agglomerate at lower magnification, but in fact, most of them are incompact at higher magnification. As far as refining behavior, the contact time of Al-5Ti-0.35C is shorter than that of Al-5Ti-1B, but the fading behavior of Al-5Ti-0.35C is more obvious than that of Al-5Ti-1B, and the fading behavior of Al-5Ti-1B can be nearly eliminated by mechanical stirring; While the fading behavior of Al-5Ti-0.35C can't be recovered by mechanical stirring. At last, refinement mechanisms of pure aluminum grains by Al-5Ti-1B and Al-5Ti-0.35C refiners have been discussed. It was elementarily found that clusters of TiC take part in nucleating ?¿-Al and a single TiC particle seems to be difficult to do that in the situation of addition of Al-5Ti-0.35C. On the other hand, a single TiB2 particle can nucleate a-Al after addition of Al-5Ti-1B.

**11:10 AM**
**Multiplied Modification of Hypereutectic Al-Si Alloy by Al-2.5P and Al-8Ti-2C Master Alloy**: *Xiangfa Liu*[1]; Yanfeng Han[1]; Zhenqing Wang[1]; Xiufang Bian[1]; [1]Shandong University, Key Lab. of Liquid Struct. & Heredity of Matls., Jinan 250062 China

The modification effect of a new type of Al-2.5P master alloy on Al-24Si alloys was investigated. It is found that excellent modification effect can be obtained by the addition of this new type of Al-P master alloy to Al-24Si melt and the average primary Si grain size is decreased below 47ìm from original 225ìm. It is also found that Al-8Ti-2C master alloy can improve thre modification effect of the Al-P master alloy. When the content of TiC particles in the Al-24Si melt is 0.03%, the improvement reaches maximum and keeps steady with increasing the content of TiC particles. Modification effect occurs at 50 minutes after the addition of Al-P master alloy and TiC particles, and keeps stable with prolonging holding time.

## General Abstracts – Ferrous Metallurgy

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Tuesday AM — Room: 224/225
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* Donald Koss, Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA; Kenneth Calvin Russell, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139-4307 USA

**8:30 AM**
**Microstructural Banding and Fracture of a Stainless Steel**: *Amy C. Stauffer*[1]; Donald A. Koss[1]; John McKirgan[2]; [1]Pennsylvania State University, Matl. Sci. & Eng. Dept., State College, PA 16802 USA; [2]Naval Surface Warfare Center, Carderock Div., W. Bethesda, MD 20817 USA

The presence of microstructural banding in the AL6XN stainless steel has been examined. The banding takes the form of high densities of small precipitates located near the mid-thickness of the plate material. Metallographic and chemical analysis of the bands indicate elevated levels of Cr and Mo, consistent with the presence of the sigma phase precipitates. The corresponding effects of the banding on fracture in both uniaxial and multi-axial tension has been examined by determining the anisotropy of the fracture behavior as a function of the orientation of the tensile axis. Those results indicate that the bands promote fracture along the plane of the bands.

**8:55 AM**
**Ferromagnetism in Plastically Strained FeAl Single Crystals**: Dongmei Wu[1]; *Ian Baker*[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

Recent research indicates that the strain-induced ferromagnetic to paramagnetic transition in FeAl arises mostly from the generation of anti-phase boundary (APB) tubes. In this work, the time dependence on the magnetic behavior of plastically-strained B2-structured FeAl single crystals was studied. It was found that the saturation magnetization, $M_s$, decreased with increasing time after cold rolling due to the reduction in APB tube density (as determined by differential scanning calorimetry, DSC). $M_s$ and susceptibility, $\chi$, were found to be composition and orientation-dependent and show peak values at certain orientations. $M_s$ and $\chi$ show a clear relationship with the energy stored in APB tubes, as determined by DSC. The results will be discussed in terms of APB tube model. This research is funded by the National Science Foundation grant DMI-9973977.

**9:20 AM**
**What is the Solubility of Carbon in Ferrite?**: *Kenneth Calvin Russell*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Rm. 13-5050, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

The solubility of cementite at the ~1000K Fe-C eutectoid temperature is generally accepted to be near 0.001 atom/atom, whereas that of the stable graphite phase is slightly less. Yet, examination of the definitive 1972 Met. Trans. paper by Prof. John Chipman of MIT raises questions. The Boltzmann-type equations used to describe these solubilities have three inconsistencies. 1. The pre-exponential for graphite solubility is orders of magnitude above the theoretical value. 2. The solubility of graphite is predicted to exceed that of metastable cementite at temperatures above ~1050K. 3. The predicted solubilities of the two phases diverge rapidly below 1000K. Chipman's paper and related work will be discussed in terms of these inconsistencies.

**9:45 AM Break**

**10:10 AM**
**Investigation of Grain Boundary Properties in Pure Iron**: *Tricia Antoinette Bennett*[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng. Dept., 3325 Wean Hall, Pittsburgh, PA 15217 USA

This work is aimed at improving the understanding of the structure and properties of grain boundaries in pure iron, namely grain boundary energy, mobility and curvature. The sample is 99.999% pure with chemical composition (ppm) as follows: C:6, Si:1, Mn:1, P:1, S:2, Al:2, N:9 and O:18. Samples were annealed at 4000C, from 0.1s to 10000s. Sample preparation consisted of mechanical grinding and a fine polish using a vibropolisher. Orientation Imaging Microscopy (OIM) revealed that the dominant texture is <1 1 1>. Individual triple junction images are imaged at 0 tilt. Large areas containing the triple junctions are then scanned using OIM to obtain the misorientation angles of the boundaries. Image analysis software is used to reduce the width of each boundary to a pixel, measure dihedral angles, and calculate energy, mobility and curvature values. Energy values returned are solutions to Herring's equations. Conclusions are made based on these measurements.

**10:35 AM**
**Solute Partition During the Bainite Reaction in Fe-C-M Alloys**: *R. E. Hackenberg*[1]; G. J. Shiflet[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div. (MST-6), MS G770, Los Alamos, NM 87545 USA; [2]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

The mechanism by which substitutional solutes (M) reduce the bainite reaction kinetics in Fe-C-M steels is not fully understood. This study assesses the role of bulk partition (redistribution) of Mo between the ferrite, austenite and carbide phases in Fe-0.24C-4Mo reacted at its bay temperature. Fine-probe EDS results of the Mo distribution around the bainite-austenite growth front and in the carbides show that Mo effects its thermodynamically necessary partition between ferrite and alloy carbides via multiple diffusion paths, and in a non-equilibrium manner. Additionally, the carbon content of the untransformed austenite was surveyed with microhardness, which indicates that Mo diffusion (and not carbon diffusion) is the rate-limiting diffusional step of bainite growth at and above the bay temperature in this and related alloys. The implications of these results for the shapes of TTT diagrams will be discussed, along with the roles of crystallography and non-equilibrium reaction paths.

**11:00 AM**
**Secondary Austenite Precipitation in Duplex Stainless Steels, Proposed Model**: *Antonio J. Ramirez*[1]; Sérgio Duarte Brandi[2]; John C. Lippold[1]; [1]The Ohio State University, Welding Eng. Prog., 1248 Arthur E. Adams Dr., Columbus, OH 43221 USA; [2]University of São Paulo, Dept. Eng. Metalurgica-PMT, Av. Prof. Mello Moares 2463, São Paulo, SP 05508-900 Brazil

Secondary austenite (A2) is an important issue on multipass welding of duplex stainless steels (DSS) because of its influence on the corrosion resistance and toughness of the welded joint. Austenite precipitation and its relationship with chromium nitride (Cr2N) were studied in five DSS alloys (UNS S32304, S32205, S32550, S32750, S32760). The Gleeble system was used to perform short duration and high cooling rate ferritization and re-heating heat treatments. The study of these microstructures on the TEM and FEG-SEM, along with chemical microanalysis in the TEM, revealed a cooperative precipitation of Cr2N particles and A2 along ferrite/austenite interfaces for the DSS UNS S32550 and S32760. At the same time, based on the same cooperative precipitation model mentioned above and the presence of Cr2N rods alongside the intragranular A2; it is proposed the nucleation of intragranular A2 from Cr2N rods. This proposition is supported by the low energy orientation relationship Cr2N/ferrite/austenite, which has been verified in this study, and the measured chemical profiles. Thus, it is presented a new mechanism for intragranular A2 nucleation related to the Cr2N; in addition to its nucleation at inclusions and dislocations.

**11:25 AM**
**Forged and Direct Quenched Ti-Nb HSLA Steels**: *Mohamed Hadji*[1]; *Yacine Si-Kaddour*[2]; [1]University of Blida, Souma, BP 270, Blida, Algeria; [2]University of Houari Boumedienne, El-Alia, BP 32, Bab-Ezzouar, Algeria

Recent advances in Ti treated and direct quenched HSLA steels provide new opportunities for the hot forger to produce tough, high-strength parts without special forging practices. Warm forging continues to make steady progress as a cost effective, precision manufacturing technique because it significantly reduces machining costs.In this paper, the effect of Ti on mechanical properties and grain refinement of forged and direct quenched Ti-Nb high strength low alloy steels is investigated. Product evaluation of these steels indicate that they are comparable to conventional quenched and tempered steels.

## Interstitial and Substitutional Solute Effects in High Temperature Materials and Alloys: Conventional Metals and Alloys

*Sponsored by:* Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Structural Materials Committee
*Program Organizers:* Mark L. Weaver, University of Alabama, Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; Michael E. Stevenson, Metals and Materials Engineering, Suwanee, GA 30024 USA

Tuesday AM — Room: 213
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* Michael E. Stevenson, Metals and Materials Engineers, Suwanee, GA 30024 USA; Mark L. Weaver, The University of Alabama, Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487-0202 USA

**8:30 AM**
**Quantitative Evaluation of Strain-Induced Vacancy Concentration in Al Using NMR: Application to Portevin-LeChatelier Effect**: *K. L. Murty*[1]; Otmar Kanert[2]; [1]North Carolina State University, Nucl. Eng. & MS&E, PO Box 7909, Raleigh, NC 27695-7909 USA; [2]Universitat Dortmund, Dept. of Physics, Dortmund, Germany

It has been well established that jerky flow in materials known as Portevin-LeChatelier Effect (PLE) due to locking of the dislocations by substitutional impurity atoms occurs following a finite plastic strain during tensile testing at appropriate temperatures and strain-rates. PLE is not usually observed at very low temperatures where diffusion of solute atoms is not rapid enough to catch up with moving dislocations. At intermediate temperatures, the plastic strain-induced vacancy concentration becomes sufficiently high to make the solute atom diffusion rapid enough to lock the mobile dislocations. Thus, the amount of prior strain required decreases with increase in temperature and decrease in strain-rate. Cottrell model based on a simple linear increase of vacancy concentration and mobile dislocation density with plastic strain could explain many of the observations. As temperature increases, however, in-situ annealing of the excess vacancies requires more strain than predicted by Cottrell model for PLE. We summarize here in-situ NMR results on strain-enhanced vacancy diffusion in aluminum during deformation. Experimental results correlated with models based on vacancy production through mechanical (versus thermal) jogs while in-situ annealing of excess vacancies is noted at high temperatures. These correlations made it feasible to obtain explicit functional dependences of the strain-induced vacancy concentration on test variables such as strain, strain-rate and temperature. Thermal vacancies dominated at high temperatures, and the critical temperature for thermal vacancy dominance increased with increase in applied strain-rate.

**8:50 AM**

**Role of Interstitial Impurities and Dynamic Strain Aging in Radiation Hardening and Embrittlement of Iron and Steels**: K. L. Murty[1]; *R. Kishore*[1]; [1]North Carolina State University, Nucl. Eng., Box 7909, Raleigh, NC 27695-7909 USA

While effects of radiation produced defects are well known in terms of increasing strength (radiation hardening) and accompanied loss in ductility (radiation embrittlement), the interstitial impurity atoms (IIAs) such as C and N also play an important role through synergistic effects of dynamic strain aging and neutron irradiation. Concern arises as to the superimposed effects of blue brittleness and radiation on the ductility of the structural steels used in reactor pressure vessels. We report here results on pure iron and various ferritic steels following neutron radiation exposure. The majority of the radiation hardening stems from friction hardening, and the source hardening term decreased with exposure to neutron radiation apparently due to the interaction of the interstitial impurities with radiation produced defects. This decrease in the source hardening suppressed DSA, which in turn led to increased ductility with a simultaneous increase in the strength (following irradiation) in the temperature range of DSA in the unirradiated condition. While the source hardening term was evaluated from an extrapolation of the work-hardening region to the elastic line for the ferritic steels, the grain size variation of the yield strength in pure iron allowed a direct evaluation and demonstrated their equivalence. DSA effects will be described in various steels including A516, A533B along with a modified 9Cr-1Mo steel following two different heat treatments. The kinetics of DSA revealed the role of N and/or C in-solution. Interestingly, the inclusion of thermal neutrons along with fast resulted in a slight reduction in the yield stress compared to the exposure to only fast neutrons. These results are explained in terms of decreased source hardening due apparently to the removal of C and N from solution to radiation-induced defects.

**9:10 AM**

**Interaction of Oxygen Atoms with Vacancies in Niobium and Tantalum**: *Mikhail S. Blanter*[1]; E. B. Granovskiy[2]; [1]Moscow State Academy of Instrumental Engineering and Information Science, Stromynka 20, 107846, Moscow, Russia; [2]Chemical Abstracts Service, 2540 Olentangy River Rd., Columbus, OH, USA

In niobium and tantalum specimens quenched from temperatures very close to the melting point, new internal friction maxima were found. The maxima were assigned to vacancy-oxygen atom complexes. On the basis of analysis of changes of the new peaks and oxygen Snoek peaks during annealing after quenching, two different types of vacancy-oxygen atom clusters are shown. One type is an oxygen atom near a vacancy, and the other is an oxygen atom inside a vacancy. Annealing of the quenched specimens at increasing temperatures first results in the formation of the clusters of the first and then of the second type. Dissociation of the clusters and annihilation of vacancies occur at the temperatures above their formation temperatures.

**9:30 AM Break**

**9:45 AM**

**The Effect of Impurities on the Mechanical Properties of Tantalum**: *Shuh Rong Chen*[1]; George T. (Rusty) Gray[1]; U. F. Kocks[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

The mechanical properties of materials with a bcc structure are very sensitive to the impurity contents. The mobility of these interstitial solutes increases at higher temperature. The dislocation-solute interactions cause the well-known dynamic strain aging under certain deformation conditions. The mechanical property of a commercially pure tantalum was investigated in compression as a function of temperature from 77 to 1273K, and strain rate from 0.0001/s to 3000/s. Under higher strain rate, the dislocations are able to pull away from the solute atmosphere and there is no apparent dynamic strain aging observed. At lower strain rate, the material exhibits very high strain-rate sensitivity at lower temperature due to inherited high Peierls stress associated with bcc structure. Deforming quasi-statically between 373 and 773K, the material shows negative strain-rate sensitivity with strongest dynamic strain aging effect observed at 573K. At even higher temperature, the yield stresses show very small strain-rate and temperature sensitivities indicate that the controlling deformation mechanism is no longer the Peierls stress. The strain-hardening behavior is strain rate and temperature dependent as opposed to almost rate independent observed at lower temperature or at high strain rate. The implication of this very complex mechanical behavior on the constitutive modeling will be discussed. Work supported by the US Department of Energy.

**10:05 AM**

**Strain-Induced Interaction of Interstitial and Substitutional Atoms in hcp Ti, Zr, and Hf**: *Mikhail S. Blanter*[1]; E. B. Granovskiy[2]; L. B. Magalas[3]; [1]Moscow State Academy of Instrumental Engineering and Information Science, Dept. of Matl. Sci., Stromynka 20, Moscow 107846 Russia; [2]Chemical Abstracts Service, 2540 Olentangy River Rd., Columbus, OH, USA; [3]University of Mining and Metallurgy, Fac. of Metall. & Matl. Sci., al. Mickiewicza 30, 30-059 Krakov, Poland

The energies of strain-induced (elastic) O-O, N-N and C-C, and O-(N,C)- substitutional interactions are calculated for the hcp IVB group metals α-Ti, α-Zr, and α-Hf. The discrete atomic structure of the host lattice is taken into account. The elastic constants, lattice spacing of the host lattice, and concentration expansion coefficients of the solid solution lattice due to dissolved atoms are used as the input numerical parameters. The displacement fields of host atoms around the solute atoms are also calculated. The interaction oscillates and has a long-range nature. In all solid solutions the coordination shells exist where the solute atoms attract each other. This interaction is stronger in α-Ti than in α-Zr and α-Hf. The interstitial atoms interact more strongly with each other as compared with substitutional atoms. A comparative analysis of the interaction energies in the hcp IVB group metals with those in bcc and fcc solid solutions revealed the determining effect of the crystal lattice type on both the interaction energy and its dependence on interatomic distance. In general, the strain-induced interactions in hcp IVB group metals are of the same order as those in bcc solid solutions and are stronger than those in fcc solid solutions.

**10:25 AM**

**The Effect of Cobalt, Ruthenium, and Palladium Solute Additions on Mechanical Properties and the Microstructural Stability of Third Generation Superalloys**: *Eric C. Caldwell*[1]; Fermin J. Fela[1]; Gerhard E. Fuchs[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

Strength and microstructural stability are of increasing importance in third and next generation superalloys. Although the strength and microstructural stability of a Ni-base superalloy is a function of the composition, several elements are known to have a greater impact. For example, recently the substitutional elements, Co, Ru and Pd have been reported to increase the microstructural stability and, in some cases, strength of advanced single crystal Ni-base superalloys. In order to better understand the effects of these elements on properties and microstructural stability, several model alloys were processed with varying levels. The microstructures of the as-cast and heat treated materials were examined and the mechanical properties and thermal stability of the heat treated materials was evaluated. Results will be discussed.

**10:45 AM**

**Assessing the Influence of Precipitation on Serrated Yielding in Inconel 718**: *Christopher L. Hale*[1]; Nicholas P. Yerby[1]; Mark L. Weaver[2]; [1]The University of Alabama, Metallurgl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]The University of Alabama, Metallurgl. & Matls. Eng., Box 870202, A129 Bevill, Tuscaloosa, AL 35487-0202 USA

The influence of precipitate distribution on the occurrence of dynamic strain aging has been investigated in Inconel 718. In process annealed and solution treated materials, serrated yielding has been observed in the temperature range 400K-900K. Three different types of serrations, identified as A, B and C serrations were observed depending upon the temperature, strain rate, and test mode (i.e., tension or compression). Types A and B serrations were observed in the lower temperature range of 400K-~750K while type C serrations were observed in the temperature range ~750K-900K. Annealing resulted in a contraction of the regime associated with serrated flow or in complete elimination of serrated yielding. These observations have been linked to solute depletion caused by precipitation.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Fracture of Materials

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee

*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

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| Tuesday AM | Room: 216 |
| October 8, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Frank McClintock, Massachusetts Institute of Technology, Mechl. Eng., Cambridge, MA 02139 USA; Tadashi Ishikawa, Nippon Steel Corporation, Steel Rsrch. Labs., Futtsu, Chiba 293-8511 Japan

**8:30 AM**

**Micromechanisms of Deformation and Fracture Processes in Thermoplastic Olefins**: *Aravind Dasari*[1]; Steve J. Duncan[2]; R. Devesh K. Misra[1]; [1]University of Louisiana at Lafayette, Cheml. Eng. Dept., PO Box 44130, Lafayette, LA 70504-4130 USA; [2]Basell Polyproylene, Ltd., PO Box 5, Wilton, Borough of Stockton TS6 8YU UK

The paper describes some aspects of micromechanisms of uniaxial tensile deformation and fracture processes in thermoplastic olefins. The micromechanism include yielding, ductile ploughing, wedging, crazing, brittle cracking, etc. These deformation processes are conveniently depicted in the form of rate of mechanical deformation-strain diagrams, providing a broad perspective of micromechanisms operating in different deformation rate-strain regimes. The domains of micromechanisms vary in size depending on the physical and mechanical characteristics of thermoplastic olefins. The study assumes relevance, both for the scientific value of probing micromechanisms of deformation and for the technological significance of thermoplastic olefins as primary substitutes for traditional materials.

**8:55 AM**

**The Mechanical Response of a Dual Phase Steel**: *Guanglei Liu*[1]; J. D. Embury[1]; Farid E. Hassani[2]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada; [2]Stelco, Inc., Hilton Works, Hamilton, Ontario, Canada

A study of the detailed mechanical response of a galvannealed 600MPa dual phase steel (DP600) has been undertaken together with a detailed analysis of the role of various microstructure components. It was established that at temperatures above -65°C damage occurred by void nucleation and growth at the ferrite-martensite interface leading to ductile fracture. At -196°C the dominant damage process was by cleavage initiated in the embedded martensitic phase. Fractographic studies reviewed that the distribution of the martensitic phase, which is related to banding of the Mn, plays an important role in the fracture process both in tension and in bending.

**9:20 AM**

**Brittle-To-Ductile Fracture Transition in Nb-Based Alloys and Intermetallics**: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

The transition of brittle-to-ductile fracture in Nb-based alloys and intermetallics is assessed on the basis of the emission or propagation of dislocations from a crack tip. Analytical models have been developed and used to compute the surface energy, unstable stacking energy, Peierl-Nabarro energy, stacking fault energy, and the antiphase boundary energy. The use of energy ratios based on these parameters for predicting brittle-to-ductile fracture transition is evaluated for Nb-based solid solution alloys and intermetallics. These analytical models are then applied to predict alloying additions that would result in solid solution toughening in Nb-based alloys. Accuracy of the model is evaluated against experimental data in the literature. Possible applications and potential difficulties of this approach to ordered Nb-based Laves phases and silicides are assessed. Work supported by the AFOSR through Contract No. F49620-01-C-0016, Dr. Craig S. Hartley, Program Manager.

**9:45 AM Break**

**10:00 AM**

**A New Evaluation Method of Fracture Toughness for Micro-Sized Specimens**: *Kazuki Takashima*[1]; Yakichi Higo[1]; [1]Tokyo Institute of Technology, P&I Lab., 4259 Nagatsuta, Midori-ku, Yokohama 226-8503 Japan

A new evaluation method of fracture toughness for micro-sized specimens has been established and fracture toughness measurements have been carried out for micro-sized amorphous alloy specimens. Cantilever beam type specimens (10 x 10 x 50$\mu m^3$) with notches were prepared from amorphous alloy films. Fatigue pre-cracks were also introduced ahead of the notches. Fracture tests and introduction of fatigue pre-cracks were performed using a newly developed mechanical testing machine for micro-sized specimens. Fracture toughness values were determined according to the ASTM standards. However, it is difficult to satisfy plane strain requirements for some micro-sized specimens as the plastic zone size is determined by stress intensity and yield stress, and these two values are independent of specimen size. This should be noted to determine fracture toughness for micro-sized specimens. This evaluation method is based on that for ordinary-sized specimens and is promising to standardization of fracture toughness measurements for micro-sized specimens.

**10:25 AM**

**Investigation of the Crack Opening Behavior in Aluminum Alloy 5083**: *Md. Mainul Islam*[1]; [1]Kyushu University, Civil & Structl. Eng., Shirohama Danchi 53-501, Higashi-ku, Fukuoka 813-0045 Japan

Fatigue life is the summation of crack initiation life and propagation life, but the technique to evaluate initiation and propagation of cracks by a unified theorem has not been established yet. Toyosada et al. considered that the load amplitude range above re-tensile plastic zone's generated load (RPG load) contributes to fatigue crack propagation. They proposed a fatigue crack propagation law based on RPG load. They showed its utility for steels used as welding structures. Moreover, they already established a simulation code for analyzing fatigue crack propagation behavior. Recently they are accomplishing their research to verify validity of simulation for estimating fatigue crack propagation behavior in various metals, e.g. aluminum alloy. Before conducting estimation of fatigue crack propagation behavior in aluminum alloys, investigation is necessary to check crack opening behavior in case of both experiment and simulation for each aluminum alloy. Objective of this study is to estimate crack opening profiles in aluminum alloy 5083 by Dugdale Model and to compare these profiles with those by experiments and by finite element method (FEM) subject to CCT (center cracked tension) specimen and to introduce a coefficient, plastic constraint factor which should be applied to Dugdale Model to achieve improved crack opening profile in aluminum alloy 5083.

**10:50 AM**

**Pre-Yield Cracking in TiAl Alloys**: *Xinhua Wu*[1]; Dawei Hu[1]; [1]University of Birmingham, IRC in Matls., Edgbaston, Birmingham B15 2TT UK

Fully lamellar and duplex samples of Ti44Al8Nb1B with various surface-finish conditions have been tested in tension and the onset of cracking, which occurs during tensile tests, has been monitored using Acoustic Emission. It has been found that cracking occurs in fully lamellar samples at stress levels below the 0.2% proof stress (625MPa) in all the surface conditions examined. The surface condition has a significant effect on the stress at which cracking is first detected. Cracking occurs at a stress of 480 MPa for the as-machined condition and 620MPa for electro-polished surfaces. Annealing of the as-machined sample at a temperature of 700ºC for 0.5h before testing results in cracking at a stress as low as 200MPa. However in the duplex microstructure, cracking does not occur below a stress of 700MPa (i.e. close to the yield stress for these samples) even in the annealed condition. The significance of these observations is discussed in terms of the microstructure of the various samples, the residual stress caused by machining and the nature of the surface of electropolished samples.

**11:15 AM**

**Influence of Local Crystallography on the Mechanisms of Fatigue Crack Nucleation and Propagation in Metallic Materials**: *Pedro D. Peralta*[1]; Nancy Dellan[1]; Campbell Laird[2]; [1]Arizona State University, Dept. of Mechl. & Aeros. Eng., Eng. Ctr. G-Wing, Rm. 346, Tempe, AZ 85287-6106 USA; [2]University of Pennsylvania, Dept. of Matls. Sci. & Eng., LRSM, 3231 Walnut St., Philadelphia, PA 19104 USA

The cyclic deformation mechanisms leading to crack nucleation in metallic materials are reviewed along with those responsible for fatigue crack growth, placing emphasis on local crystallography effects elucidated through experiments on bicrystals and multicrystals of FCC materials. Models for the kinematics of fatigue crack growth are discussed with respect to local slip geometry and the kinetics of crack propagation as well Prof. Knott's fundamental contributions to the field. It is found that fatigue crack growth in bicrystals is strongly affected by the local slip geometry and anisotropy. Furthermore, recent studies using Orientation Imaging Microscopy reveal that the path followed by a fatigue crack in a multicrystal can be correlated to slip geometry inside a grain for transgranular fracture and the grain boundary misorientation for intergranular crack growth. The factors affecting the preference of one crack path over the other are discussed in terms of current models for fatigue crack growth.

**11:40**
**Issues in Ductile Fracture:** *T. Thompson*[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley CA 94720 USA

**12:05**
**Fracture Mechanics of Functionally Graded Materials (FGMs): Stress Intensity Factor Solutions and Designing Against Fracture:** *K.S. Ravi Chandran*[1]; Imad Barsoum[1]; [1]The University of Utah, Metallurgl. Eng., 135 South 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

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## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Functional Materials - I

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Tuesday AM — Room: 214
October 8, 2002 — Location: Columbus Convention Center

*Session Chair:* Sungho Jin, University of California-San Diego, Dept. of Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

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**8:30 AM**
**Microstructural and Magnetic Characterization of the Magnetic/Structural Transition in Gd<5/>(Si<x/>Ge<1-x/>)<4/> Materials**: *Karl A. Gschneidner*[1]; V. K. Pecharsky[1]; A. O. Pecharsky[1]; J. S. Meyers[1]; L. S. Chumbley[1]; F. Laabs[1]; W. Choe[1]; G. J. Miller[1]; D. C. Jiles[1]; J. Leib[1]; C. C.H. Lo[1]; M. Han[1]; J. A. Paulsen[1]; J. E. Snyder[1]; T. A. Lograsso[1]; D. L. Schlagel[1]; [1]Iowa State University, Dept. of Matls. Sci. & Eng., Ames Lab., 255 Spedding, Ames, IA 50011-3020 USA

Gd<5/>(Si<x/>Ge<1-x/>)<4/> undergoes a magnetic/structural first order transformation between ~30K for x = 0 to ~290K for x = 2.05. The high temperature monoclinic paramagnetic phase transforms to an orthorhombic ferromagnetic phase with decreasing temperature and/or increasing magnetic field. These phases have distinctly layered structures in which subnanometer thick slaps are connected by pairs of Si(Ge) atoms. In the orthorhombic/ferromagnetic phase all of the interslab Si(Ge)-Si(Ge) bonds are connected, while in the monoclinic/paramagnetic phase half of the Si(Ge)-Si(Ge) bonds are broken. This breaking and reforming of the interslab bonds results in a colossal magnetostriction (~1% along the a-axis), a giant magnetocaloric effect (about twice that of gadolinium) and a giant magnetoresistance (~25%–the same as found in commercial recording devices). Our initial SEM, TEM and MFM (magnetic force microscopy) measurements on single crystal and polycrystalline samples will be discussed and correlated with magnetic, electrical and thermal property measurements.

**9:00 AM** ***Cancelled***
**Engineering of Microstructures for Giant Magnetoresistance**: *A. Hütten*[1]; [1]University of Bielefeld, D-33615 Bielefeld, Germany

**9:30 AM**
**Nano Multilayers, from Steels to Magnets–Design for Mechanical/Functional Properties**: *Greg Jan Kusinski*[1]; Gareth Thomas[1]; [1]MMFX Technologies, 2 Corporate Park 102, Irvine, CA 92612 USA

The development of high-tech materials requires functional, multicomponent microstructures and multilayered materials designed, processed and controlled at the micron and nanometer levels. It is now quite well recognized that to optimize and design materials for specified properties, materials are best utilized as composites. The nature of the components, of these composites, their structures, morphologies and interfacial characteristics are most important. In particular, multilayered nano-structures are attractive for mechanical properties and many functional properties (e.g. magnetic). In order to understand the properties of any such multilayered system, and hence to be able to design pre-determined sets of properties, it is necessary to know their structure. For this reason, characterization of physical, chemical, and magnetic structures at relevant length scales is of particular importance. Examples are given of multilayer design from the microstructural viewpoint. The design criteria for steels with superior strength-toughness-corrosion properties based on the multilayerd microstructure composed of lath martensite and retained austenite are presented. Similarly, the magnetic properties of Co/Pt multilayers are highly dependent on the interfaces between ferromagnetic and nonmagnetic interfaces.

**10:00 AM**
**Phase Separation and Atomic Ordering in Mixed III-V Layers and Their Influence on Device Behavior**: *Subhash Mahajan*[1]; [1]Arizona State University, Cheml. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

We will show that atomic species in III-V layers, differing in their covalent tetrahedral radii, are not distributed at random on their respective sublattices. Two types of deviations from randomness are observed: phase separation and atomic ordering. Phase separation is two-dimensional in nature and occurs at the surface while the layer is growing, whereas atomic ordering takes place in near surface regions. Both microstructural features co-exist in layers grown by vapor phase techniques, and evolve to reduce the strain energy of the system. The carrier mobility is reduced in the presence of phase separation and thus it could have deleterious effects on the performance of transistors. On the other hand, the occurrence of phase separation and atomic ordering appears to enhance the reliability of light emitting devices. We therefore suggest that the microstructure of an active layer in a device must be tailored for optimal performance. The support of the above work, spanning many years, by DOE, ONR and Sandia Laboratories is gratefully acknowledged.

**10:30 AM Break**

**10:45 AM**
**Magnetic Structure of Nano-Crystalline Fe-Base Films**: *Jeff Th. De Hosson*[1]; [1]University of Groningen, Appl. Physics, Nijenborgh 4, Groningen 9747 AG The Netherlands

Nano-crystalline magnetic materials show soft magnetic properties, provided that the grain size is smaller than the width of domain walls. Because of the interest of professor Gareth Thomas not only in structural but also in functional materials this paper concentrates on applications of Lorentz microscopy and electron holography to unravel influences of the grain size, grain size distribution as well as topographical effects on the magnetic microstructure. The latter is often represented by so called magnetization ripple structure. Nano-crystalline FeZr(N) films on various substrates have been extracted on a TEM grid or are directly deposited on a silicon nitride membrane. The specimens are observed with a JEOL 2010F transmission electron microscope equipped with a post column energy filter. The extended Lorentz microscopy supplied the information about the mean ripple wavelength and ripple deviation angle. Off-axis electron holography provided a direct visualization of the magnetic lines of force within the sample.

**11:15 AM**
**Novel Nanostructured Functional Materials**: *Jagdish Narayan*[1]; [1]North Carolina State University, Matls. Sci. & Eng., 2147 Burlington Labs., Raleigh, NC 27695-7916 USA

We have synthesized nanostructural materials in a controlled way for novel photonics, electronics, magnetics and structural applications. These materials were designed and synthesized via self-assembly pulsed laser deposition method where size, shape and distribution of nanocrystallites were controlled to achieve desired properties for novel applications. Our results from systems show that bandgap engineering can be achieved by controlling the size and interfacial properties of nanocrystallites. Multilayer structures with embedded nanocrystallites of uniform size and multilayer structures where the size is different in subsequent layers have been produced to achieve the desired characteristics of photonic, electronic, and magnetic devices. similarly, mechanical properties can be controlled by grain size and interfacial properties. The primary focus of the present research is on synthesis and processing, and correlations of atomic structure and chemistry with properties and modeling of thin film structures and devices.

**11:45 AM**
**Biomimetics: Materials Design and Processing Through Biology**: *Mehmet Sarikaya*[1]; [1]University of Washington, Matls. Sci. & Eng., Roberts Hall, Box 352120, Seattle, WA 98195 USA

Structural and compositional control of materials at the molecular scale is a key to synthesis of novel functional systems. Biological tissues are models for engineering materials as they have excellent combination of physical and chemical properties due to their highly controlled chemical specificity, phase distribution, and morphologies as well as hierarchical microstructures. A common denominator in hard tissues (bacterial particles and ordered films, spicules, spines, shells, bones and dental composites), is the presence of biomacromolecules (proteins) in addition to inorganic phase(s). Macromolecules may be enzymes, nucleators, habit modifiers, and scaffolds, and control intricate architectures from the nano- to macro-meter scales. Developing truly biomimetic systems for practical technological applications requires genetic engineering of proteins for use as “molecular erector” sets for self-assembly of ordered structures at ambient conditions. I will give examples of bio-hard tissues with emphasis on structure-property correlations, and present protocols for novel materials via protein-assisted assembly.

**12:15 PM**
**Forces on Screw Dislocations Near Approaching Cavities**: *Xanthippi Markenscoff*[1]; *Vlado A. Lubarda*[1]; [1]University of California, San Diego, Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

The solution for a screw or edge dislocation in a multiply connected solid containing two cavities is not unique, but depends on the selected cut used to impose the displacement discontinuity and to create the dislocation. The Peach-Koehler force and the location of the equilibrium dislocation position is determined for screw dislocation in the ligament. The dislocation induced stress amplification in the ligament between two approaching cavities of equal or unequal sizes is then derived.

## Microstructure/Property Relationships in Alpha/Beta Titanium Alloys: Microstructure Effects and Property Prediction

*Sponsored by:* Structural Materials Division, SMD-Titanium Committee
*Program Organizers:* Patrick L. Martin, US Air Force, AFRL/MLLM, WPAFB, OH 45433 USA; James Cotton, The Boeing Company, Seattle, WA 98124-2499 USA

Tuesday AM — Room: 220/221
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* Rodney R. Boyer, The Boeing Company, Boeing Matls. Tech., Seattle, WA 98124 USA; Michael J. Kaufman, University of Florida, Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

**8:30 AM Invited**
**Property Variations in Ti Alloys: Which Factors Really Matter?**: *J. C. Williams*[1]; R. E. Schafrik[2]; [1]Ohio State University, Dean, Sch. of Eng., Columbus, OH 43210 USA; [2]GE Aircraft Engines, MD M89, One Neumann Way, Cincinnati, OH 45215 USA

Thermomechanical processing (TMP) and heat treatment (HT) are widely recognized as primary factors in controlling the properties of Ti alloys. What is less often recognized is that the properties most affected by TMP and HT are typical properties. However, critical components are designed to minimum properties. The difference between typical and minimum is often determined by anomalies. In Ti alloys, a number of possible anomalies are known including hard alpha, high density inclusions, beta flecks and micro-texture. These will be described along with their origins and the effects they have on properties. There also is a growing interest in being able to estimate material properties by modeling. The benefits that accrue from modeling, including the need to model minimum properties, will also be discussed. Modeling the minimum properties has a high pay-off but will require a different approach than that used to describe typical values. This matter will be conceptually discussed and some possible approaches suggested.

**9:10 AM**
**Source of Variability in Crack Nucleation and Total Fatigue Lives of Ti-6Al-2Sn-4Zr-6Mo**: *Sushant K. Jha*[1]; James M. Larsen[2]; Andrew H. Rosenberger[2]; [1]Systran Federal Corporation, 4027 Col. Glenn Hwy., Ste. 210, Dayton, OH 45431-1672 USA; [2]Air Force Research Laboratory, AFRL/MLLMN, WPAFB, Dayton, OH 45433-7817 USA

Fatigue Life Variability of Ti-6Al-2Sn-4Zr-6Mo (Ti-6-2-4-6) was studied at different stress amplitudes. The scale of crack size distribution at specific life intervals indicated the variability in total life was controlled by processes prior to about 15 to 30 micro-m crack size. Fracture surface analyses of crack origins showed that cracks nucleated at one or more equiaxed primary alpha particles. However, the total lives were independent of the “crack nucleation area”. Additionally, the size distribution of these areas did not vary with stress amplitude. This study demonstrates that the source of variability in fatigue lives of Ti-6-2-4-6 lies in the cycles for crack nucleation. Monte Carlo simulations using a microstructure-based crack nucleation model are performed and shown to reasonably predict the variability in life.

**9:35 AM**
**Influence of Microstructure, Micro-Texture and Hydrogen Content on the Dwell-Fatigue Response of a Near-Alpha Titanium Alloy**: *V. Sinha*[1]; M. J. Mills[1]; R. B. Schwarz[2]; J. C. Williams[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, Struct./Prop. Relations Grp., MST-8, MS G755, Los Alamos, NM 87545 USA

In this experimental work, the dwell-fatigue response of a typical high temperature near-alpha titanium alloy, Ti-6Al-2Sn-4Zr-2Mo-0.1Si (Ti-6242) has been examined. This alloy is used extensively in the compressor section of aeroengines. In the bimodal microstructure, the level of micro-texture has been varied by changing the three critical Thermo-Mechanical Processing (TMP) parameters: the forging strain, solution treatment temperature and cooling rate after solution treatment. The volume fraction of primary alpha in the bimodal microstructure has also been varied by changing the solution treatment temperature. For the bimodal microstructures, the effects of the level of micro-texture and the volume fraction of primary alpha on the dwell-fatigue behavior of Ti-6242 are examined. The failure modes and associated fractographic features for the different microstructural conditions are also discussed. The influence of hydrogen content on the dwell-fatigue response of Ti-6242 alloy is also being examined. (This work is being supported by The Federal Aviation Administration).

**10:00 AM Break**

**10:15 AM Invited**
**Prediction of Property/Microstructure Relationships in Alpha/Beta Ti Alloys for Tensile Deformation at Room Temperature**: *Hamish L. Fraser*[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210-1124 USA

A rules-based approach to the problem of predicting property/microstructure relationships in alpha/beta Ti alloys has been adopted. Thus, Bayesian neural networks are being developed using databases which relate microstructural features, composition and properties. Such databases are generally not available, proprietary versions usually containing heat-treatment schedules and properties, but not including results of quantitative microstructural analyses. Hence, the first part of the present study has involved the development of a high quality database for the alloys Ti-6Al-4V and Ti-550. A wide range of microstructures has been produced by heat-treating using a Gleeble™ thermo-mechanical simulator, and tensile coupons cut from these samples have been tested at room temperature. The microstructural features have been determined quantitatively using a set of rigorous stereological procedures that have been developed recently. These databases are used to develop neural networks which are used in two ways. Firstly, they are used to make predictions of properties of samples of these two alloys. Secondly, the networks are used to determine the existence of functional dependencies of tensile properties on the various microstructural and compositional parameters. These functional dependencies are used in turn to develop physically-based models for predicting properties. This research has been supported in part by the National Science Foundation, Division of Materials Research, and by the US Air Force through the Metals Affordability Initiative.

**10:55 AM**
**Role of Oxygen in Alpha-Beta Titanium Alloys on the Fracture Toughness and Environmentally-Assisted Cracking**: Peter Pao[1]; *M. Ashraf Imam*[1]; C. R. Feng[1]; Harry N. Jones[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Washington, DC 20375-5343 USA

The lower density of titanium and its alloys, including excellent general corrosion resistance in seawater with high strength to weight ratio, makes them an attractive material selection for wide range of applications. The mechanical properties of titanium alloys depend strongly on alloy composition, on the phases present and their volume fractions, and their spatial arrangement in the microstructure. Interstitial elements such as oxygen and hydrogen in relatively small concentrations, play a prominent role in a number of ways. Even though substantial work has been done on the effects of interstitial elements, for example oxygen and hydrogen, in titanium and titanium alloys, fundamental questions such as the mechanism by which interstitial elements govern mechanical properties in general, and toughness and environmentally-assisted cracking (EAC) in particular, have not been adequately addressed. The role of oxygen in alpha-beta titanium alloys on the fracture toughness and environmentally-assisted cracking (EAC) will be discussed.

**11:20 AM**
**Prediction of Tensile Yield Strength in the Alloy Timetal 555**: *Daniel J. Evans*[1]; Hamish L. Fraser[2]; [1]Air Force Research Laboratory, AFRL/MLLMD, WPAFB, OH 45433-7817 USA; [2]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210-1124 USA

A research program is described aimed at predicting room temperature tensile yield strength and ductility of Timetal 555 (Ti-5.2Al-4.8Mo-4.8V-1.25Cr-1Fe) using Bayesian neural networks. At first, databases which relate microstructural features, composition and properties for this alloy are being developed. A wide range of microstructures has been produced by heat-treating using a Gleeble™ thermo-mechanical simulator, and tensile coupons cut from these samples are being tested at room temperature. The microstructural features are being determined quantitatively using a set of rigorous stereological procedures that have been developed recently. These databases are then used to develop neural networks which provide predictions of properties of samples of the alloy. The quality of the prediction depends on the richness of the database itself, and Bayesian statistics are used to assess errors associated with predictions, and so identify where additional data is required. This research has been supported in part by the Air Force Research Laboratory and the Regents Eminent Scholar Endowment at the Ohio State University.

## Modeling the Performance of Engineering Structural Materials: Deformation Behavior and Time-Dependent Plasticity

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

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| Tuesday AM | Room: 215 |
| October 8, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Donald Lesuer, Lawrence Livermore National Laboratory, 26373 Crescent Ct., Livermore, CA 94550 USA; Glenn Daehn, Ohio State University, Dept. of Matls. Sci. & Eng., 116 W. 19th Ave., Columbus, OH 43210-1179 USA

TUESDAY AM

**8:30 AM Keynote**
**Shortening the Insertion Time for Materials Technologies–The 21st Century Metals Challenge**: *Dennis M. Dimiduk*[1]; Rollie Dutton[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, WPAFB, OH 45433-7817 USA

Beginning with the first flight of the Wright brothers in 1903, growth in air-breathing propulsion capability has depended upon improvements in both structural design and materials. For the pacing technologies the design community and the materials and processes (M&P) community collectively contributed to bring about the remarkable hardware that is the basis of today's engines. However, future prospects for continued improvement are not clear, in part because of the long and expensive process for developing structural materials. The materials science and engineering paradigm teaches that the choice of synthesis or process method leads to the evolution of a certain structure that exhibits a set of properties that is subsequently used by a designer. Unfortunately, aspects of materials and processes critical to design, such as predicting costs associated with a material, or the development of a material representation for design, are not explicitly included. Too often the practice of this paradigm focuses overwhelmingly on development of individual properties, while bounding affordability and reliability are typically delayed in time. Our research seeks to establish and validate new approaches for metals development that will accelerate the insertion of materials into production, principally through better performance prediction (including all aspects of performance). The effort is premised upon the concept that the required technical content and fidelity of the "designer knowledge base" must drive the use of models and experiments. Critical to this will be understanding how to effectively use materials models, how to link them across various length and time scales, how to couple them with experiments to yield the appropriate information for the designer, how to represent and manage the contents of the designer knowledge base, and how to integrate these tools into the business environment. This presentation offers a view of accelerated insertion of materials, and discusses key technical limitations needing attention.

**8:55 AM**
**On Modeling Precipitation Induced Hardening in Crystal Plasticity**: *Chung-Souk Han*[1]; Robert H. Wagoner[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

A material description for the elasto-plastic deformation of crystals with non-shearable precipitates at finite strains is presented in this article. On the basis of the elastic inclusion model of Bate et al. 1981 the constitutive equations are formulated in a crystal plasticity setting. The anisotropic kinematic hardening for the actual yield condition of the slip systems is described by the averaged accommodation tensor over all precipitates of an aggregate. For needle and disk-like shaped precipitates the incorporation of precipitate rotations is suggested to describe the evolution of back stress terms in the deformation process. The dependencies of the kinematic hardening description on shape, size, and orientation of the precipitates within the deformation process are discussed.

**9:20 AM**
**Phase Field Modeling of Dislocation Network**: *Chen Shen*[1]; Yunzhi Wang[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The formation and dynamic evolution of dislocation networks (substructures) is critical for the understanding of mechanical behavior of crystalline materials. Recently a phase field approach has been developed to describe three dimensional (3D) dislocation system at a mesoscopic level. Since it does not require tracking individual dislocation lines, it has the potential ability to handle self-consistently any topological changes during dynamic evolution of arbitrary 3D dislocation networks. The current formulation of the method, however, does not account for dislocation reactions such as 1/2<110> + 1/2 <101> = 1/2 <011> which is essential for the understanding of dislocation network formation and coarsening. In this presentation we will describe the extension of the phase field model to allow for dislocation reaction and node formation, in accord with Frank's rule. Various examples of dislocation interaction and network formation and relaxation will be presented. The work is supported by the National Science Foundation.

**9:45 AM**
**Plastic Instability of Wake Morphology in Solid Commercial Aluminum Alloys**: *Sheng-Tao John Yu*[1]; Ray Jahn[2]; [1]Wayne State University, Mechl. Eng. Dept., 5050 Anthony Wayne Dr., Rm. 2146, Eng. Bldg., Detroit, MI 48202 USA; [2]Ford Motor Company, Ford Rsrch. Lab., 2101 Village Rd., MD 3135 SRL 2327, Dearborn, MI 48124 USA

Plastic deformation features of large-scale roll-up and waviness have been observed experimentally at the interface of two adjoining aluminum plates subject to the simultaneous application of linear in-plane vibration and normal pressure. Despite the absence of an externally applied heat source, the morphologies of the plastic instability features observed in solid aluminum samples bear resemblance to those associated with the Rayleigh-Taylor instability commonly observed in fluid mechanics. Since relatively large deformations at the interface occur, both elastic motions and plastic "flows" at the joint surface are of concern. The physical processes can be best described as elasto-visco-plastic flows in continuum mechanics. There exist complex energy transmission mechanisms between the various energy modes–thermal energy, kinetic energy, and potential energy–due to internal transient stress waves. The process cannot be characterized as a traditional solid mechanics deformation problem, and so cannot be straightforwardly modeled by the elliptic and/or parabolic formulations employed by conventional finite-element analyses. The plastic instability phenomenon observed in solid aluminum is modeled as a hyperbolic system, consisting of equations for the evolving stress tensor components, bounded by conservation of mass, momentum, and energy. The hyperbolic equation set is solved by the CESE method, a novel numerical framework specially suited for accurate and efficient solutions of complex hyperbolic problems. The CESE method is distinguished by the simplicity of its design principle, i.e., uniform treatment of space and time in calculating flux conservation. In the presentation, details of the model formulation and the numerical method as well as preliminary results will be reported. The origin of the observed plastic instability leading to such an unusual wake morphology will be discussed.

**10:10 AM Break**

**10:25 AM**
**Modeling Rate and Scale Dependent Plasticity**: *Yoon-Suck Choi*[1]; Amit Acharya[2]; Triplicane A. Parthasarathy[1]; Dennis M. Dimiduk[3]; [1]UES, Inc., AFRL/MLLM, WPAFB, OH 45433-7817 USA; [2]Carnegie Mellon University, Dept. of Civil & Environ. Eng., Pittsburgh, PA 15213 USA; [3]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, WPAFB, OH 45433-7817 USA

The rate and scale dependence of plasticity was modeled using a gradient theory incorporating lattice incompatibility proposed by Acharya, Bassani and Beaudoin. The 1-parameter rate-dependent constitutive model proposed by Kocks, Estrin and Mecking was modified by including a measure of slip plane lattice incompatibility in order to account for scale effects on the finite deformation of crystals. Lattice incompatibility is characterized by the gradient of the inverse of the elastic deformation field, which provides naturally a scale dependence of constitutive behavior, without explicit introduction of length-scale into the constitutive relationship. The basic time integration scheme followed was the forward gradient method of Peirce, Asaro and Needleman. A tangent stiffness was formulated for a hardening response enhanced with the lattice incompatibility. The constitutive model was implemented within the finite element software ABAQUS, using a user material subroutine (UMAT). The model was applied to Ni-base two-phase single crystals for the simulation of precipitate size dependence of flow stress, and the results will be discussed.

**10:50 AM**
**Modeling High Temperature Creep Behavior in Gamma TiAl Alloys**: *Karthikeyan Subramanian*[1]; Gopal B. Viswanathan[1]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43212 USA

The Jogged-Screw model has been adopted and suitably modified to explain observations of 1/2[110]-type jogged-screw dislocations in equiaxed and fully lamellar Ti-48Al in creep deformed microstructures. Tall jogs have been observed which are assumed to be dragged nonconservatively. Other modifications, including the assumption of a stress-dependent limit to the tall jog height, lead to excellent predictions of minimum creep rate and stress exponent. The model has been further refined for the fully lamellar microstructure taking into consideration the constrained nature of deformation in the lamellae. An isostress averaging approach seems to predict reasonable creep rates and stress exponents in the high stress regime in the FL alloys. The creep mechanisms at low stresses have also been investigated. Transmission Electron Microscopy was used to characterize key modeling parameters like jog spacing, jog height and dislocation density as a function of stress and strain.

**11:15 AM**

**Recovery as the Rate Controlling Step in Creep**: Holger Brehm[1]; Byeong Soo Lim[1]; *Glenn Steven Daehn*[1]; [1]Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Most crystalline solids at elevated temperature deform such that the steady state strain rate increases with the fifth power of applied stress. This process can be understood as the interplay of three processes: 1) dislocations overcoming obstacles, 2) plastic flow breeding new dislocations, increasing the obstacle density and 3) diffusion controlled processes reduce the obstacle density, promoting flow. A quantitative model of this type is consistent with broad experimental trends. Of these three processes, the mechanisms of the dislocation recovery (coarsening) process are the most poorly understood. We will show that recovery processes can be broadly understood as a traditional coarsening process, and that with this element creep and recovery can be quantitatively linked.

## Pb-Free and Pb-Bearing Solders - II

*Sponsored by:* Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA

Tuesday AM — Room: 211
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* C. R. Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chungli City, Taiwan; Fay Hua, INTEL, Matls. Tech. Operation, Santa Clara, CA 95054 USA

**8:30 AM Invited**

**Formation and Growth of (Au,Ni)Sn4 Phase in Pb-Free and Pb-Bearing Solders on Ni/Au Metallization**: *Ming Li*[1]; Ka Yau Lee[1]; [1]Institute of Materials Research and Engineering, 3 Research Link, 117602 Singapore

Interfacial reactions between various solder bumps and Cu/Ni/Au bond-pad metallization during solid state annealing had been investigated. A distinctive difference in interfacial microstructure between Pb-bearing (40Pb/60Sn and 70Pb/30Sn) and Pb-free (96.5Sn/3.5Ag) solder bumps was observed. A continuous layer of (Au,Ni)Sn4 intermetallic compound was formed at the solder-Ni3Sn4 interface in the SnPb systems. In contrast, big lumps of (Au,Ni)Sn4 particles were found scatter above the solder-Ni3Sn4 interface in the Pb-free system. The different morphologies of the (Au,Ni)Sn4 phase in Pb-bearing and Pb-free solder systems investigated were attributed to the balance of various surface energies in the systems. The layer growth of the (Au,Ni)Sn4 intermetallic compound had been explained on the basis of nucleation, growth and ripening. A simple kinetic analysis of ripening of a set of mono-size spheres onto a flat surface was also given to describe the time dependence of the layer growth.

**9:00 AM Invited**

**Penetration Mechanism of Sn-Based Solders into Cu6Sn5 Layer**: *Yong-Seog Kim*[1]; Sang Hoon Yun[1]; Jong Hwan Park[1]; Yong-Ho Lee[1]; [1]Hong-Ik University, Matls. Sci. & Eng., 72-1 Sangsu Dong, Mapo Ku, Seoul 121-791 Korea

In an attempt to understand the channel formation mechanism during reflow soldering of Sn-based solders on Cu UBM, penetration behavior of the solders into Cu6Sn5 substrate was investigated. Cu6Sn5 substrate was prepared by mechanical alloying of elemental Cu and Sn powder mixtures, followed by hot pressing at 400°C for 7 hours. The Sn-based solders were placed on the polished surface of the substrates and heated to several reflow temperatures in order to measure the depth of solder penetration. Microstructural observation of the samples indicated that the speed of the penetration is orders of magnitude of faster than that of the growth rate of Cu6Sn5 intermetallic layer during reflow soldering. The SEM/BEI, TEM, and XRD analysis of the sample revealed that the penetration of the Sn-based solders into the substrate is assisted by several phenomena such as wetting of the grain boundaries, dissolution of Cu6Sn5 and Cu3Sn phase formation.

**9:30 AM**

**Solidification Characteristics of Copper Particle-Reinforced Lead-Free Composite Solders- Effect of Cu Particle Size**: *Dechao Lin*[1]; *Guo-Xiang Wang*[1]; T. S. Srivatsan[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

Eutectic Sn-3.5%Ag solder is one of the best candidates to replace lead-bearing solders, and has attracted great attention in industry. Recent experiments found that addition of metallic powders as reinforcements is a viable means for increasing the strength of this solder. This presentation reports solidification experiments of eutectic Sn-Ag solders reinforced by either micro-size or nano-size copper powders. Composite solders are obtained by blending the Sn-Ag solder powders (micro-size) with either nanosized or microsized Cu powders. Reflow experiments of fluxed paste are then performed in an oven. Both optical microscope and SEM are then employed to observe the microstructure characteristics of the solder matrix and the intermetallic compounds. Special attention is paid to the dissolving kinetics of micro-size copper powder and the subsequent formation of Cu-Sn intermetallic compounds. A study of the distribution and geometry of the formed intermetallic compounds in various cases helps understand the role of the nanopowders in composite solders. Strength of the solidified composite solders is also quantified through microhardness test. Results reveal an increasing in hardness of composite solders with the addition of either nanosized or microsized Cu powders.

**9:55 AM**

**Using Sn-Bi Alloys to Improve Materials Science Instruction**: *Mark A. Palmer*[1]; [1]Kettering University, IMEB Dept., 1700 W. Third Ave., Flint, MI 48439 USA

Sn-Bi has been considered as a lead-free solder for low temperature applications. Because the melting temperature of Sn-Bi alloys range from 138C to 271C, Sn-Bi alloys can be used in an undergraduate laboratory experience. Lab experiments and classroom demonstrations illustrating: melting point depression, diffusion, grain growth, second-phase coarsening and sintering will be presented. The incorporation of these experiments into and their impact upon a basic materials engineering course will be discussed. This work was supported by the National Science Foundation DUE 9980982.

**10:20 AM Break**

**10:40 AM Invited**

**Effects of Heating and Compression on the Electrical Resistivity of Soldered and Conductive Adhesive Joints**: *Deborah D.L. Chung*[1]; *Kyu Dong Kim*[1]; [1]University at Buffalo, The State University of New York, Dept. of Mechl. & Aeros. Eng., Furnas Hall, Rm. 608, Buffalo, NY 14260-4400 USA

Soldered and conductive adhesive joints are used for electrical interconnection in electronic packaging. The reversible and irreversible effects of heating (up to 60ºC) and compression (stress perpendicular to the joint interface) on the contact electrical resistivity of eutectic tin-lead solder and silver-epoxy joints were distinctly observed in real time during variation in temperature and stress. Reversible effects were due to (i) increase in volume electrical resistivity of solder or adhesive with temperature and (ii) stress (above 0.02 MPa for solder and above 0.005 MPa for adhesive) induced reversible damage. Irreversible effects were due to (i) decrease in the joint thickness, as induced by compression and/or heating, and (ii) damage, as induced by stress (above 0.12 MPa for solder and above 0.009 MPa for adhesive). The thermal and mechanical reliability was superior for soldered joint than adhesive joint.

**11:10 AM Invited**

**The Intermetallic Phases at the Sn-3.5Ag-X(Cu,Zn)/Alloy 42 Interface**: *Shih-Chin Chang*[1]; [1]National Tsing-Hua University, Dept. of Matls. Sci. & Eng., Hsinchu 30043 Taiwan

Sn-3.5Ag-X(Cu,Zn) lead-free solders were used to bond alloy 42 with different reflow temperature and time. The morphology and the development of the intermetallic phases at the lead-free solders Sn-3.5Ag-X(Cu,Zn)/Alloy 42 interface and their effects on the mechanical properties were studied. The interface can be exposed by a special technique of pulling after taper grinding. Lying on the interface was a single layer of very long and thin needle phase which was almost invisible when the interface was examined along the cross section. A mechanism is proposed for the formation and growth of the needle phase at the interface.

**11:40 AM**

**Solid-State Aging Reactions Between Ni and SnCu Lead-Free Solders with Different Cu Concentrations**: W. T. Chen[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City, Taiwan

The eutectic Sn-0.7Cu solder is considered a very promising lead-free replacement for the Sn-37Pb solder, especially for wave soldering applications. For industrial uses, a 0.2% uncertainty in composition is generally accepted. In other words, for the Sn-0.7Cu solder, the Cu concentration can range from 0.5 to 0.9 weight percentage. However, our recent study revealed that, during soldering, the Cu concentration had a very strong effect on the reactions between Sn-Cu solders and Ni. When the Cu concentration was low (x=0.3 and lower), the reaction product was (Ni1-xCux)3Sn4 at the interface of a solder joint. At high Cu concentrations (x=0.6 and higher), the reaction product was (Cu1-yNiy)6Sn5. When the Cu concentration was in-between (x=0.4 and 0.5), both (Ni1-xCux)3Sn4 and (Cu1-yNiy)6Sn5, formed. In other words, during soldering, the Cu concentration must be strictly controlled in order to obtain consistent results. In this study, we would like to extend our earlier study to investigate whether this strong concentration dependency also occurs during the solid-state aging of the solder joints. We aged solder joints at the solid-state at several different temperatures for time as long as 1000 hours. The solder compositions studied include Sn-0.2Cu, Sn0.3Cu, Sn-0.4Cu, Sn-0.5Cu, Sn-0.6Cu, Sn-0.7Cu, and Sn-1.0Cu. Analysis techniques used include optical microscope, SEM, EPMA, and XRD.

**12:05 PM**

**The Microstructure Characterization of Ultrasmall Eutectic Bi-Sn Solder Bumps on Au/Cu/Cr and Au/Ni/Ti UBMs**: *Un-Byoung Kang*[1]; Young-Ho Kim[1]; [1]Hanyang University, Div. of Matls. Sci. & Eng., 17 Haengdang-dong, Seongdong-ku, Seoul 133-791 Korea

Flip chip technology is increasingly used due to its high packaging density and good electrical performance. Recently, the pad size and the pitches of chips get smaller and finer because of further miniaturization and integration of electronic components. Bi-Sn solder has raised great attention for low temperature solder alloy. This paper presents the evolution of microstructure in ultrasmall eutectic Bi-Sn solder bumps on Au/Cu/Cr and Au/Ni/Ti UBMs. Ultrasmall 58wt%Bi-42wt%Sn solder bumps with the diameter of 50 μm- were fabricated using lift-off method and reflowed using RTA system. The large Bi phase and the lamellae of Bi-Sn were observed in the Bi-Sn solder bumps formed on Au/Cu/Cr UBM. Increased cooling rate promoted the development of lamellar structure on solder surface. For Bi-Sn solder bumps on Au/Ni/Ti UBM, the Bi phases were mainly segregated on solder surface and two phase-like structures were observed. Anisotropic and faceted intermetallic compounds were found on the solder surface reflowed at slow cooling rate. As the cooling rate increased, the solder surface became smooth and the lamellar structure appeared. The effect on microstructural change and intermetallic formation of cooling rate and UBM structure will be discussed.

## Rapid Prototyping of Materials: Rapid Prototyping of Composites

*Sponsored by:* Materials Processing & Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector; MPMD-Powder Materials Committee, SMD-Composite Materials Committee-(Jt. ASM-MSCTS)

*Program Organizers:* Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3901 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Meisha L. Shofner, Rice University, MEMS, Houston, TX 77005 USA

Tuesday AM Room: 212
October 8, 2002 Location: Columbus Convention Center

*Session Chair:* David L. Bourell, University of Texas at Austin, Mechl. Eng., Austin, TX 78712 USA

**8:30 AM Invited**

**Microstructure Evaluation of Nb-Ti-Cr-Si Alloys Processed Using the LENS™ Technique**: *Ryan Richard Dehoff*[1]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Niobium based refractory intermetallics show promising potential for elevated temperature, structural applications. Nb-Ti-Si "in-situ" metal-ceramic composites contain silicide phases Nb3Si and Nb5Si3 in a body centered cubic Nb solid solution matrix. Cr additions have been found to increase oxidation resistance at high temperatures. The processing technique used in the present study of this system is the laser engineered net-shaping or LENS™ process. The goal of this research is to characterize the microstructures formed in the Nb-Ti-Si-Cr alloy system when the LENS™ process is conducted using elemental powder blends as feedstock. Among the potential advantages of this route are: (a) the ability to easily produce graded compositions and (b) a more uniform microstructure resulting from the negative enthalpy of mixing associated with the silicide phases[1]. Mechanical properties testing as a function of composition and microstructure is also planned. [1]K. Schwender, R. Banerjee, P. C. Collins, C. A. Brice, and H. L. Fraser, Scripta Mat., 45(10), 2001 pp. 1123-1129.

**9:00 AM**

**Rapid Manufacturing of Metal Matrix Composite Materials Using Three-Dimensional Printing (3DP)**: *Rhonda Lynn Anderson*[1]; [1]Concurrent Technologies Corporation, 5780 W. Werner Rd., Bremerton, WA 98312 USA

Three Dimensional Printing, (3DP), is under development for the rapid and flexible production of end-use parts, prototypes, and tools. The process has unprecedented flexibility, incorporating a rapid prototyping process with an electronic manufacturing environment to create complex geometry parts through solid freeform fabrication. This paper will introduce the 3D printing process for metal matrix composite materials. Components which would benefit from this system in their design and manufacture with respect to practical applications will be discussed. In addition, material testing and results as well as case studies of components will be presented.

**9:30 AM**

**Processing and Microstructural Characterization of Carbon Nanofiber Reinforced Silicon Nitride Matrix Composites**: *Erica L. Corral*[1]; Joseph Cesarano[2]; John Stuecker[2]; Enrique V. Barrera[1]; [1]Rice University, Dept. of Mechl. Eng. & Matls. Sci., MS-321, PO Box 1892, Houston, TX 77251-1892 USA; [2]Sandia National Laboratories, Adv. Matls. Lab., 11001 University Blvd., Ste. 100, Albuquerque, NM 87112 USA

Carbon fiber reinforced silicon nitride composites and colloidally processed silicon nitride have shown increased mechanical strength and reliability when compared to monolithic parts. In order to achieve an advanced structural material, this work presents the novel approach of colloidally processing silicon nitride with vapor grown carbon nanofibers and single-walled carbon nanotubes via a rapid prototyping fabrication method, called robocasting. High solids loading aqueous slurry formulations were developed using colloidal processing methods to manipulate charging behavior between carbon nanofibers and silicon nitride particle surfaces. Forty-five percent solids loading suspensions with pseudo-plastic rheology (but bordering dilatancy) were used to successfully fabricate near-net shape composite parts using robocasting. Parts were then sintered for one hour at 1740°C in nitrogen atmosphere. The quality of the composites was assessed using scanning electron microscopy, before and after sintering, evaluating carbon nanofiber dispersion and bonding. This work is supported by the National Science Foundation Alliances for Graduate Education and the Professoriate at Rice University and The Advanced Technology Program under Grant Number 003604-0039-2001. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

**10:00 AM**

**Processing Issues Associated with Reinforced Polymers for Use with Solid Freeform Fabrication**: *Meisha Lei Shofner*[1]; Karen Lozano[2]; Fernando Jamie Rodriguez-Macias[3]; Ranji Vaidyanathan[4]; Enrique V. Barrera[1]; [1]Rice University, Mechl. Eng. & Matls. Sci., MS 321, 6100 Main St., Houston, TX 77005 USA; [2]University of Texas Pan American, Eng. Dept., 1201 W. University Dr., Edinburg, TX 78539 USA; [3]Rice University, Dept. of Chem., MS 60, 6100 Main St., Houston, TX 77005 USA; [4]Advanced Ceramics Research, 3293 Hemisphere Loop, Tucson, AZ 85706 USA

Composite materials are particularly desirable for use with Solid Freeform Fabrication (SFF) techniques. The unique processing approach can be simpler and faster than traditional fabrication techniques for these materials, and it allows complex shapes to be constructed. Recent interests have been directed toward reinforcing extrusion-based SFF polymeric feedstock materials with fillers such as glass fiber and nanofiber systems. These materials exploit SFF technology to achieve fiber alignment while improving the functionality of the parts produced by these techniques. Issues of fiber distribution and dispersion, fiber/matrix interaction, process porosity, and processing viscosity are key to the development of the starting materials and the success of the resulting part. In this presentation, the state of the art understanding of reinforced feedstock materials will be addressed with attention to the newly produced nanocomposite systems.

**10:30 AM Break**

**10:50 AM Invited**
**Rapid Prototyping and Characterization of a WC-Based Cermet/Tool Steel Functionally Graded Materials Synthesized by Laser Cladding**: *R. Kovacevic*[1]; J. H. Ouyang[1]; H. Mei[1]; [1]Southern Methodist University, Dept. Mechl. Eng., Rsrch. Ctr. for Adv. Mfg., 1500 International Pkwy., Ste. #100, Richardson, TX 75081 USA

This paper presents the rapid prototyping and characterization of a WC-based cermet/tool steel functionally graded materials (FGMs) synthesized by laser cladding. One-step laser cladding process is capable of creating a fully dense and metallurgically bonded near-net-shape tool product with microstructural and compositional gradients of WC-based cermets in high-speed tool steel. By controlling the amount of different supplied powders under the optimized laser cladding conditions, WC-based cermet/tool steel FGMs are successfully synthesized layer by layer. X-ray diffractometer (XRD), scanning electron microscope (SEM), electron probe microanalyzer (EPMA), and optical microscope are employed to investigate the crystallize phases, chemical composition and microstructure of FGMs. The FGM exhibits an expected gradient structure, a high bonding strength, and a distribution in functionality of synthesized layers between the superhard outside surface with an excellent wear resistance and the inside core with a combination of high toughness and hardness at elevated temperatures. The interfacial microstructure at the overlapped region of different layers and the combining state of different phases between the hard WC particles and the tool steel have been investigated to evaluate the partial dissolution and localized precipitation of particles in laser molten pool. The Vickers hardness and wear resistance of the FGMs distinctly depends on the content and distribution of hard particles. Surface roughness and the geometrical property of the synthesized FGM tool product is also controlled by changing the heat input and cooling conditions during laser cladding. The WC-based cermet/tool steel FGMs may find great potentials as high-temperature wear resistant materials in many engineering applications.

**11:20 AM**
**In-Situ Reactive Rapid Prototyping of Intermetallic Compounds by LENS™ Processing**: *Weiping Liu*[1]; John N. DuPont[1]; [1]Lehigh University, Dept. of Matls. Sci. & Eng., Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Intermetallic alloys based on aluminides (e.g. $Ni_3Al$, NiAl, $Ti_3Al$, TiAl) are considered to be attractive materials for high temperature structural applications. Laser Engineered Net Shaping (LENS™) is a rapid prototyping process, which involves laser processing fine metal powders into three-dimensional shapes directly from a CAD design. So far, this technique has been used in the laboratory to fabricate a variety of metals and alloys from pre-alloyed powders. In this work, an attempt has been made to fabricate aluminide intermetallic compounds in-situ from elemental powders, based on the concept of reactive synthesis, using the LENS™ process. In-situ reactive alloying was achieved by delivering elemental Ni or Ti and Al powders from two different powder feeders, eliminating segregation observed in the samples deposited by using the pre-mixed elemental powders. Aluminides of various compositions were obtained easily by regulating the ratio of their feed rates. The aluminide deposits exhibited high solidification cracking susceptibility and porosity formation. Effects of the LENS™ processing parameters and alloy compositions on the defects formation and microstructure were studied. Potential methods for eliminating these defects will be described.

**11:50 AM**
**Dental Restoration Through Laser Densification of Dental**: *Leon L. Shaw*[1]; X.-X. Li[1]; J.-W. Wang[1]; H. L. Marcus[1]; T. B. Cameron[2]; C. Kennedy[2]; [1]University of Connecticut, Metall. & Matls. Eng., Inst. of Matls. Sci., Storrs, CT 06269 USA; [2]Dentsply Ceramco, Burlington, NJ 08016 USA

Densification of dental porcelain powder via a moving laser beam has been investigated in order to build dental restorations through layer-by-layer solid freeform fabrication (SFF) approaches. Both single line scans and multi-line scans with different overlaps between adjacent scans have been studied. It is found that a variety of defects such as porosities, cracks, distortion and macro-discontinuity can appear in laser-densified porcelain bodies if laser processing parameters (e.g. the laser processing temperature, laser scan rate, scan overlapping distance, and substrate pre-heating) are not appropriate. The phenomena observed have been discussed in terms of the vaporization of dental porcelains, surface tension effects, Marangoni forces, thermal transient and residual stresses. Methods and laser processing conditions that can lead to the prevention of the aforementioned defects have been discussed.

## Characterization and Representation of Material Microstructures in 3-D: Atom Probe and Focused Ion Beam Methods

*Sponsored by:* Structural Materials Division,
*Program Organizers:* Mike Uchic, Air Force Research Laboratory, Dayton, OH 45433-7817 USA; Dennis M. Dimiduk, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Milo V. Kral, University of Canterbury, Department of Mechanical Engineering, Christchurch, New Zealand; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375 USA

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| Tuesday PM | Room: 222/223 |
| October 8, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Milo V. Kral, University of Canterbury, Dept. of Mechl. Eng., Christchurch, New Zealand; Gary J. Shiflet, University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

**2:00 PM Invited**
**Chemical Composition of Interfaces and Precipitates as Obtained by the 3-Dimensional Atom Probe**: *Reiner Kirchheim*[1]; Guido Schmitz[1]; Talaat Al-Kassab[1]; [1]Universität Göttingen, Institut für Materialphysik, Hospitalstr. 3-7, Göttingen D-37073 Germany

The 3-dimensional or tomographic atom probe is based on field ion microscopy where the screen is replaced by a 2-dimensional, position sensitive detector. Thus the lateral coordinates of atoms which were removed from a conducting sample by a high voltage pulse can be determined. The time of flight reveals their chemical nature, and continuous stripping allows in-depth analysis. The lateral resolution of about 0.1 nm and the even better depth resolution permit a chemical analysis on the sub-nanometer scale. Results of this new technique are presented for (i) the initial stages of interdiffusion at the boundary between two metals, (ii) P-segregation at grain boundaries in nanocrystalline Ni-P alloys, (iii) initial stages of nucleation and growth in Cu-Fe and Cu-Ti alloys, (iv) internal oxidation and oxygen segregation at metal/oxide interfaces, and (v) distribution of hydrogen in metallic multilayers. It will be also shown that the new technique not only allowed a characterization on the atomic scale but verified and/or falsified existing models for the examples given before.

**2:30 PM Invited**
**Atom Probe Tomography–Three-Dimensional Characterization of Materials on the Atomic Scale**: *M. K. Miller*[1]; S. S. Babu[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6136 USA

The spatial coordinates and the elemental identities of the atoms in metallic and semiconducting alloys may be determined by atom probe tomography. Several methods including atom maps, isoconcentration surfaces, concentration profiles have been developed to reconstruct the microstructure with near atomic resolution and to visualize the solute distribution in alloys. The data may be statistically analyzed to detect and quantify concentration fluctuations due to phase separation, precipitation and segregation to interfaces. The tendencies for solute clustering and chemical short range order may also be estimated. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

**3:00 PM**
**Three-Dimensional Microstructural Characterization at the Atomic Level by Atom-Probe Microscopy**: *Dieter Isheim*[1]; Christian B. Fuller[1]; Stephan S.A. Gerstl[1]; Emmanuelle A. Marquis[1]; Chantal K. Sudbrack[1]; Kevin E. Yoon[1]; Olof C. Hellman[1]; David N. Seidman[1]; [1]Northwestern University, Dept. Matl. Sci. Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Three-dimensional atom-probe microscopy (3DAPM) permits a three-dimensional analysis of microstructures in metallic materials based on the chemical identities of individual atoms and their positions in real space. 3DAPM results in the reconstruction of a volume typically 15 x 15 x 100 $nm^3$ that contains about one million atoms and thus is a powerful experimental tool for the analysis of materials with microstructures on a nanometer scale. Data visualization and extraction of the microstructural and compositional characteristics at this length scale presents a conceptual and computational challenge, as the primary information contained in the reconstruction is discrete at the atomic level. We present the concepts and methods for our studies of the temporal evolution of nanoscale multiphase microstructures and phenomena associated with heterophase interfaces in these multiphase microstructures by 3DAPM for several alloy systems.

Examples discussed include Ni-base superalloys, Al(Sc, Mg or Zr) alloys, fully lamellar $\alpha_2/\gamma$ titanium aluminide alloys, and internally nitrided Fe(Mo, Sb or Sn) alloys.

**3:25 PM Invited**
**Nanoscale Focused Ion Beam Tomographic Imaging**: *R. Hull*[1]; A. Kubis[1]; D. N. Dunn[2]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., 116 Engineers Way, Charlottesville, VA 22904 USA; [2]IBM, 2070 Rt. 52, Hopewell Junction, NY 12533 USA

As nano-scaled structures becoming of increasing importance in science and engineering, techniques for reconstructing three-dimensional structural, crystallographic and chemical relationships become increasingly critical. We describe a technique which uses focused ion beam sputtering to expose successive layers of a 3D sample, coupled with secondary electron imaging and secondary ion mass spectroscopy of each sputtered surface. Computer interpolation of these different slice images then enables reconstruction of the 3D structure and chemistry of the sample. These techniques are applicable to almost any inorganic material, at a spatial resolution of tens of nanometers, and fields of view up to (tens of mm)$^3$. Examples of the application of this technique are drawn from microelectronic via structures, complex lamellae precipitates in a Cu-15In alloy, and cellular structures in Ni-Al superalloy materials. Prospects for extrapolation of these techniques to higher spatial resolution and much higher chemical sensitivities will be discussed.

**3:55 PM Break**

**4:15 PM**
**3D Microstructural Analysis of a Ni Base Superalloy Using a Dual Beam FIB-SEM**: *Michael D. Uchic*[1]; Dennis M. Dimiduk[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

A Dual Beam Focused Ion Beam and Scanning Electron Microscope has been used to serial section and image the three dimensional microstructure of an industrially relevant Ni base superalloy, in order to discern the distribution and morphology of various microstructural phases. Using automated software, the FIB-SEM can be used to sequentially section and image opaque microstructures with high fidelity, especially in terms of controlling the sectioning process. At present, we have used this instrument to examine a volume of material approximately 30 microns$^3$ with a serial section thickness of 100nm. Using image processing and visualization software, we will show 3D reconstructions of the grain structure and precipitate distributions of the Ni base superalloy, and we will present quantitative measurements of some of these microstructural features in both 2D and 3D.

**4:40 PM**
**Microstructural Tomography of a $Ni_{70}Cr_{20}Al_{10}$ Superalloy Using Focused Ion Beam Microscopy**: *Marc De Graef*[2]; Michael D. Uchic[1]; Robert Wheeler[3]; Dennis M. Dimiduk[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA; [2]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., Pittsburgh, PA 15213 USA; [3]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

A Focused Ion Beam has been used to simultaneously image and serial section the secondary γ' precipitate structure of a model nickel base superalloy. This method is capable of resolving the 3D structure of this particular alloy system with extremely high resolution, where the voxel size of the 3D reconstruction is approximately 20 nm$^3$, and the total examined volume of material is approximately 4 μm$^3$. In this talk, we discuss the advantages and limitations of the experimental technique, as well as the methods used to process and analyze the data. In addition, we will compare these results with the findings of the recent serial sectioning study of γ' precipitate structures by Lund and Voorhees.

**5:05 PM**
**Three Dimensional Quantitative Characterization and Representation of Microstructures in α/β Ti Alloys Using Serial Sectioning in a FIB**: *Eunha Lee*[1]; Robert Williams[1]; G. Babu Viswanathan[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

For research programs aimed at developing predictive capabilities for property/microstructure relationships it is necessary to determine appropriate three-dimensional representations of microstructure. In the present research, the microstructure of α/β Ti alloys is the subject of study, in both the β-heat treated and α/β processed conditions. Three-dimensional representations are being obtained by serial sectioning in a dual-beam Focused Ion Beam (FIB) instrument. Thus, the focused ion beam is used to continually section through a sample and at preset intervals, images of the microstructure are recorded. A series of images, typically more than 150, are then assembled into a movie using commercial software. Potential problems with this technique are discussed, for example, the imaging conditions and ion-induced contrast mechanisms, and image alignment, and solutions described. The resulting three-dimensional representations are compared with the results of robust stereological procedures using conventional two-dimensional sections. This research has been supported in part by the funding of a Focused Research program by the National Science Foundation, Division of Materials Research, with Dr. K. Linga (KL) Murty as Program Director.

**5:30 PM**
**Investigation of Crack Initiation Regions of Dwell-Fatigued Near Alpha Titanium Alloys Utilizing FIB Technology**: *Jonathan Paul Blank*[1]; Vikas Sinha[1]; G. B. Viswanathan[1]; Michael J. Mills[1]; Bahman Zoofan[2]; Stanislav I. Rokhlin[2]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]The Ohio State University, Welding Eng., 1248 Arthur E. Adams Dr., Columbus, OH 43221 USA

Titanium alloys are attractive for a number of applications in the aircraft industry due to their high specific strength, low density, excellent corrosion resistance and relatively good creep resistance up to 500°C. One of the main concerns recently has been the detrimental effects of dwell time on the fatigue life of several titanium alloys used. Understanding and predicting failure due to dwell fatigue has been very difficult because of the nature of cracks, which initiate below the surface and appear to be closely related to the local, intrinsic microstructural features. The possibility of obtaining 3D microstructural information about crack initiation regions under dwell fatigue conditions has become a reality. In-situ ultrasonic monitoring during testing and x-ray radiography after testing has been used to locate, observe and monitor crack initiation. Serial sectioning using a Focused Ion Beam (FIB) is used to expose crack initiation regions to allow for 3D characterization of the crack geometry relative to the existing microstructure and local microtexture of the crack region. The FIB has also used to "lift and pluck" TEM foils from potentially interesting areas around the crack regions to study deformation processes.

## Forming and Shaping of Light Weight Automotive Structures: Applications - I

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

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*Session Chair:* Mahmoud Demeri, Ford Motor Company, Mfg. Sys., Dearborn, MI 48121 USA

**2:00 PM Invited**
**Advances in Light Weight Auto Structures Using Advanced High Strength and Ultra-High Strength Steels**: *Dennis M. Urban*[1]; [1]Ispat Inland, Inc., R&D, 3001 E. Columbus Dr., E. Chicago, IN 46312 USA

Advanced High Strength Steels (AHSS) and Ultra-High Strength Steels (UHSS) represent two of the best opportunities for simultaneous weight reduction, cost reduction, and increased safety in the automotive industry. Recently, there has been great deal of attention on the advances in both the application of these grades and manufacturing using these materials. This presentation will discuss both the advances in design using AHSS and UHSS as well as the manufacturing processes that enable these design advances.

**2:30 PM Invited**
**Advances in Aluminum Auto Structures**: *Bruno Barthelemy*[1]; [1]Ford Motor Company, PDC Design Ctr., MD 524, 21175 Oakwood Blvd., Dearborn, MI 48124 USA

Current production Aluminum vehicles demonstrate up to 50% weight savings in BIW compared to their steel counterpart. This primary weight saving may in turn enable secondary weight savings in the chassis and powertrain areas resulting in a 25% weight saving for the complete vehicle. Even though many of the technical issues related to the Aluminum have been resolved over the last decade, no high volume production vehicles are in production. The current business equation is not affordable. On one side the variable cost penalty is too high and the first time investment is so expensive that the automotive companies are reluctant to take the risk. This presentation will highlight some of the issues that must be addressed in order to introduce Aluminum BIW in high volume production vehicles.

**3:00 PM Invited**
**Affordable Vehicle Lightweighting**: *Paul Geck*[1]; [1]Ford Motor Company, 2001 Beech Daly Rd., Dearborn Heights, MI 48121 USA

The needs to increase passenger safety, minimize vehicle weight and reduce manufacturing cost are the most significant factors driving the automotive industry to use alternative materials in their vehicles. Selection of lightweighting materials from steel, aluminum, magnesium, plastics and composites is reviewed in terms of weight reduction, performance and cost. The current automotive materials paradigms, gage & joining methods optimization and the drivers for body design are discussed. The performance and cost versus strength of steel and aluminum structures are compared. Opportunities for weight reduction in steel vehicles and the arguable case for substituting aluminum for steel to achieve below 25% weight reduction for a pick-up truck platform are presented. Indications show that weight reduction of more than 25% may be achievable only by resorting to expensive, non-ferrous materials. The current steel-based technology and the use of advanced high strength steels offer potential for cost effective weightsavings.

**3:30 PM Break**

**3:40 PM Invited**
**Cost Effective Synthesis of Ti-6Al-4V Alloy Components Produced Via the P/M Approach**: *F. H. (Sam) Froes*[1]; O. M. Ivasishin[2]; D. G. Savvakin[2]; V. S. Moxson[3]; K. A. Bondereva[2]; A. N. Demidik[2]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]National Academy of Science of Ukraine, Inst. for Metals Physics, 36 Vernadsky St., Kiev 03142 Ukraine; [3]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA

The near net shape powder metallurgy (P/M) approach to production of titanium components allows a substantial widening of the field of applications of titanium alloy, reducing the cost of parts compared to traditional processes. The most cost-effective P/M process is based on the use of blended elemental (BE) powders of titanium and masteralloys as input materials. In this paper, the P/M BE technique will be reviewed including shape making capabilities, mechanical properties and cost of parts. Examples of components, which have been produced for industries such as automobiles and aerospace, will be given.

**4:10 PM Invited**
**Aluminum Structures for Crashworthiness**: *Hikmat Mahmood*[1]; [1]Ford Motor Company, Vehicle Structural Design, 2101 Village Dr., Dearborn, MI 48124 USA

Developing crashworthy aluminum structures for automotive body represents a major challenge. Automotive components are usually made of sheet metal with complex shapes that could make manufacturing very difficult for direct material substitution. Moreover, the aluminum to be used should have strength and ductility characteristics to justify material substitution from the commonly used steel. In general aluminum does present an opportunity for weight savings and efficiency in energy management, if it is considered early in the design process. In this presentation several component and subsystem structures are presented and compared to that of steel in frontal and roof crush. In this presentation aluminum components considered are made of extrusion, casting, and sheet metal materials. Also, an application of aluminum space frame behavior in frontal crash will be discussed. The result of the space frame analysis conducted by a nonlinear collapsible finite element beam will be presented with static and dynamic test validation.

**4:40 PM Invited**
**Collaborative Development of Lightweight Materials for Automotive Structural Applications**: *Andrew M. Sherman*[1]; Phil Sklad[2]; [1]Ford Motor Company, Rsrch. Lab., MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA; [2]Oak Ridge National Laboratory, Bldg. 4515, Rm. 216, MS 6065, 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA

The Partnership for a New Generation of Vehicles (PNGV) program initiated cooperative R&D between the federal government and the United States Council Automotive Research (USCAR) to develop technologies to reduce the nation's dependence on petroleum and reduce emissions by improving fuel economy. A key enabler for the attainment of these goals is a significant reduction in vehicle weight. Thus the major focus of the PNGV (now FreedomCAR) materials program is to develop materials technologies to reduce weight by up to 40%. Various materials, including high strength steels, aluminum, magnesium, titanium, and metal and polymer-matrix composites were evaluated for weight saving potential in automotive structural applications and a prioritized ranking of challenges was developed. To address these, government and industry collaborative R&D focuses on several key areas: cost reduction of materials and processes, high-volume manufacturing technologies, design data and test methodologies, joining, and recycling and repair. This talk presents an overview of the program and a description of some of the research that is being carried out to address these challenges.

## General Abstracts – High Temperature Alloys

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Tuesday PM Room: 224/225
October 8, 2002 Location: Columbus Convention Center

*Session Chairs:* Mysore A. Dayananda, Purdue University, Sch. of Matls. Eng., W. Lafayette, IN 47907 USA; Peter K. Liaw, University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA

**2:00 PM**
**Investigation on Cr-Ta and Cr-Ta-Mo Alloys with Lamellar Structure**: Daifeng Wang[1]; *Peter K. Liaw*[1]; Chain T. Liu[2]; Lee Heatherly[2]; Easo P. George[2]; [1]University of Tennessee, Matls. Sci. & Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

A directional solidification (DS) process using a high-temperature optical-floating-zone furnace to obtain an aligned eutectic microstructure of eutectic Cr-Ta and Cr-Ta-Mo alloys was studied in order to improve the ductility and fracture toughness. The effects of DS parameters (growth speeds and rotation rates) were examined. Regularly aligned lamellar microstructures were obtained in the Cr-Ta and Cr-Ta-Mo alloys at optimum DS parameters. The influence of DS conditions and heat-treatments on the hardness at room temperature and tensile properties at room and elevated temperatures (800ºC and 1,200ºC) was investigated. It was observed that the tensile properties of the Cr-based alloys at room and elevated temperatures could be improved by obtaining aligned microstructures, and that the fracture toughness of the directionally solidified alloys at room temperature was increased. Molybdenum alloying had increased the strength of the Cr-Ta alloy in the temperature range investigated.

**2:25 PM**
**Fatigue-Crack-Growth Behavior of a Heat-Resistant Alloy**: *G. Wang*[1]; Y. Lu[1]; L. Chen[1]; R. L. McDaniels[1]; S. A. White[1]; P. K. Liaw[1]; J. D. Landes[2]; K. M. Chang[3]; G. Shen[4]; D. Furrer[4]; B. Skillings[4]; [1]University of Tennessee-Knoxville, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [2]University of Tennessee-Knoxville, Mechl. Eng., Knoxville, TN 37996 USA; [3]West Virginia University, Mechl. & Aeros. Eng., Morgantown, WV 26506 USA; [4]Ladish Company, Inc., Cudahy, WI 53110 USA

Fatigue-crack-growth tests were conducted on a heat-resistant alloy in the temperature range from 24 to 816°C in vacuum. Note that the tests at 24°C were done in air. The specimen was a center-cracked tension, M(T), geometry. And all specimens were precracked using fatigue loading at 200°C. Tests were conducted at 15 Hz in an advanced servo-hydraulic testing machine at 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. Fatigue-crack-growth rates were observed and measured via a Questar Telemicroscope System. The influence of test temperature on fatigue-crack-growth behavior was determined. The fatigue-crack-growth-rate per cycle was found to generally increase with decreasing test temperature. The fatigue-crack-propagation mechanisms were discussed. This research is sponsored by Ladish Co., Inc.

**2:50 PM**
**Directional Recrystallization of Cold-Worked Nickel**: *I. Baker*[1]; J. Li[1]; B. Iliescu[1]; B. Bollinger[1]; A. Badmos[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

The effects of annealing temperature, hot zone velocity and temperature gradient ahead of the hot zone during directional annealing have been investigated for cold-rolled nickel using both EBSP and optical microscopy. At 643K, a fine-grained, cube-textured equi-axed grain structure was produced at slower velocities, whereas the material did not recrystallize at high velocities. At 1273K, columnar grains were produced over a wide range of hot zone velocities (2-100 mm/h) when a large temperature gradient (1000°C/cm) was maintained ahead of the hot zone. The column width decreased with increasing hot zone velocity. For a low temperature gradient ahead of the hot zone, only equi-axed microstructures were produced. Under the optimum conditions, of a high temperature gradient and a velocity of 5mm/hr, directional recrystallization of fine-grained (~20 µm) cube-textured nickel recrystallized at low temperature produced a single crystal. The results will be compared with simulations of the processing. Research supported by AFOSR grant F49620-00-1-0076 and NSF grant DMI9976509.

**3:15 PM**
**Diffusion Structures and Diffusion Paths for Mo-Silicides Exposed to Al Vapor**: Edward J. Ciecko[1]; *Mysore A. Dayananda*[1]; [1]Purdue University,

Sch. of Matls. Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA

Vapor-solid diffusion couples were assembled with diffusion disks of $MoSi_2$, single crystal $Mo_5Si_3$, and $Mo_5Si_3B_x$ exposed to Al vapor produced by $Mo_3Al_8$ powder. The couples were annealed at 1000°C for various times ranging from 12 to 168 hours. Couples were examined for diffusion structures by optical and scanning electron microscopy and analyzed for concentration profiles by electron microprobe analysis. The various phases identified in the diffusion structures include $Mo(Si,Al)_2$, $Mo_3(Al,Si)_8$, $Mo(Si,Al)_2B_x$, and $Mo_3(Al,Si)_8B_x$. The diffusion structures are discussed in light of the diffusion paths. A partial isotherm is presented for the Al-Mo-Si system at 1000°C based on the diffusion paths.

**3:40 PM Break**

**4:05 PM**
**Mechanical Behavior and Dislocation Substructure in Ternary and Quaternary RuAl Alloys**: *T. K. Nandy*[1]; Q. Feng[1]; M. F.X. Gigliotti[2]; T. M. Pollock[1]; [1]University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]General Electric Company, Corporate R&D, Schenectady, NY 12301 USA

Mechanical behavior of ternary and quaternary RuAl alloys were investigated at room temperature and 973K. Additions of Nb, Mo, Pt and B have been investigated. Flow stress data indicate that increasing ruthenium results in strengthening of the alloys. Incorporation of boron leads to additional strengthening. No significant alloying effect on the strain rate sensitivity was observed. Deformation at high temperature is characterized by lower strain rate sensitivities as compared to the room temperature deformation for all alloys. Based on the macroscopic flow parameters, possible mechanisms for the high temperature deformation are discussed. Results of detailed dislocation studies on selected compositions following room and high temperature deformation to study the effects of alloying additions on the dislocation behavior of the alloys will be discussed.

**4:30 PM**
**Microsample Characterization of Coatings for GRCop-84 for High Temperature High Heat Flux Applications**: *Piyush Jain*[1]; [1]Johns Hopkins University, Mechl. Eng. Dept., 3400 N. Charles St., Latrobe Hall, Baltimore, MD 21218 USA

NASA Glenn has developed GRCop-84, a high conductivity, high strength copper alloy for application in rocket motor combustion chamber liners, nozzle ramps and other actively cooled structures. Metallic coatings such as NiCrAlY and Cu-8-30%Cr are being developed for prohibiting blanching, reducing 'dog-house' failures, increasing operating temperatures and decreasing cooling requirements. Microsample tensile testing approach has been adopted to develop a fundamental understanding of the substrate-coating interaction that occur during thermal cycling. Measurement of high temp CTE, Young's modulus and tensile, creep and fatigue strength of both as deposited and thermally cycled coatings will help in characterizing small-scale and high-scale specific coating and properties. Microsample testing results will be integrated with microstructural observation and finite element analysis to provide a mechanistic foundation for coating optimization and life prediction modeling.

**4:55 PM**
**Concentration Dependence of the Crystalline Lattice Parameter of the Ni3Al Phase**: *Marina Vladimirovna Fedorischeva*[1]; *Denis V. Esikov*[2]; *Eduard V. Kozlov*[1]; [1]Institute Strength Phisics and Material Science, Acflemicheskii 2/1, Tomsk 634055 Russia; [2]Tomsk State University, Lenin Av., Tomsk 634050 Russia

The widely known intermetallic compound Ni3Al represents the interest because of positive temperature dependence due to its mechanical properties. The fine structure of the Ni3Al intermetallic is not investigated in detail. Most debatable is the value of lattice parameter and its concentration dependence. A material of research was the Ni3Al intermetallic with density 99% obtained by a method of self-propagation high-temperature synthesis under compression with aluminium concentration (20-27 at.%). The thin structure was examined by X-ray analysis. The concentration dependence of a crystal lattice parameter of the Ni3Al intermetallic was compared with a concentration dependence calculated by the Wegards' low. It was established, that there is high super structural contraction. In the present activity the lattice parameter of •'-phase of the Ni3Al intermetallic was measured in concentration interval 20-27at.%. The reliable values of a lattice parameters for •'-phase of a stoichiometric compound was obtained. The relation a(ñ) of a crystal lattice parameter is considered at deviation from a stoichiometric composition. The considerable deviation from a Vegard's rule for Ni-Al system is established and the hypothesis about reasons of this phenomenon is expressed. The analysis of a concentration dependence of the long-range order parameter is carried out. The static displacement of atoms from equilibrium position are studied. They are linearly are connected with root-mean-square long-range order parameter. It was earlier supposed, that the super structural contraction is due to decreasing of the size of an ion of aluminium at fusion it with a nickel. Last is connected to reallocation of ð-electrons of aluminium in a d-zone of a nickel. It is necessary to mark, that deviation from a Vegard's rule in the field of existence of the short-range order (5-12 at.% Al), appears in the greater degree. Accordingly, the values of the sizes of ions of aluminium are lower in the field of a •-phase (phase with the short-range order), than for alloys with the long-range order parameter (•'-phase of the Ni3Al intermetallic. The data of calculation of the size of an ion of aluminium in alloy of a system Ni-Al are adduced. Doubtlessly there is a connection between redistribution of electrons of ð-aluminium both d-nickel and resizing of ions of aluminium and abnormal temperature relation of •'-phase.

**5:20 PM**
**Structure and Properties of the Eutectic Ti-Ti5Si3 Alloys with Nb**: *Nataliya V. Antonova*[1]; *Daniel B. Miracle*[1]; Sergey A. Firstov[2]; [1]Wright Patterson Air Force Base, Air Force Rsrch. Lab., Matls. & Mfg. Direct. (AFRL/MLLM), 2230 Tenth St., Dayton, OH 45433-7817 USA; [2]Institute for the Problems of the Materials Science NASU, 3 Krzhizhanovski St., Kiev-142 03680 Ukraine

Discontinuously reinforced Ti alloys represent a class of materials with significant promise for improved structural properties and elevated temperature performance. Knowledge of structure/properties relationships are required to establish the possible applications for this class of material. Investigation of the alloys on the base of the Ti-Al-Si system in a/a2 phase region with Nb additions 2.5, 3.5 and 5 at.% is emphasized. The objective of the present study included to establish phase relationships in the Ti-rich region with participation of a liquid phase and at 800°C; study influence of Nb on the structure and properties of the Ti-Al-Si alloys. Alloys have been prepared by arc-melting technique. Selected alloys were heat treated at 1350°C for 24 h and then homogenized at 800°C for 1000h, followed by IWQ. The structure was characterized by means of X-Ray diffraction, DTA, EPMA, SEM and TEM. Vickers' hardness of the primary titanium grains has been tested. Calculation program "Thermo-Calc" was applied to represent phase diagram. Ti-Si phase diagram is discussed. Additions of Nb to the Ti-Al-Si alloys were found to change the morphology of the primary Ti grains and as a result hardness of the grains was enhanced. At 5 at.% Nb, secondary silicide Ti5Si3 precipitates occurred. Ti3Si phase has been stabilized due to formation of continuous solid solution (Ti,Nb)3Si with isostructural Nb3Si phase.

## Interstitial and Substitutional Solute Effects in High Temperature Materials and Alloys: Intermetallics

*Sponsored by:* Structural Materials Division, SMD-High Temperature Alloys Committee, SMD-Structural Materials Committee
*Program Organizers:* Mark L. Weaver, University of Alabama, Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA; Michael E. Stevenson, Metals and Materials Engineering, Suwanee, GA 30024 USA

Tuesday PM — Room: 213
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* Mark L. Weaver, The University of Alabama, Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487-0202 USA; Michael E. Stevenson, Metals and Matls. Eng., Suwanee, GA 30024 USA

**2:00 PM**
**Double Kink Nucleation on Partial Dislocations: A New Model for Solution Hardening and Softening**: Terence E. Mitchell[2]; *Peter M. Anderson*[1]; M. I. Baskes[2]; S. P. Chen[3]; R. G. Hoagland[2]; Amit Misra[2]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210-1179 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Nucl. Matls. Tech. Div., NMT-16, MS G721, Los Alamos, NM 87545 USA

Double kink nucleation and motion on partial dislocations are examined by elasticity theory for materials with a high Peierls stress. The stacking fault is found to affect kink nucleation, propagation, and annihilation. Good agreement is obtained between model and experiment for an intermetallic, MoSi2, which softens for alloying elements (V, Nb, Cr, Al) that decrease the stacking fault energy, and which hardens for Re additions that increase the stacking fault energy. For W additions, normal hardening effects from size and modulus misfit of the solute may mask the modest amount of softening predicted from the decrease in stacking fault energy. The model requires that dislocations are dissociated by more than a few atomic distances; hence the analysis does not apply to materials such as bcc metals which have only a core dissociation.

**2:20 PM**
**Dynamic Strain Aging in Polycrystalline RuAl Alloys**: *T. K. Nandy*[1]; Q. Feng[1]; M. F.X. Gigliotti[2]; Tresa M. Pollock[1]; [1]University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]General Electric Company, Corporate R&D, Schenectady, NY 12301 USA

Flow behavior of RuAl intermetallics alloys has been investigated in a temperature range of 298-1173K at different strain rates ($10^{-4}$ to $10^{-2}s^{-1}$). Stress-strain curves at temperatures ranging from 773-973K showed evidence of serrated flow. This was accompanied by an occurrence of yield points, flow stress plateau, maxima in the work hardening rate and minima in the strain rate sensitivity, which are typical features of dynamic strain aging (DSA). The characteristics of the flow behavior in these intermetallic alloys have been compared to NiAl, which also exhibits dynamic strain aging in a similar temperature-strain rate regime. Static aging experiments were performed to examine the role of solute atoms in the strain-aging phenomenon. The general deformation characteristics of RuAl will be discussed along with possible mechanisms for the DSA behavior.

**2:40 PM**
**Hydrogen-Induced Internal Friction Behavior in Ordered Intermetallic Compound of $Ni_3Al$ Single Crystal**: *Chuanbin Jiang*[1]; [1]Chinese Academy of Sciences, Inst. of Metal Rsrch., Shenyang Natl. Lab. of Matl. Sci., 72 Wenhua Rd., Shenyang, Liaoning 110016 China

Anelastic relaxation behavior of hydrogen atomic diffusion in $Ni_3Al(B)$ was investigated in single crystal by an inverted torsion pendulum after high content hydrogen (about 1700 ppm) was introduced. Two internal friction peaks emerged in the hydrogen-charged specimen(there is not internal friction peak between 150K to 550K in the hydrogen-free specimen). A small peak $P_0$ emerged at about 270K with $Q^{-1}$ about 0.00019 and a big peak $P_1$ at about 387K with $Q^{-1}$ 0.00144 at frequency of 4.6 Hz. The activation energy of these two peaks is 0.66eV and 1.01eV respectively. The configuration of these two defects was investigated by different oriented specimens and which suggested that $P_0$ was caused by the reorientation of H-H pairs and $P_1$ caused by B-H pairs. The results give sidelight evidence to the motion of hydrogen atoms in FCC metals.

**3:00 PM**
**Site Preference Occupation for Ni, Ti, and Cr Substituting for Fe in D0_3 Phase of FeAl Alloy**: *Simon Dorfman*[1]; David Fuks[2]; Vlad Liubich[2]; Larisa Kutsenko[2]; [1]Technion, Phys., Haifa 32000 Israel; [2]Ben-Gurion University of the Negev, Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel

We presented the results of ab initio study of the site preference occupation for Ni, Ti, and Cr substituting for Fe in the D0_3 phase of FeAl alloy. We demonstrated by the direct calculations of the total energies that Ni occupies Fe positions in the first sublattice, Ti occupies Fe positions in the second sublattice, and Cr does not show any preference in substitution of Fe in the alloy. We demonstrated also that Ni addition to the D0_3 phase does not change the type of bonding in the alloy, while Cr addition leads to sufficient hybridization of Cr d-electrons with s- and p-electrons of Al when the concentration of Cr increases.

**3:20 PM Break**

**3:35 PM**
**The Intensity of Dislocation Accumulation and Work Hardening of Ni3Ge Intermetallic Single Crystals with the L12 Structure**: *Nina Alexandrovna Koneva*[1]; *Julia Vladimirovna Solov'eva*[1]; Eduard Viktorovich Kozlov[1]; Vladimir Alexandrovich Starenchenko[1]; [1]Tomsk State University of Architecture and Building, pl. Solyanaya 2, Tomsk, Russia

The peculiarities of the work hardening in ordered L12 alloys are connected with the presence of Kear-Wilsdorf locks and the locks formed by point defects precipitation on the edge dislocations. These two mechanisms influence both to the magnitude of the critical resolved shear stress and to the work hardening rate. This latter is connected with the change of the dislocation accumulation rate. Blocking of superdislocation segments leads to the occurrence of dipole dislocation configurations. The evolution of deformation microstructure with temperature of Ni3Ge single crystals was investigated experimentally by TEM. The intensity of dislocation accumulation was measured. The contributions to the flow stresses determined by different dislocation mechanisms were estimated. The peculiarities of the dislocation structure were analyzed with the connection of the temperature anomaly of the flow stresses in the alloys with the L12 structure.

**3:55 PM**
**Model of Deformation and Thermal Strengthening of Single Crystals of Alloys with the L12 Structure**: *Vladimir Alexandrovich Starenchenko*[1]; *Julia Vladimirovna Solov'eva*[1]; *Nina Alexandrovna Koneva*[1]; A. N. Gavrilov[1]; O. D. Pantyukhova[1]; [1]Tomsk State University of Architecture and Building, 634003, pl. Solyanaya 2, Tomsk, Russia

Deformation causes generation and annihilation of the crystal lattice defects in the alloys with the L12 structure. Simultaneously the change in the alloy long-range atomic order takes place. That influences on the intensity of the deformation defects accumulation. Specific mechanisms of blocking superdislocations: Kear-Wilsdorf mechanism for screw dislocations and climbing for edge dislocations manifest itself in the alloys with the L12 structure. In the work the processes of accumulation and annihilation of superdislocations, generation and annihilation of point defects are simulated. In this case one takes into account the accumulation of the superdislocations locks of screw and edge orientations as well as the influence long-range atomic order state of the alloy on this process. The long-range order destruction caused by deformation is simulated. It is assumed that simultaneously the thermal restoration of the long-range order due to vacancy migration takes place. A set of simultaneous differential equations defining the deformation curves of alloys with the L12 structure against temperature was obtained.

**4:15 PM**
**Model of Plastic Deformation and Thermal Strengthening of the L12 Single Crystals**: *Vladimir Alexandrovich Starenchenko*[1]; J. V. Solov'eva[1]; N. A. Koneva[1]; A. N. Gavrilov[1]; O. D. Pantyukhova[1]; [1]Tomsk State University of Architecture and Building, pl. Solyanaya 2, Tomsk, Russia

Deformation causes generation and annihilation of the crystal lattice defects in the alloys with the L12 structure. Simultaneously the change in the alloy long-range atomic order takes place. That influences on the intensity of the deformation defects accumulation. Specific mechanisms of blocking superdislocations: Kear-Wilsdorf mechanism for screw dislocations and climbing for edge dislocations manifest itself in the L12 alloys. The processes of accumulation and annihilation of superdislocations, generation and annihilation of point defects are simulated. One takes into account the accumulation of the superdislocations locks of screw and edge orientations as well as the influence long-range atomic order state of the alloy on this process. The long-range order destruction caused by deformation is simulated. It is assumed that simultaneously the thermal restoration of the long-range order due to vacancy migration takes place. A set of simultaneous differential equations defining the deformation curves of alloys with the L12 structure against temperature was obtained.

**4:35 PM**
**Flow Stresses Relaxation in the Ni3Ge Intermetallic Crystals with Different Compression Axis Orientations**: *Eduard Viktorovich Kozlov*[1]; Julia Vladimirovna Solov'eva[1]; V. V. Norkin[1]; *Nina Alexandrovna Koneva*[1]; Vladimir Alexandrovich Starenchenko[1]; [1]Tomsk State University of Architecture and Building, pl. Solyanaya 2, Tomsk, Russia

Experiments with stress relaxation turned to be the sources of information about thermoactivated deformation processes. These experiments allow to determine the effective activation volume of thermoactivated dislocation glide. In this paper the flow stresses relaxation of the intermetallic single crystal Ni3Ge with the [001], [234], [139] compression axes orientations is examined. The test temperature was varied from 77K to 700K. The relaxation curves were investigated at different degrees of plastic deformation within the interval from 0 to 20%. The activation volumes were determined from relaxation curves. The dependencies of activation volumes on the strain degree, deforming stresses and dislocation densities are compared with the temperature dependencies of the yield strength and flow stresses. It was established that the change in the activation volume turned to be multistage. Possible mechanisms and nature of stress relaxation in intermetallics with the L12 are discussed.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Fracture Mechanics and Mechanisms

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

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| Tuesday PM | Room: 216 |
| October 8, 2002 | Location: Columbus Convention Center |

*Session Chairs:* John Hancock, University of Glasgow, Dept. of Mechl. Eng., Glasgow G12 8QQ Scotland; Clyde Briant, Brown University, Div. of Eng., Providence, RI 02912 USA

**2:00 PM**
**Ductile Fracture Mechanisms in Low Alloy Steels**: *Clyde L. Briant*[1]; Erik T. Sylven[1]; Alan Needleman[1]; M. Shabrov[1]; Leo Chuzhoy[2]; Donald

Sherman[2]; [1]Brown University, Div. of Eng., 182 Hope St., Providence, RI 02912 USA; [2]Caterpillar, Caterpillar Techl. Ctr., PO Box 1875, Peoria, IL 61656-1875 USA

This paper will present a study of the origins of ductile fracture in an SAE 4130 modified steel. Samples for this study were cooled at different rates and were tested using round samples of different notch radii to affect the degree of triaxial stress. The primary particles in the steel were MnS and Ti(C,N). It was found that in almost all cases the Ti(C,N) particles cracked rather than debonding from the matrix. The load at which cracking occurred increased as the notch radius decreased. It was found that there was a critical load, below which no cracking was observed. As the load was increased beyond this point, particles began cracking in the center of the sample; the volume of material that contained cracked particles grew from the center to the surface with increasing load. It was also found that there was little growth of the voids until immediately before fracture.

**2:25 PM**

**Influence of Mesotexture on Effective Grain Size and Microstructure-Toughness Relationship in Thermomechanical Control-Rolled Microalloyed Steels**: *Debashish Bhattacharjee*[1]; Claire L. Davis[1]; John F. Knott[1]; [1]The University of Birmingham, Sch. of Eng., Metall. & Matls., Edgbaston, Birmingham B15 2TT UK

Prediction of impact toughness in ferrite-pearlite structural steels requires an input of average ferrite grain size. This is due to impact energy values comprising of both crack initiation and propagation components and propagation energy being influenced by crack path deviations at grain boundaries. However, it has been shown that single cleavage facets in thermomechanically control rolled (TMCR) microalloyed steels can consist of multiple grains with low angle boundaries (up to 11°) between them. Thus the average microstructural unit experienced by the crack front (i.e., cleavage facet) is significantly larger than the average metallographic, 2-D grain size. It has been shown in this paper that 2-D grain size is insufficient information in TMCR microalloyed steels for the prediction of toughness. In these steels it is necessary to use an "effective grain size" based on the metallographic grain size and mesotexture for adequate toughness predictions. This paper also shows how different reheating and rolling schedules affect the development of mesotexture in a microalloyed steel.

**2:50 PM**

**Fracture Behaviour of Plate Steels Having Dual-Phase Microstructures**: *J. Douglas Boyd*[1]; Peter Poruks[1]; Igor Yakubtsov[1]; [1]Queen's University, Dept. of Mechl. Eng., Kingston, Ontario K7L 3N6 Canada

Advanced thermomechanical processing and accelerated cooling of low-C microalloyed steels are employed to produce a fine (intragranularly nucleated) bainite microstructure, designated "acicular ferrite + martensite/austenite" (AF+M/A). This dual-phase microstructure exhibits the best combination of strength and impact toughness in steels having low alloy concentrations (low C-equivalent). The mechanism of ductile fracture in steels having dual-phase microstructures is studied by measuring damage accumulation (vol.% voids) as a function of true strain in tensile tests for several steels having a range of % M/A. The damage accumulation data are fitted to the ductile fracture models of i)Brown and Embury and ii)Thomason, and qualitative correlations are made with Charpy impact data. Results will also be presented on the brittle fracture behaviour of dual-phase AF+M/A steels determined from instrumented Charpy impact tests.

**3:15 PM**

**Elastic Perfectly-Plastic Crack Tip Fields in Plane Stress and Plane Strain**: *John Hancock*[1]; [1]University of Glasgow, Dept. of Mechl. Eng., Scotland

The asymptotic fields for a stationary crack in mode I and mixed mode I/II loading are considered in plane stress and plane strain conditions. The assumption that plasticity surrounds the tip at all angles is relaxed, so that without loss of generality the fields are considered to comprise elastic and perfectly plastic sectors. Rice has shown that under plane strain conditions, the equilibrium equations combine with the yield criterion and the assumption that the crack tip stresses are bounded leads to two simple conditions in the plastic sectors. In plane strain, either the mean stress or the shear stress in cylindrical coordinates is independent of angle. Similarly, in plane stress either the mean stress is independent of angle or the radial stress deviator is zero. In both plane stress and plain strain these conditions lead to constant stress or sectors or fans, although the form of the stresses is fundamentally different in plane stress and plane strain. The elastic and plastic sectors are assembled subject to the requirement of continuity of tractions the sector boundaries. In plane strain this leads to a family of fields parameterised by constraint (mean stress). As a result the toughness in cleavage conditions and the resistance to ductile tearing depends on constraint, which is a function of geometry size and loading. However, in plane stress conditions a single field emerges in the leading sector. The plane strain and plane stress slip line fields are verified computationally using modified boundary layer formulations and the implications for geometry and size dependent fracture toughness are discussed.

**3:40 PM**

**Notch Fracture Mechanics in Solids Prone to Strain Localization:** Donato Firrao[1]; [1]Politecnico de Torino, Dip. Di Scienza dei Materiali Corso Duca Degli Abruzzi 24, 1-10129 Torino, Italy

Abstract Text Unavailable

**4:05 PM Break**

**4:20 PM**

**Molecular Dynamics Simulations of Shear Banding and Fracture in Amorphous Metals**: *Michael L. Falk*[1]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

We have simulated the fracture of a model bulk metallic glass using molecular dynamics simulation. The effect of interatomic potential on the ductility of the glass at low temperature has been investigated. In addition the effect of the liquid state prior to quenching has a significant effect on the mechanical properties of the low temperature glass. We will discuss relationships between these observations and micromechanical models of deformation in noncrystalline solids. These models describe the microstructure in terms of state variables that correspond to the number of "shear transformation zone" regions in the glass. Connections will also be made to recent investigations of dynamical structure in supercooled liquids.

**4:45 PM**

**The Local Conditions for Void Initiation in Materials with Second Phase Particles**: *Otmar Kolednik*[1]; Ilshat Sabirov[1]; Dominik Duschlbauer[2]; Heinz Pettermann[2]; [1]Austrian Academy of Sciences, Erich Schmid Inst. of Matls. Sci., Leoben A-8700 Austria; [2]Vienna University of Technology, Inst. of Light Weight Struct. & Aeros. Eng., Vienna A-1010 Austria

Stereo image pairs are taken from fracture mechanics specimens in the scanning electron microscope. The stereo image pairs are analyzed automatically by a recently developed digital image analysis system. The key part of the image analysis system is a matching algorithm which is able to find homologue points in the two images. The system provides a three-dimensional model of the depicted region consisting of 20000 to 40000 points. Corresponding regions on both halves of the broken fracture mechanics specimens are analyzed. Crack profiles are extracted perpendicularly to the pre-fatigue crack front. The profiles are drawn so that each crosses a particle that is located close to the crack front. For each particle, its size and location with respect to the crack tip are determined. From the corresponding profiles, the critical crack tip opening displacement, which is a measure of the local fracture initiation toughness, and the crack tip opening displacement in the moment of void initiation are determined. The global composite stress tensor at the moment of void initiation is calculated for each individual location of a particle with the HRR-field theory, treating the composite as a homogeneous material. The maximum particle normal stress is computed by a Mori-Tanaka type mean field approach which accounts for different material properties of matrix and inclusion, the inclusion shape and orientation, and the inclusion volume fraction. The investigation is conducted for different types of steels, aluminum alloys and metal matrix composites.

**5:10 PM**

**Continuum-Based Costitutive Behavior of Hot Fracture of Aluminum Alloys During Thermomechanical Processing**: *Paul T. Wang*[1]; [1]Alcoa, Inc, Process Tech. Div., 100 Technical Dr., Alcoa Center, PA 15069 USA

Constitutive behavior of hot fracture for aluminum alloys during thermomechanical processes is investigated at a continuum scale in which the effects of stress, strain, strain rate, and temperature on fracture are considered. Sets of hot tensile, compression, and torsion tests of high Mg aluminum alloys were performed under the control of prestrain, strain rate and temperature. Two essential hot fracture mechanisms were observed, which are ductile fracture and hot shortness. Optical microscopy and SEM were used to characterize the local hot fracture morphology. To establish a constitutive equation of hot ductile fracture for aluminum alloys, two types of fracture constitutive models are considered. These are a state equation type and a void evolution type models. The state equation type incorporates effective strain to fracture, hydrostatic stress, and a temperature compensated strain rate as principle parameters, while the void evolution type considers nucleation and growth of voids prior to fracture. Experimental results from hot tensile, compression and torsion tests were used to establish the models. In the case of ductile fracture, it was found that the strain to fracture depends on two parameters, the stress and a parameter which includes a combined effect of temperature and strain rate. Potential applications of these fracture constitutive models to hot metal forming of aluminum alloys are discussed.

**5:35 PM**
**Improvement of Crack Arrest Toughness by Ultra-Fine-Grained Microstructure in Surface Layers of Steel**: *Tadashi Ishikawa*[1]; [1]Nippon Steel Corporation, Steel Rsrch. Labs., 20-1, Shintomi, Futtsu, Chiba 293-8511 Japan

Brittle fracture is one of the major causes of serious damage to steel structures when they have an accident. The structure integrity will be increased by the use of steel having higher crack-arrest toughness (arrestability) for important structural members of ships. Studies on the crack arrestability of steel have revealed that the shear lips formed at the surface of steel when brittle cracks propagate offer great resistance to the further propagation of these cracks. This knowledge led to the development of steel (SUF steel) having ultra fine grains at the surface and in the subsurface region (surface layers) that promote the formation of shear lips. In the present paper, the mechanism of brittle crack arresting behaviour is discussed using SUF steel plates. Large-scale fracture model tests are also conducted to demonstrate the validity of steel plates having high crack arrestability on structure integrity.

## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Functional Materials - II

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Tuesday PM — Room: 214
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* Mehmet Sarikaya, University of Washington, Matls. Sci. & Eng., Seattle, WA 98195 USA; N. J. Kim, Center for Advanced Aerospace Materials, Pohang 790-330 Korea

**2:00 PM**
**Materials, Structure and Applications of Some Advanced MEMS Devices**: *Sungho Jin*[1]; [1]University of California-San Diego, Dept. of Mechl. & Aeros. Eng., Bldg. EBU 2, 9500 Gilman Dr., La Jolla, CA 92093-0411 USA

Since the famous lectures by Richard Feynman, “There is Plenty Room at the Bottom” in 1959 and “Infinitesimal Machinery” in 1983, the drive toward nano- and micro- materials/devices has been one of the most fascinating scientific and technological goals. While the design and fabrication of standard MEMS devices can relatively easily be accomplished through available MEMS foundries, differential advantages such as new functionalities and improved reliability may more readily be obtained by incorporating new/advanced materials, for example, nano materials or active functional materials. In this presentation, some unique approaches related to the advanced materials, design of MEMS structures and potential applications of electron-emitting MEMS, optical MEMS, and RF microswitch MEMS will be discussed.

**2:30 PM**
**Nanoscale Phenomena in Synthetic Functional Oxide Heterostructures: Role of Interfaces**: *Ramamoorthy Ramesh*[1]; [1]University of Maryland, Matls. Sci. & Eng., College Park, MD 20742 USA

As we gather together to celebrate Gareth’s 70th birthday, I can't help but stand back and marvel over the impact he has had as an advisor, mentor, colleague or friend to all of us. Personally, I know that I have been enriched in many ways by my association with “GT”. After I graduated from his group, I spent three years exploring the structure and microstructure of HTSC materials (both bulk and thin films) at Bellcore, primarily working at the National Center for Electron Microscopy at Berkeley. In 1991, I started a basic science program that was aimed at creating model perovskite oxide heterostructures using ferroelectrics as a case study. Our original goal was to study the rather enigmatic problem of polarization fatigue using epitaxial films as model systems. However, in the process of doing so, we discovered a novel solution to this problem, namely the use of conducting oxide electrodes as contacts rather than conventional metals such as Pt. The key here, of course, is the atomic scale microstructure of electrical interfaces in the ferroelectric capacitor. A considerable amount of intellectual property based on this invention has been generated and is currently being licensed for development and manufacture. Over the past six years, we have focused considerable effort on understanding the growth and characterization of functional oxide thin films and heterostructures, specifically ferroelectric, dielectric and magnetic perovskites. Using both epitaxial and polycrystalline materials on a variety of substrates as test vehicles, we have been carrying out systematic studies on the effect of composition, point defect chemistry, strain and processing variables on the microstructure and physical properties. A novel aspect of our work is the combined use of focused ion beam milling and scanning force microscopy techniques to understand the influence of film microstructure on the relevant properties at the nanoscale. This presentation will address two problems and approaches to understand and solve them: (i) the materials science of thin conducting barrier layers that enable the integration of the complex oxides on a silicon surface; (ii) domains in epitaxial films and mapping of their nanoscale dynamics using scanned force microscopy. My presentation will have two components. The first part will summarize some of the key discoveries (enabled through the use of advanced electron microscopy techniques) in the area of perovskite oxides. In the second part, I will focus on some of our recent observations on nanoscale phenomena in ferroelectric thin films using scanned probes. In this presentation, I will describe some possible areas where fundamental measurements in conjunction with theoretical studies and modeling will enable a better understanding of the complex phenomena involved in these materials, especially the role of structural, chemical and functional interfaces (such as domain walls). The work at Maryland is supported by the NSF-MRSEC.

**3:00 PM**
**Annealing Behavior and the Magnetic Properties of Cold-Deformed Fe-Pd Based Intermetallic Alloys**: Jorg M. Wiezorek[1]; *Anirudha R. Deshpande*[1]; *Amal A. Al-Ghaferi*[1]; Huiping Xu[1]; [1]University of Pittsburgh, Matl. Scis. & Eng., 864 Benedum Hall, Pittsburgh, PA 15260 USA

Fe-Pd alloys of equiatomic (Fe50Pd50) or slightly Pd-rich (Fe42Pd58) composition are attractive for permanent magnet applications. Property optimization depends on development of detailed understanding of microstructural evolution during annealing that induces the order transformation. The two chosen compositions are of interest because Fe42Pd58 orders congruently, whereas Fe50Pd50 exhibits a two-phase region. Here the evolution of grain size as a function of the competition between recovery and recrystallization phenomena and the order-disorder transformation is studied systematically. The alloys have been deformation processed by cold rolling and equal-channel angular pressing. The cold deformed alloys are annealed at systematically varied temperatures below the critical order-disorder temperature. Grain sizes and grain morphology are determined by SEM and TEM. The order-disorder transformation and other solid-state reactions during annealing are characterized by DSC. Magnetic hysteresis behavior of these annealed samples is determined by VSM. An understanding of the effect of annealing temperature on the grain size and the magnetic properties was developed for these two different alloys.

**3:20 PM Break**

**3:35 PM**
**Microstructural Study of Rapid Age Hardening in Al-Mg-Cu (Cu-Lean) Alloys**: *Libor Kovarik*[1]; Perena Gouma[2]; Christian Kisielowki[3]; Steve A. Court[4]; Michael J. Mills[1]; [1]The Ohio State University, Watts Hall #477, 2041 College Rd., Columbus, OH 43210 USA; [2]State University of New York at Stony Brook, Matl. Sci. & Eng., Old Engineering 201, Stony Brook, NY 11794 USA; [3]National Center for Electron Microscopy, Ernest Orlando Lawrence Berkeley Natl. Lab., One Cyclotron Rd., Berkeley, CA 94720 USA; [4]Alcan International, Ltd., Banbury Lab., Southam Rd., Banbury, Oxfordshire OX162SP UK

Optimization of the properties of Al-Mg-Cu alloys may be practiced if there exists a better understanding of the rapid hardening process that proceeds after several minutes of aging. In some applications, this hardening provides the major source of strengthening. Currently, the exact cause of the rapid hardening is disputed in the literature. In our study of Al-Mg-Cu alloys (Cu:Mg ratio = 0.14), the rapid hardening issue is addressed by High Resolution Transmission Electron Microscopy (HRTEM) together with reconstruction of the exit wave from the focal series. The presence of {110} super-lattice reflections is detected in the FFT spectra of HRTEM images, which suggests formation of ordered regions (GPB zones) within the matrix prior to S?? formation. The possible crystal structure models of GPB zones will be discussed based on our experimental evidence and HRTEM image simulations. The mechanism of strengthening based on the presence of diffuse, ordered GPB zones will also be discussed.

**3:55 PM**
**Microstructure Design of Advanced Materials: WC-Co Cermets and Their Novel Architectures**: *K. S. Ravi Chandran*[1]; Zak Fang[1]; [1]The University of Utah, Metallurgl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Design and development of advanced materials for superior strength and toughness is a perpetual effort in meeting the material needs for demanding applications. The WC-Co cermet is one of the truly advanced materials, due to its unique combination of properties. Although such cermets are widely used, further expansion of their use is limited because of low toughness. The mechanical basis for their excellent properties is not fully understood. Recently, a new composite WC-Co cermet based on a novel architecture has been discovered. In this research, origins of the unique mechanical properties of these cermets are closely examined using some microstructure-based models. It is shown that the superior properties arise as a result of the spatial arrangement of WC grains within Co, the strength and stiffness of WC and the constrained plastic deformation behavior of high strength Co binding the WC grains. It is also shown that the high toughness of "functional" cermets with hierarchical microstructures can be understood on the basis of microstructure-based mechanical models. The microstructure-models illustrate pathways for designing advanced composite materials based on the architecture of WC-Co cermets.

**4:15 PM**
**Microstructure-Property Relationships of Nanostructured Al-Fe-Cr-Ti Alloys**: *Leon L. Shaw*[1]; M. Zawrah[1]; J. Villegas[1]; H. Luo[1]; D. Miracle[2]; [1]University of Connecticut, Metall. & Matls. Eng., Storrs, CT 06269 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45431 USA

Nanostructured Al93Fe3Ti2Cr2 alloy powder was prepared via mechanical alloying (MA), followed by either hot press or extrusion to form bulk material. The microstructural stability of the alloy was characterized using a variety of analytical instruments including differential scanning calorimetry, X-ray diffraction, scanning electron microscopy, and transmission electron microscopy. Mechanical properties were measured via microhardness and compression tests. It was found that MA-processed Al93Fe3Ti2Cr2 alloys had substantially higher hardness and compressive strength than that of 6061Al, but with very limited ductility. The enhanced strength is attributed to grain refinement, formation of supersaturated solid solutions, and intermetallic precipitates.

**4:35 PM**
**A Peierls Model of the Critical Stress to Transmit a Screw Dislocation from a Disordered to an Ordered Phase**: *Yao Shen*[1]; *Peter M. Anderson*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

This presentation will describe the features and predictions of a 2D Peierls model of screw dislocation transmission from a disordered to an ordered phase. In this model, perfect dislocations in the disordered and ordered phases are represented by a distribution of N model Volterra dislocations with fractional Burgers vector, db=b/N. The incoming and outgoing slip planes, and also the interface, are described by a local shear stress-shear displacement relation that is derived from the gamma surface for that region. Consequently, the configuration of the dislocation core changes during the transmission process, including potential core spreading into the interface. The results of an idealized case are presented in which a pair of screw dislocations in the disordered phase are pushed into the ordered phase along slip planes which are oriented perpendicular to the interface. The Burgers vectors of the two phases are such that no residual content remains in the interface following transmission. The effect of unstable stacking fault energies associated with the slip planes and interface, the mismatch in elastic moduli, and the antiphase boundary energy of the ordered phase are varied to understand the effect on the critical stress for dislocation transmission.

**4:55 PM**
**Prediction of Property/Microstructure Relationships in Alpha/Beta Ti Alloys for Tensile Deformation at Room Temperature**: *Rajarshi Banerjee*[1]; Gopal Babu Viswanathan[1]; Mary C. Juhas[2]; Yunzhi Wang[1]; John W. Wilkins[3]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]The Ohio State University, Col. of Eng., 122 Hitchcock Hall, 2070 Neil Ave., Columbus, OH 43210 USA; [3]The Ohio State University, Dept. of Physics, Columbus, OH 43210 USA

The notion of microstructural design for a set of targeted mechanical properties lies at the heart of the problem of the accelerated maturation of materials. Thus, in the absence of an integrated set of theoretical and experimental tools to permit the prediction of properties for a material possessing a given microstructure, the costs of alloy development will be too high and development cycles too long for useful engineering practice. To effect such a set of modeling tools for materials design, a comprehensive research program has been initiated involving a number of academic institutions, national laboratories and industrial companies. The research is aimed at providing predictive tools for property/microstructure relationships and the evolution of microstructure under a combination of stress and thermal exposure. The inputs to these models include predictions provided by fundamental ab-initio calculations, and the development of these types of computational tools is also a component of the research. An example of the prediction of tensile properties at room temperature in Ti alloys will be given which demonstrates the integration of modeling and experiment over the wide range of length scales implicit in this problem. This research is supported in part by the National Science Foundation, The US AFOSR, the US AFRL and a number of partnering companies.

## Microstructure/Property Relationships in Alpha/Beta Titanium Alloys: Processing Effects on Behavior

*Sponsored by:* Structural Materials Division, SMD-Titanium Committee
*Program Organizers:* Patrick L. Martin, US Air Force, AFRL/MLLM, WPAFB, OH 45433 USA; James Cotton, The Boeing Company, Seattle, WA 98124-2499 USA

Tuesday PM — Room: 220/221
October 8, 2002 — Location: Columbus Convention Center

*Session Chairs:* J. R. Wood, Allvac, Monroe, SC 28111-5033 USA; Pat Martin, Air Force Research Laboratory, Metals Branch, WPAFB, OH 45433-7817 USA

**2:00 PM Invited**
**Experiences with Titanium Alloys for Liquid Rocket Engine Applications**: *John A. Halchak*[1]; [1]Boeing, Rocketdyne Div., 6633 Canoga Ave., Canoga Park, CA 91304 USA

Their severe operating environments characterize liquid rocket engines. These include temperature extremes, reactive propellants, thermal shock, high pressures, extreme acoustic environments and destructive modal vibrations. The high strength-to-weight ratios of titanium alloys make them attractive for application in rocket engines–offering the potential of superior performance while minimizing component weight. Investigations into applications of titanium alloys began in the early 1960's. However, it was found that the reactivity of titanium severely limited applications and produced processing challenges not encountered in other applications. Alternative forging procedures, machining parameters, heat treatments, welding techniques and cleaning processes had to be developed. Accommodations had to be made to deal with room temperature creep. Now, after more than thirty years of usage, titanium alloys remain the cost-effective material for high-performance turbopump applications. This talk deals with the challenges that were encountered in the application of titanium alloys to rocket engine components and how the various challenges were overcome.

**2:40 PM**
**Phase Transformations in Ti-6Al-4V-xH Alloys**: *F. H. (Sam) Froes*[1]; Javaid I. Qazi[1]; Jawad Rahim[1]; Oleg N. Senkov[2]; Sunil Patankar[1]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Ti-6Al-4V alloy specimens containing 0, 10, 20 and 30 at.% hydrogen were prepared. Microstructures, phases and phase transformations were then determined in the temperature range of 20 to 1000°C using optical microscopy, transmission electron microscopy, X-ray diffraction and microhardness measurements. Alloying with hydrogen was achieved by exposure of specimens to different hydrogen partial pressures at 780°C. Increasing the hydrogen content from 0 to 30 at.% lowered the beta transus temperature of the alloy from 1005°C to 815°C, significantly slowed down the kinetics of beta-to-alpha transformation and led to formation of orthorhombic martensite instead of hexagonal martensite in quenched specimens. A hydride phase was also detected in the specimens containing 20 to 30 at.% hydrogen. The martensite decomposition behavior and beta phase time-temperature-transformation (TTT) diagrams were determined for different hydrogen concentrations.

**3:05 PM**
**Effects of Working and Heat Treatment on Microstructural Evolution and Crystallographic Texture of Alpha and Alpha" Phases in Ti-6Al-4V**: *Liang Zeng*[1]; *Thomas R. Bieler*[1]; [1]Michigan State University, Dept. Cheml. Eng. & Matls. Sci., EB 2527, E. Lansing, MI 48824-1226 USA

Crystallographic texture has been measured on titanium fasteners at several stages of production. As-received wire had a moderately strong fiber texture with prism plane normals aligned with the wire axis. The wire was worked by extrusion, solution heat-treated and water quenched, then aged prior to thread rolling. Extrusion strengthened the as-received texture. After solutionization and quenching, a secondary fiber with basal plane normals aligned with the wire axis emerged at the expense of the initial texture. This 90° rotation is similar to rotations observed in a rolled plate subjected to a super-b transus anneal. The a" texture was stronger and much less symmetric than the a texture at all stages of the process. The relationship between the orientations of a, a", and the processing history are discussed in the context of the known physical metallurgy of titanium alloys.

TUESDAY PM

**3:30 PM Break**

**3:45 PM**
**The Formation of Nanocrystalline Structure in the Thread Root of Ti-6Al-4V Fasteners**: *Liang Zeng*[1]; *Thomas R. Bieler*[1]; [1]Michigan State University, Dept. Chem. Eng. & Matl. Sci., EB 2527, E. Lansing, MI 48824-1226 USA

TEM, XRD and OIM were used to investigate the microstructure of Ti-6Al-4V fasteners. The fasteners were solutionized, quenched, aged, followed by grinding and thread rolling operations to form the fastener. XRD texture measurements experiments showed that the thread rolling process intensifies the preferred orientations present before thread rolling, i.e. [10-10] and [0001] directions parallel to the wire axis. OIM analysis showed that the apparent grain size of undeformed material in backscattered electron images was smaller than regions with nearly the same orientations. Strains in the thread root are large, possibly near 2.0, based upon shape changes of grains. TEM analysis showed nanocrystalline structure in some regions of the thread root with a grain size around 50nm. Diffraction patterns show rings indicating that the prior alpha/beta orientations were broken down to form nearly randomly orientated alpha/beta orientations. The significance of nanostructured material on properties in thread root regions is discussed.

**4:10 PM**
**Processing, Microstructure and Tensile Properties of Ti-17 Bar**: *Brian Marquardt*[1]; J. R. Wood[1]; [1]Allvac, An Allegheny Technologies Company, R&D, 2020 Ashcraft Ave., Monroe, NC 28110 USA

The beta rich Allvac® Ti-5Al-2Sn-2Zr-4Cr-4Mo Alloy, also known as Ti-17, is a high strength, deep hardening alloy that is often used for large aircraft engine components such as compressor discs. For certain aerospace fasteners and potential automotive applications, the tensile strength and/or shear strength of standard grade Ti-64 is inadequate. Therefore, recent work at Allvac has focused on the evaluation of Ti-17 in bar product form. An ingot was first produced by plasma cold hearth melting (PAM) and vacuum arc remelting (VAR) to a 30 inch (760mm) diameter. The ingot was pressed and then rotary forged (GFM) to a 5 inch (130mm) diameter. Separate billets were then rolled on a continuous rolling mill at temperatures above and below the beta transus. Heat treatment studies were conducted on the bar products and selected microstructural conditions were tested for tensile and shear properties. Microstructural evolution will be presented along with microstructure property relationships.

**4:35 PM**
**A Combinatorial Approach to Developing Property/Microstructure Relationships in Titanium Alloys Using Laser Deposition**: *R. Banerjee*[1]; P. C. Collins[1]; S. Banerjee[2]; H. L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210 USA; [2]Bhaba Atomic Research Center, Matls. Grp., Trombay, Mumbai, India

Compositionally graded binary Ti-xV and Ti-xMo and ternary Ti-6Al-xV alloys have been deposited using the laser engineered net-shaping (LENS™) process. A compositional gradient, from elemental Ti to Ti-25at%V or Ti-25at%Mo, has been achieved within a length of ~25 mm in case of the binary alloys, while in the case of the ternary system the gradient lies from Ti-6Al to Ti-6Al-25V. The feedstock used for depositing the graded alloy consists of elemental Ti, Al, and V (or Mo) powders. Though the microstructural features across the graded alloy correspond to those typically observed in alpha/beta Ti alloys, the scale of the features is substantially refined in a number of cases. Using structural characterization techniques such as SEM and TEM coupled with microhardness measurements across the graded alloys, the influence of variations in the volume fractions, distribution and morphology of alpha and beta phases on the hardness has been studied. Tensile properties will be determined using sub-scale coupons. In addition, several phase transformations are encountered in the graded alloy samples, both during LENS™ deposition and during subsequent heat treatments. The ability to achieve such substantial changes in composition across rather limited lengths makes this process a highly attractive candidate for combinatorial materials science studies. This research has been supported in part by the US AFOSR, with Dr. Craig Hartley as Program Manager.

**5:00 PM**
**The Generation of a Database Relating Microstructure and Tensile Properties in Ti-6Al-4V Using a Resistive Heating System and Advanced Image Process Protocols**: *J. Tiley*[1]; T. Searles[2]; Hamish L. Fraser[2]; [1]Air Force Research Laboratory, AFRL/MLLMD, WPAFB, OH 45433-7817 USA; [2]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210 USA

There is a need to generate sophisticated databases that relate microstructure and properties in advanced alpha/beta Ti alloys. Such databases are required to permit the development of predictive capabilities regarding property/microstructure relationships through data-mining and rules-based approaches. A major problem in developing such data involves the need to accurately and reproducibly control the heat-treatment, particularly the cooling rate from a given heat-treatment temperature. For this purpose, a thermo-mechanical simulator, a Gleeble™ 1500 system, has been used which uses electrical resistance as the heat source and which can provide for very accurate and sensitive thermal excursions. Thus, a variety of heat treatment conditions have been used to develop a wide range of microstructural variations in Ti-6Al-4V. These process conditions have included the steps traditionally used by industry to develop specific properties and included variations in heating rates above the beta transus, hold times, hold temperatures, cooling rates, and duplex treatment conditions. Also included has been an analysis of Gleeble™ system parameters including width between cooling jaws, sample geometry, and cooling water temperatures. The temperature variations along the sample were measured and compared to develop profiles for process conditions. The sample microstructures were quantitatively evaluated to determine grain size, beta and alpha concentrations, alpha-lath thickness, primary and grain boundary alpha thickness, microstructural tortuosity, colony size factors, and other parameters using advanced stereological techniques.

## Modeling the Performance of Engineering Structural Materials: High Strain Rate and Impact Response

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Tuesday PM    Room: 215
October 8, 2002    Location: Columbus Convention Center

*Session Chairs:* T. Srivatsan, University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

**2:00 PM**
**High Strain Rate Deformation of Gamma-Met PX at Elevated Temperatures**: *Mostafa Shazly*[1]; Vikas Prakash[1]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., 10900 Euclid Ave., Cleveland, OH 44106-7222 USA

Gamma titanium aluminides has received a considerable attention over the last decade. These alloys are known to have low density, good high temperature strength retention and good oxidation and corrosion resistance. However, poor ductility and low fracture toughness were limiting factors in full utilization of these alloys. Poor ductility also was a road block in the manufacturing of these alloys in sheet form. Gamma-met PX has been developed to overcome this road block. An experimental and numerical research project at Case Western Reserve University has been established to investigate the initiation and spread of damage under impact loading conditions for this alloy. As a step towards this goal, this article explores the high strain rate compression properties of Gamma-met PX at room and elevated temperatures using SHPB.

**2:25 PM**
**High Strain-Rate Behavior of Glassy Polymers**: *Vikas Prakash*[1]; Namit Mehta[1]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., 10900 Euclid Ave., Cleveland, OH 44106-7222 USA

In order to better understand the dynamic response of glassy polymers compression tests are conducted on amorphous polycarbonates at strain-rates ranging from 0.001/s to 10^6/s. The quasi-static response of the polycarbonate is obtained by using a servo-hydraulic test machine. In the intermediate strain-rate regime (~1000/s to 3000/s), adiabatic and isothermal compression tests are conducted using the SPHB. Moreover, high-strain-rate pressure-shear plate-impact experiments are conducted to obtain the material behavior in the strain-rate regime of 105-106/s. Also, by using a suitable choice for the thickness of the target plates, a step drop in normal pressure is induced. The pressure drop experiments allows us to investigate the evolution of flow stress to changes in normal pressure. By combining the results from the three series of tests, pressure, temperature and strain-rate dependence of flow stress of polycarbonate is determined.

**2:50 PM**
**Attenuation and Dispersion of Shock Waves in Layered Material Systems**: *Liren Tsai*[1]; Vikas Prakash[1]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., 10900 Euclid Ave., Cleveland, OH 44106-7222 USA

The utilization of layered heterogeneous material systems in the development of armor provides a potential for a quantum leap in ballistic performance in a variety of lightweight armor applications. In an attempt to better understand the behavior of heterogeneous material systems under shock-wave loading conditions, an integrated analytical/computational/experimental research program is underway at CWRU. In order to understand the effects of layer thickness and impedance mismatch, plate impact experiments on 2-D layered heterogeneous material systems are conducted on several different target thicknesses of elastic-elastic bilaminates. Moreover, in order to obtain the effects of material inelasticity on both wave-front and late-time dispersion plate-impact experiments are conducted on different thicknesses of viscoelastic-elastic bilaminates. The experimental results are compared with analytical predictions for both wave-front and late-time solutions for step-pulse loading on layered half-space.

**3:15 PM**
**Modeling Blast Resistant Protection Systems Composed of Polymers and Fabric**: *John E. Crawford*[1]; [1]Karagozian & Case, 625 N. Maryland Ave., Glendale, CA 91206-2245 USA

A variety of devices have been designed and tested for protecting building occupants from the effects of a terrorist bombing. One of the common features of many of these devices is their reliance on unusual materials, such as Kevlar and Carbon fabric, polyurethane forms and sprays, and a variety of adhesives, as major components and to provide key features needed to resist blast effects. The paper describes some of the designs, test results, and finite element models related to the development of such devices.

**3:40 PM**
**Impact Damage in Brittle Materials at Elevated Temperatures**: *David Nathenson*[1]; Vikas Prakash[2]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., Cleveland, OH 44106-7222 USA; [2]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., Cleveland, OH 44106-7222 USA

The problem of categorizing impact damage in brittle materials at elevated temperatures is of concern to the aircraft engine industry in their quest for lighter and stronger materials. To this end, a study is being conducted at CWRU into the behavior of soda lime glass as a prelude to high temperature investigations of other brittle materials. The current study on soda lime glass comprises impact of various thickness glass panes at velocities around 500m/s. Impacts are carried out by accelerating small diameter steel particles with compressed air. A high speed camera and a VISAR interferometer are used to determine particle velocity and cracking patterns. Projectile velocity, impact angle, and specimen thickness are varied. The purpose of this study is to gain a comprehensive picture of cracking patterns in sodalime glass before embarking on the study of the opaque silicon nitride.

## Pb-Free and Pb-Bearing Solders - III

*Sponsored by:* Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA

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*Session Chairs:* Eric J. Cotts, Binghamton University-SUNY, Physics & Matls. Sci., Binghamton, NY 13902-6016 USA; Iver Eric Anderson, Ames Laboratory, Metal & Ceram. Scis., Ames, IA 50011 USA

**2:00 PM Invited**
**Microstructure and Property Response of Sn-Ag-Cu Solder Joints to Accelerated Aging**: *Iver Eric Anderson*[1]; Bruce A. Cook[1]; Joel Harringa[1]; Robert L. Terpstra[1]; [1]Ames Laboratory, Metal & Ceram. Scis., 222 Metals Dvlp., Ames, IA 50011 USA

Further investigations of a Pb-free family of near-eutectic Sn-Ag-Cu and Sn-Ag-Cu-X alloys that were patented for electronic solder applications required the study of accelerated aging effects, since many new electronic system applications involve service at high temperature and stress levels. Near eutectic Sn-Ag-Cu and Sn-Ag-Cu-X (X = Co, Fe) alloys have been the focus of this effort to gather joint reliability data terms of solder joint microstructure, resistivity, and mechanical properties. Differences in accelerated aging response will be reported in solder joints due to microstructure coarsening effects that occur at 150°C, a maximum under-hood temperature in automobiles, after exposure time intervals out to 1,000 hrs. Project funding received from Iowa State University Research Foundation with additional support from USDOE-BES, contract no. W-7405-Eng-82.

**2:30 PM**
**Dynamic Thermal/Mechanical Characterization of Lead-Free Solder Joints**: *H. Rhee*[1]; K. N. Subramanian[1]; A. Y. Lee[1]; T. R. Bieler[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Dynamic thermal/mechanical analysis (DTMA) was carried out at various temperatures to investigate the behavior of eutectic Sn-3.5Ag solder joints under simulated service conditions using Solids Analyzer (RSA-III). Various performance parameters were evaluated with this equipment by subjecting geometrically realistic eutectic Sn-3.5Ag solder joints to temperature variations and mechanical working. Solder joints used in this study had a nominal joint thickness of ~100μm with 1mm x mm solder joint area. After the DTMA simulation, the microstructures of the solder joints were also characterized using SEM. Dynamic thermal/mechanical properties such as ultimate shear stress as a function of simple shear rate, shear stress-simple shear relationships, and stress-relaxation behavior at various temperatures and imposed strains were evaluated to gain a better understanding of parameters contributing to thermomechanical fatigue. Acknowledgement: Work supported by the National Science Foundation under grant NSF DMR-0081796.

**2:55 PM**
**Effects of Anisotropy of Tin on the Thermomechanical Behavior of Solder Joints**: J. G. Lee[1]; A. Telang[1]; H. Jiang[1]; *K. N. Subramanian*[1]; T. R. Bieler[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Properties of body centered tetragonal Sn are extremely anisotropic. As a consequence, large stresses can develop at the Sn grain boundaries due to CTE mismatch between the adjacent grains during temperature excursions. The stress component normal to the grain boundary, as well as the shear stress in the plane of the grain boundary, that develop during thermal excursions were computed on the basis of elastic deformation conditions, for several relevant crystallographic orientations between the adjacent grains. Stresses normal to the boundary can cause grain boundary decohesion, while shear stresses in the boundary can cause grain boundary sliding and surface relief effects. Surface damage in thermomechanically fatigued Sn-based solder joints will be used to illustrate these phenomena. Acknowledgement: Project funded by National Science Foundation under grant No. NSF DMR 0081796.

**3:20 PM Break**

**3:40 PM Invited**
**Some Aspects of the Evolution of the Microstructure of Pb Free Solder Joints**: *Eric J. Cotts*[1]; L. P. Lehman[1]; Anis Zribi[1]; L. Zavalij[1]; R. Kinyanjui[1]; [1]Binghamton University-SUNY, Physics & Matls. Sci., PO Box 6016, Binghamton, NY 13902-6016 USA

Changes in the metallurgy of solder joints has led to new challenges in understanding the evolution of their microstructure. The constituents (e.g. Cu, Ag) added to Sn to lower its melting point and increase its wettability as a Pb free solder, precipitate as intermetallics in the solder matrix, and may form compounds at interfaces as well. Thus the complexity of the problem of solder joint reliability has increased, and so has its importance, as these precipitates can significantly affect the mechanical properties of solder joints. The formation of intermetallic compounds at solder/metallization interfaces, and the evolution of precipitates in the solder matrix must be understood if reliable constitutive relations are to be developed for these materials. To this end we have examined precipitate formation and evolution in Pb free solder matrices for different reflow profiles on Ni or Cu metallizations. SnAgBi and SnAgCu solder joints were examined.

**4:10 PM Invited**
**Bulk Specimen and Solder Joint Mechanical Properties of Sn/Ag/Cu Solder Alloy**: *John Hock Lye Pang*[1]; [1]Nanyang Technological University, Sch. of Mechl. & Prod. Eng., Nanyang Ave., 639798 Singapore

Due to legislation to ban Lead (Pb) in electronic solders, lead-free solder alloys are needed to replace Sn/Pb solders. The NEMI program on lead-free solders has recommended Sn/Ag/Cu alloy as a prospective replacement for Sn/Pb alloy. In addition to the solderbility and process issues for Sn/Ag/Cu solder alloy, the mechanical properties for tensile, shear, creep and fatigue properties are needed. This invited paper will report on bulk specimen and solder joint shear stress strain properties for 95.5Sn/3.8Ag/0.7Cu solder alloy. Bulk specimens were cast from Sn/Ag/Cu solder bars while the solder joint specimen were actual single BGA solder ball of size 0.5mm soldered on to a lap shear FR4 substrate specimen. The shear stress versus strain properties were tested at three temperatures namely, 25ºC, 100ºC and 150ºC and at two displacement rates of 0.05mm/min and 5mm/min respectively. The shear stress strain curves were used to compute the elastic-plastic properties with respect to temperature and strain-rate.

**4:40 PM Invited**

**Microstructure Evolution in Pb-Free Solders**: *Nik Chawla*[1]; [1]Arizona State University, Mechl. Behavior of Matls. Fac., Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

The mechanical and physical properties of Pb-free solders are directly related to their microstructure. Thus, the characterization and understanding of changes in solder microstructure induced by processing or thermal aging, whether in monolithic or joint form, is extremely important. This talk will attempt to present an overview of microstructure evolution in Pb-free solders and solder joints. In particular, the effect of cooling rate on the morphology and evolution of intermetallics at the solder joint/Cu substrate interface will be discussed. Finally, the implications of microstructure evolution on mechanical behavior of solders and solder joints under shear and creep conditions will be presented. Research supported by the National Science Foundation (Program Manager: Dr. K.L. Murty, contract# DMR-0092530).

**5:10 PM**

**Effect of Microstructure Evolution on Local Mechanical Properties in Pb-Free Solder Joints**: *H. Rhee*[1]; A. U. Telang[1]; J. P. Lucas[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Microstructure evolution during service has significant implications on deformation behaviors such as creep, stress relaxation, and fracture of solder joints. Knowledge of the properties and deformation behavior due to the evolved microstructure will assist development of reliability prediction models. Using nanoindentation testing (NIT) of creep, modulus and hardness, deformation mechanisms can be obtained in highly localized regions in the solder joint. NIT was performed on un-aged and isothermally-aged Sn-Ag based lead free solder joints. The evolved microstructure regions of the joints examined with NIT was characterized using optical, SEM and orientation imaging microscopy (OIM), and correlated with mechanical properties of as-fabricated and aged joints. Determining the deformation behavior and properties of local evolved microstructure can provide a better understanding of the overall deformation processes in solder joints subjected to thermomechanical loading. This research was supported by the National Science Foundation under grant NSF-DMR-0081796.

## Rapid Prototyping of Materials: Rapid Prototyping of Metals

*Sponsored by:* Materials Processing & Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector; MPMD-Powder Materials Committee, SMD-Composite Materials Committee-(Jt. ASM-MSCTS)

*Program Organizers:* Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3901 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Meisha L. Shofner, Rice University, MEMS, Houston, TX 77005 USA

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| Tuesday PM | Room: 212 |
| October 8, 2002 | Location: Columbus Convention Center |

*Session Chair:* Meisha L. Shofner, Rice University, Mechl. Eng. & Matls. Sci., Houston, TX 77251-1892 USA

**2:00 PM Invited**

**Metallurgical Analysis of Laser Formed Metal Parts**: *Khershed P. Cooper*[1]; Frank G. Arcella[2]; Harry N. Jones[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech., Code 6325, 4555 Overlook Ave. SW, Washington, DC 20375 USA; [2]AeroMet Corporation, 7623 Anagram Dr., Eden Prairie, MN 55344 USA

Rapid Prototyping has achieved a level of maturity which places it at the threshold of being implemented as a manufacturing tool. For metals, laser forming appears to be a promising approach for building fully dense parts. AeroMet's Laser Additive Manufacturing (LAM) process is designed to fabricate near net-shape parts. The objective is not precision, but high throughput and minimum machining. LAM is ideally suited for the fabrication of refractory metals such as rhenium. Our goal was to perform systematic metallurgical analysis of laser-formed rhenium to further validate this enabling technology. Metallurgical analyses included determination of grain size and, if present, porosity. We also determined oxygen pick-up, if any, and microhardness maps. Grain size was large due to the large melt pool and ensuing slow solidification kinetics. Very few pores were detected, their size not exceeding 2μm. Bulk analysis showed oxygen below the detection limit of 0.001%. A few implications of these results will be discussed.

**2:30 PM**

**Complex Titanium Alloys Deposited from Elemental Powder Blends by Laser Engineered Net Shaping (LENS™)**: *David J. Bryan*[1]; *Peter C. Collins*[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The directed laser deposition process known as LENS™ (laser engineered net shaping) has many desirable characteristics, including the capability of creating a near-net shaped part from a computer generated CAD file while affording the ability to achieve a composition gradient within the part. Traditionally, pre-alloyed powders have been used in the LENS™ process. However, the use of elemental blends as a feedstock allows for a substantial decrease in the cost of producing components as well as flexibility and ease in varying compositions to achieve desired properties. Initial studies on the deposition of simple binary Ti-alloys, such as Ti-Cr and Ti-Nb, suggested that an important factor influencing the ability to produce a homogeneous in situ alloy using elemental powders is the thermodynamic enthalpy of mixing of the constituent powders. Subsequently, the laser deposition of more complex b-titanium alloys, such as the non-burning alloy developed at the IRC in Birmingham (Ti-25V-15Cr-2Al-0.2C) and Timet-21S (Ti-15Mo-3Al-2.7Nb-0.2Si), from elemental blends has been investigated and the results will be discussed in this paper. In addition, a comparison of the microstructure and mechanical properties of Timet-21S LENS™ deposits made from both elemental blends as well as pre-alloyed powders will be presented.

**3:00 PM**

**Direct Laser Fabrication of Ti-Based Alloys**: Junfa Mei[1]; Jing Liang[1]; Rob Sharman[1]; Wayne Voice[2]; *Xinhua Wu*[1]; [1]University of Birmingham, IRC in Matls., Edgbaston, Birmingham B15 2TT UK; [2]Rolls-Royce plc, PO Box 31, Derby, UK

Direct laser fabrication has been used to produce samples from Ti64 and from a burn-resistant Ti alloy (Ti-25V-15Cr-2Al-0.2C). Experiments have been carried out in which the influence of process variables on the microstructures observed has been investigated. Ti64 tends to form long columnar grains throughout the entire manufacture of parts whereas the burn-resistant alloy tends to form equiaxed grains for virtually all processing conditions. In the burn-resistant alloy a virtually fully dense sample is formed for a wide range of conditions, but in Ti64 it is possible to produce a highly porous structure under some conditions. It has been found that there is no simple relationship between the influence of the different process variables on the structures which are formed, but work is underway and will be reported, in which it is hoped that the influence of a change in processing conditions can be predicted.

**3:30 PM Break**

**3:50 PM**

**Prediction of Microstructure in Laser-Deposited Titanium Alloys**: *Nathan W. Klingbeil*[1]; Chad J. Brown[1]; Srikanth Bontha[1]; Pamela A. Kobryn[2]; Hamish L. Fraser[3]; [1]Wright State University, Mechl. & Matls. Eng., 209 Russ Eng. Ctr., 3640 Col. Glenn Hwy., Dayton, OH 45435 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMP, Bldg. 655, Ste. 1, WPAFB, OH 45433-7817 USA; [3]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Laser deposition of titanium alloys is currently under consideration for application to aerospace components, and offers significant increases in efficiency and flexibility compared to conventional manufacturing methods. However, the successful transition of this promising technology will ultimately depend on the ability to predict and control the microstructure and resulting mechanical properties of the deposit. In this study, continuum finite element modeling of the laser deposition process is used to predict thermal gradients and cooling rates (the key parameters controlling microstructure) as a function of deposition process parameters. In addition, 3-D cellular automata finite element modeling is used to investigate the stochastic grain nucleation and growth process during bulk solidification, and the results are interpreted in the context of the continuum finite element modeling. Finally, the computational results are compared with both thermal measurements and observed microstructures in LENS™ deposited titanium alloys.

**4:20 PM**

**Functionally Graded Copper-Steel Using LENS™ Process**: *Fredrick F. Noecker*[1]; John N. DuPont[1]; [1]Lehigh University, Dept. of Matl. Sci. & Eng., 5 E. Packer Ave., Whitaker Lab., Bethlehem, PA 18015 USA

Laser Engineered Net Shaping™ (LENS™) is an emerging Solid Freeform Fabrication (SFF) process capable of producing fully dense metallic parts with complex shapes directly from a computer-aided drawing (CAD) without the need for molding or tooling. Conventional manufacturing processes are limited in their ability to minimize segregation, nor are they capable of locally altering the composition and resultant microstructure at different locations within the part in order to tailor the properties for enhanced performance. The LENS™ process also shows promise in producing components with reduced or eliminated microsegregation, refined microstructures and graded compositions. One potential application is the production of steel-Copper die casting materials. Copper is currently deposited on dies made out of steel to enhance thermal management. The deposition of Copper onto steel is problematic due to difficulties that can occur at the Fe-Cu interface, such as, a large solidification temperature range in the Fe-Cu system (which can cause solidification cracking), formation of undesirable phases, and differences in coefficient of thermal expansion. The current research goal is to develop LENS™ processing parameters to optimize the deposition of functionally graded steel-Copper alloy. Steel-Cu functionally graded samples were made under various processing conditions to produce a variety of concentration gradients. Additionally, the use of a Ni bi layer, functionally graded into steel then graded to pure Cu, was explored as a means of eliminating cracking in the steel-Cu build functional grade. The resulting microstructures were characterized by various microscopy techniques. The influence of processing conditions and interfacial composition on the resultant microstructure will be discussed. This information is useful for successful deposition of crack-free copper layers onto steel for die casting applications.

**4:50 PM**
**Elevated Temperature Characterization of Direct Laser Deposited Ti-8Al-1Er**: *Ralph W. Bush*[1]; Craig A. Brice[2]; Jay P. Baer[1]; E. Tim Skaar[1]; [1]US Air Force Academy, Dept. of Eng. Mech., 2354 Fairchild Dr., Ste. 6L-155, USAF Academy, CO 80840 USA; [2]Lockheed Martin Aeronautics Company, Fort Worth, TX, USA

Direct laser deposited Ti-8Al-1Er combines the technology of the Laser Engineered Net Shaping (LENS™) process with in-situ alloying of an $\alpha$ titanium alloy. The result is a low cost, precipitation strengthened titanium alloy. The room and elevated temperature tensile and creep properties of direct laser deposited Ti-8Al-1Er were measured and compared to those of Ti-6Al-4V and Ti-6242. Room temperature strengths of Ti-8Al-1Er are comparable to those of Ti-6Al-4V and Ti-6242. Elevated temperature strengths of Ti-8Al-1Er are superior to those of Ti-6Al-4V and comparable to Ti-6242. Larson-Miller analysis of the creep results indicate that the creep resistance of Ti-8Al-1Er is superior to that of Ti-6Al-4V, but inferior to that of Ti-6242. The primary concern with the new product is its low ductility at room temperature. This work indicates that direct laser deposited in-situ alloyed titanium products may hold promise for elevated temperature applications.

## Assessment of Materials Engineering Programs-EC 2000

*Sponsored by:* TMS-Education Committee
*Program Organizers:* Mark A. Palmer, Kettering University, Manufacturing Engineering, Flint, MI 48504-4898 USA; Dennis Readey, Colorado School of Mines, Metallurgical and Materials Engineering, Golden, CO 80401 USA

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| Wednesday AM | Room: 213 |
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*Session Chairs:* TBA

**8:30 AM**
**Course Level Assessment–Keeping it Simple and Comprehensive**: *Mark A. Palmer*[1]; William J. Riffe[1]; [1]Kettering University, IMEB, 1700 W. Third Ave., Flint, MI 48504 USA

ABET EC 2000 has required engineering schools to assess the effectiveness of instruction. Ultimately, it is at the course level where the most significant impacts occur; therefore, the effective assessment of courses is necessary. Driven by this need, a very complicated methodology, originally developed to meet NSF funding requirements (under a previous contract, has been simplified in preparation for an upcoming ABET visit. Data is collected using three or more independent measures, at course completion, during follow-on courses and using post graduation surveys. Using this system, the effectiveness of course objectives as measured by course outcomes can be performed easily and presented to a reviewer in a concise method.

## Characterization and Representation of Material Microstructures in 3-D: Visualization and Representation

*Sponsored by:* Structural Materials Division,
*Program Organizers:* Mike Uchic, Air Force Research Laboratory, Dayton, OH 45433-7817 USA; Dennis M. Dimiduk, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Milo V. Kral, University of Canterbury, Department of Mechanical Engineering, Christchurch, New Zealand; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375 USA

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| October 9, 2002 | Location: Columbus Convention Center |

*Session Chairs:* George Spanos, Naval Research Laboratory, Physl. Metall. Branch, Washington, DC 20375 USA; Robert O. Rosenberg, Naval Research Laboratory, Ctr. for Computatl. Sci., Washington, DC 20375 USA

**8:30 AM Keynote**
**Visualization and Representation**: *Gerald A. Higgins*[1]; [1]Digital Human Project at the Federation of American Scientists, Dir., Washington, DC, USA

Research in biomedical modeling and simulation has reached a critical juncture where integration using diverse data (genomic, protein, cellular, tissue, organs, systems) derived from various biomedical domains can significantly enhance our understanding of complex phenomenon in normal human function and disease states. For these efforts to succeed, researchers need to develop common technical resources for sharing model and simulation data in a distributed fashion. The diverse array of simulations, models and images used in experimental biology and clinical medicine need to be documented, and resources need to be made available to provide investigators with information about what currently exists, and where these are housed. Next, more formal instruments need to be developed for model sharing and integration. It may be appropriate to develop a common modeling language, and construct mechanisms that support collaboration and model sharing while maintaining the intellectual property rights of individual developers. The long term vision of the Digital Human project is the development of accurate and validated models and simulations of cells, tissues, organs and organ systems that can serve as frameworks for experimental analysis, patient care, and training in biology and medicine. Computational models of physiology need to be integrated into existing models of anatomy to produce a breathing and bleeding model of the human. Different levels of hierarchy need to be integrated, including data from tissue biomechanics, systems physiology, gross anatomy, cell biology, the proteome and the genome. Current efforts are being conducted on a variety of fronts, including the development of a unified ontology for multi-level biomedical modeling and simulation, creation of a standardized geometrical framework, and applications in a variety of domains including systems biology research and medical simulation training.

**9:10 AM Invited**
**Scientific Visualization of Volumetric Data**: *Robert O. Rosenberg*[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA

With the advent of Supercomputing in the mid 1970's, issues arose concerning the analysis and interpretation of the resulting data, given that so much data could be generated in so little time. The solution was the development of Scientific Visualization–the capability of using computer graphics and high-end computer graphics workstations to explore and bring out key features of the data. Today, two fields, biomedicine and materials science place a strong demand on Scientific Visualization, not from any computational results, but from 3D reconstructions based on 2D images obtained from CT, MRI, Optical and Scanning Electron Microscopy to generate volumetric data. The Visible Human Project challenged Scientific Visualization experts to render the 30GB Visible Female in real-time at 30 frames per second. We will present an overview of some of the latest techniques and trends, in software and hardware, used to visualize this type of volumetric data.

**9:40 AM Invited**
**Recovering Three-Dimensional Structure from Serial Sections of Brain Tissue**: *John C. Fiala*[1]; [1]Boston University, Dept. of Biology, 5 Cummington St., Boston, MA 02215 USA

The brain has an enormously complex three-dimensional (3D) structure. Billions of highly-branched cells communicate pairwise at specialized junctions of submicron scale. Serial sectioning of epoxy-embedded brain tissue at 50 nm and imaging with transmission electron microscopy introduces misalignments and distortions that make the problem of recovering 3D structure difficult. Alignment of whole images can be realized on the computer using nonlinear transformations calculated from a set of correspondence points. The series of realigned images forms a large reconstructed volume with the fine details preserved. This representation greatly aids the analysis of 3D structure. In reconstructed volumes it is easier to trace small, branched structures by observing the apparent motion of profiles through the sections. It is also easier to make accurate stereological measurements. Free software for carrying out this reconstruction and analysis is available at www.synapses.bu.edu/tools/.

**10:10 AM**
**MicroConstructor–An Alternative to Serial Sectioning?**: Mark A. Miodownik[1]; David Basanta[1]; Peter Bentley[2]; *Elizabeth A. Holm*[3]; [1]King's College London, Mechl. Eng. Dept., Strand, London WC2R 2LS UK; [2]University College London, Compu. Sci. Dept., London, UK; [3]Sandia National Laboratories, Matls. Modlg. Dept., Albuquerque, NM 87185-1411 USA

At a recent international Materials modelling conference there was general consensus that there was a need to incorporate experimental microstructures as starting configurations for computer models of various kinds. In two dimensions (2D) this is relatively straight forward since microstructures can be imaged in 2D at various length scales by a number of well-know techniques. But in three dimensions (3D) it is very problematic because getting real 3D information is experimentally very challenging. In this paper we will describe an general approach to solving this problem of simulation start structures. We will describe a program called MicroConstructor, that will take any two dimensional (2D) scanning electron microscope image (SEM) micrograph and generate a three dimensional (3D) discrete computer microstructure which is statistically equivalent in terms of the microstructural variables of interest. The basis of the code is a genetic algorithm that evolves the 3D microstructure so that its cross-sections match the 2D data.

**10:35 AM Invited**
**Gauging Importance of Dislocation Interactions Through Direct Comparisons of Experimental Observations and Computational Predictions**: *S. Swaminarayan*[1]; R. McCabe[1]; Z. Q. Wang[2]; R. LeSar[1]; N. Ghoniem[2]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [2]University of California at Los Angeles, Los Angeles, CA, USA

Unit dislocation processes form the basis of plastic deformation. These interactions are inherently 3D in nature. In order to study these interactions computationally we have developed a 3D dislocation dynamics code with curved dislocation segments and an implicit time integration method. This combination of curved segments and implicit integration allows us to run simulations with fewer segments and longer time steps. To validate this code we compare the results predicted by our code directly with those observed in the TEM using stereo microscopy. This comparison not only gives us a direct gauge of the accuracy of the simulations, but also insight into the relative importance of the several different interactions present in the simulation.

**11:05 AM Break**

**11:25 AM**
**Towards Realistic Mesoscopic Models for Microstructural Evolution**: *Anthony D. Rollett*[1]; Melik Demirel[1]; Priya Manohar[1]; Bassem El-Dasher[1]; Joe Fridy[2]; David Saylor[3]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA; [3]NIST, 100 Bureau Dr., Gaithersburg, MD 20899-3460 USA

Annealing crystalline materials almost invariably leads to changes in their microstructure. Reduction in interfacial area drives grain growth via the curvature on boundaries imposed by local equilibrium at triple lines. In addition, if stored energy is present from prior plastic deformation, recovery and recrystallization can occur. Influencing these processes is a marked anisotropy of grain boundary properties in both energy and mobility in all materials. Maps of these key properties are now available: in the case of MgO, a complete map of grain boundary energy as a function of the five macroscopic degrees of freedom has been determined. These properties can now be readily incorporated into mesoscopic models such as Monte Carlo (Potts), phase field, vertex, finite element (FEM) and other models. Detailed comparisons of experimentally determined and simulated grain growth in columnar (2D) structures in Al foil will be described that verify curvature-driven coarsening. Reconstruction will be shown of statistically accurate 3D microstructures based on electron back scatter diffraction scans on orthogonal planes in aluminum. These form the basis for the initial structures in 3D modeling of grain growth and recrystallization.

**11:50 AM**
**Reconstruction of 3D Microstructure from Orthogonal Sections**: *David M. Saylor*[1]; Joseph M. Fridy[2]; Hasso Weiland[2]; Anthony D. Rollett[3]; [1]National Institute of Standards and Technology, Ceram./Matls., 100 Bureau Dr., Stop 8521, Gaithersburg, MD 20899-8521 USA; [2]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA; [3]Carnegie Mellon University, Matls. Sci. & Eng. Dept., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

The procedures for constructing statistically representative, three dimensional microstructures based on plane sections are described. By using EBSD (OIM) data from orthogonal section planes, information can be obtained on both grain shape and microtexture. Through an appropriate tessellation of space with Voronoi cells, the grain shape can be captured. This structure contains both volumetric and interfacial information which is used in a simulated annealing procedure to fit both the measured texture and the misorientation distribution. The resulting microstructure is thus statistically representative of the original material. No serial sectioning is required.

**12:15 PM Invited**
**Representation of Alloy Microstructure with the Karhunen-Loeve Transform**: *Jeff P. Simmons*[1]; Dennis M. Dimiduk[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

Traditionally, microstructural characterization is used in materials science to solve a number of problems such as aspects of processing, property estimates, or determination of mechanisms. Materials scientists make evaluations of microstructures, based on experience and intuition, materials models, or observations of a property. However, methods are needed to communicate to computers exactly what it is that a materials scientist sees when evaluating microstructure. Image processing techniques provide information through measurement and quantification of details that are previously understood to be important. However, these techniques are fundamentally lossy since information that is not appreciated at the time of analysis is discarded. For example, the current practice of characterizing microstructures by particle size distributions, has an inherent loss of all information about interparticle spacing and correlation–structural elements most important to deformation properties. Although different areas of a same sample may yield a collection of distinguishable photographs, the "microstructural information" may be the same in each image. So long as each image is representative, each one could be equally used by the materials scientist. The small differences in exact particle locations, quantities, or sizes are unimportant, provided that the materials scientist would judge that they represent the same microstructure. However, the small differences are exactly what a computer examining images would identify as unique aspects of structure. This work examines "microstructural information" as a mathematical representation that is suitable for an automated technique to recognize differing images of the same microstructure as being the same. Further, it attempts to maximize retention of "microstructural information" so that typical images could be reconstructed from the representation. The work demonstrates the successful "eigenface" approach (Karhunen-Loeve transform) previously used to represent human faces. However, this technique attempts to characterize the population and its variability, not specific individuals within.

## Forming and Shaping of Light Weight Automotive Structures: Applications - II

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

Wednesday AM — Room: 210
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA

**8:30 AM Invited**
**Automotive Applications of DP and TRIP Steels**: *Mahmoud Y. Demeri*[1]; [1]Ford Motor Company, Mfg. Sys., 2101 Village Rd., Ford Rsrch. Labs., MD 3135, Dearborn, MI 48121 USA

The steel industry is developing different grades of Advanced High Strength Steels (AHSS) as alternative materials for lightweighting applications and other vehicle cost and performance requirements. Two types of AHSS, namely, DP and TRIP steels offer significant formability, crash performance, weight reduction and cost neutrality benefits have been considered for many current and future automotive applications. Although the list of potential applications of these materials is increasing, fundamental issues related to understanding their basic deformation mechanisms, material consistency, manufacturability, supplier strategies, coatings, performance assessment and material qualification process must be addressed.

**9:00 AM**
**High Strength Steel Application for Body Weight Reduction of Super Duty Trucks**: *Everett You-Ming Kuo*[1]; [1]Ford Motor Company, Ford Rsrch. Lab., 2101 Village Rd., MD 3135, Dearborn, MI 48121 USA

High mileage NVH performance is one of the major concerns in vehicle design for long-term customer satisfaction. Traditionally, vehicles are designed to have good functional performance at 0 mileage (i.e, good first-impression vehicles). However, the performance tends to degrade as mileage accumulates due to fatigue, corrosion, loosening, wear and aging of different parts. Typical design strategies and corresponding CAE assessment methods in the Automotive Industry aim at achieving good vehicle 0-mileage NVH performance. But high mileage NVH performance is becoming more crucial from the viewpoint of long term customer satisfaction. In addressing this issue, Ford has developed high mileage NVH robustness methodologies (design strategies, performance metrics and corresponding CAE assessment methods) in the last five years. The present study presents an application of these methodologies for a design feasibility study concerning cab weight reduction of a super duty truck using high strength steel and adhesive bonding. The study indicates that downgaging and substitution of mild steel with high strength steel and application of adhesive bonding in selected areas of a cab structure can achieve 28 lbs weight reduction while maintaining the high mileage NVH performance of the basaeline vehicle.

**9:25 AM**
**Lightweight Flexible Body Architectures**: *Matthew J. Zaluzec*[1]; [1]Ford Motor Company, Mfg. & Vehicle Design Rsrch. Lab., USA

The economies of scale dictate the design, flexibility and styling of automotive body architectures. As consumers demand safer, lighter, and environmentally friendly vehicles, there needs to be a paradigm shift in materials and manufacturing technology for next generation vehicles. The application of aluminum, magnesium and composite materials requires not only up front product design, but also manufacturing ready technologies that can bring new materials technology to the forefront of implementation readiness. The Ford Research Laboratory targets technology migration at the niche and specialty products where the constraints of high volume do not pose a technical roadblock. This presentation will review development of advanced and hybrid body architectures used in the production on products ranging from the premier automotive group's Aston-Martin AMVO3 Vanquish to the niche specialty THINK electric vehicle currently in production.

**9:50 AM**
**Design and Manufacturing of Magnesium Components for Automotive Applications**: *Naiyi Li*[1]; Michael Maj[2]; Paul Dellock[2]; [1]Ford Motor Company, Mfg. Sys. Dept., Ford Rsrch. Lab., 2101 Village Rd., MD3135, Dearborn, MI 48124 USA; [2]Ford Motor Company, Vehicle Sys. Dept., 2001 Beech Daly Rd., Dearborn Heights, MI 48121 USA

Magnesium applications in automotive industry are increasing at average 15% annual growth rate in last decade due to energy and environmental concerns. This paper will address recent research and engineering of magnesium alloys in automotive industry by an integrated assessment of material, design and manufacturing. It will review and discuss the following, 1) magnesium alloys and material properties of interest to designers compared with other materials i.e., aluminum, polymers and steel, 2) various manufacturing processes for lightweight magnesium components, particularly using casting or forging processes, 3) examples of conversions from steel or aluminum to magnesium, from perspective of stiffness and NVH considerations. The immediate and future R&D activities necessary for implementation of magnesium in body, chassis, interior and powertrain will be depicted by means of example components and projects. To aid the automotive industry to achieve cost and weight objectives, the potential of using lightweight magnesium components continues to expand.

**10:15 AM Break**

**10:25 AM**
**Magnesium Door Inner Development**: *Gregory T. Bretz*[1]; Patrick Blanchard[1]; Saravanan Subramaniam[1]; [1]Ford Motor Company, Ford Rsrch. Lab., 20000 Rotunda, MD 3135, PO Box 2053, Dearborn, MI 48121 USA

Magnesium applications for components and assemblies have been increasing throughout the industry. Benefits of using magnesium include its light-weight characteristics, good strength to weight ratio, good fatigue strength, high damping capacity and excellent fabricated dimensional stability and production cycle rates. However, magnesium also has drawbacks; among them higher cost, poor ductility and porosity. With these thoughts in mind a recent study was conducted within Ford Motor Co. to examine potential magnesium applications. One such study involved a review of closure systems. In this case a front side door, for a mid sized mass produced vehicle. This paper will focus on design issues, requirements, material alloy choices, stiffness and machining and assembly issues leading up to the manufacture of the door. Some discussions on cost and weight saving will also be presented.

**10:50 AM**
**Application of Alumina-Chrome Carbide Composites in Automotive Engines**: *Maksim V. Kireitseu*[1]; Serge Yerakhavets[1]; [1]Institute of Machine Reliability (INDMASH), Dept. of Mech. & Trib., Lesnoe 19-62, Minsk 223052 Belarus

A new alumina-chromium carbide-based coating has been developed for practical application in automobiles. The coating is found to have good friction and wear properties over a wide temperature range. A pyrolitic deposition of CrC layer was used to strengthen alumina layer formed on aluminum parts of the engine by micro arc oxidizing technology. CrC layer has strengthened alumina layer by filing surface defects and pores. Friction and wear were determined using a pin on disk tribometer at 25-900°C temperatures in hydrogen, helium, and air. Pin materials included several metallic alloys and silicon carbide. It was revealed that appropriate additions of disulfide molybdenum, or diamond particles to the baseline carbide composition significantly reduced friction coefficients while preserving, and in some cases, even enhancing wear resistance. The results of this study demonstrate that the composite is a promising coating to be considered for high-temperature aerospace and advanced heat engine applications. The excellent results in hydrogen make this coating of particular interest for use in automotive engines. Based upon observed tribological mechanics, a model has been developed to describe thermo-mechanical behavior of the alumina-chrome carbide composite under friction.

**11:15 AM**
**Application of Hot Forging Process Simulation for Automotive Parts**: *Young Sang Ko*[1]; Hyounsoo Park[1]; Sung Do Wang[2]; Seung Cheal Jung[1]; [1]Hyundai Motor Company, Adv. Rsrch. Ctr., 772-1, Changduk-dong, Whasung-si, Kyunggi-do 445-850 Korea; [2]Kia Steel Company, Ltd., R&D Ctr., 1-6, Soryong-dong, Kunsan, Cheollabukdo 573-400 Korea

A newly developed 3D forging process simulation has been introduced to optimize the process control of automotive forging parts. We analyzed forgeability of steels at various operating parameters such as forge load, temperature and die shape. The simulation data are also correlated the experimental results in thermomechanical characteristics. Forging simulation, focused on automotive parts–cranks shaft, connecting rod, chassis parts, is carried out to maximize strength and toughness by process and alloy optimization. The correlation of manufacturing variables and microstructures of forged samples has been evaluated by using thermo-mechanical simulator. The FEM simulation of virtual forging is in good agreement with real forged parts, and consequently, we could improve up to 10% of increase in fatigue limit compared to the conventional forged parts. Thus, one may expect more compact sized parts, which in turn, result in weight-down and better fuel economy for better vehicle performance.

## General Abstracts – Powder Metallurgy

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

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| Wednesday AM | Room: 224/225 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chair:* Jim Foley, Ames Laboratory, Ames, IA 50011 USA

**8:30 AM**
**Study of Augmented Sintering Process by Dilatometry Experiments**: *Manoj Mohan Thete*[1]; Philip Nash[1]; [1]Illinois Institute of Technology, Metallurgl. & Matls. Eng., 10 W. 32nd St., E-1, Chicago, IL 60616 USA

Acceleration of sintering kinetics due to thermal cycling has been studied by characterization of property and microstructural changes in FN-0205 associated with augmented sintering process through extensive dilatometry. Green compacts were subjected to repetitive heating and cooling, above and through the phase transformation region by varying heating rates and isothermal holding times. All the process parameters were deter-

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mined by prior isothermal sintering experiments and subsequent property measurements. Substantial property improvement was observed in terms of densification and transverse rupture strength for all the experiments of augmented sintering cycles. While density change and strength decreased with increasing heating rates and decreasing isothermal holding times, an optimum combination of both was seen to produce better properties. Cyclic phase transformations between alpha and gamma showed no significant influence on properties, except at 15°C/min. Effective times for diffusion in various cycles were calculated to help explain the observed property changes.

**8:55 AM**

**Characterization and Developement of Sintering Model for Ti6Al4V Powder Metallurgy Compacts**: *Rajendra Madhukar Kelkar*[1]; Philip Nash[1]; [1]Illinois Institute of Technology, Thermal Processing Tech. Ctr., 10 W. 32nd St., Eng.-1 Bldg., Chicago, IL 60616 USA

This study focuses on characterization of Ti6Al4V (pre-alloyed powder) sintered part. Green compacts of transverse rupture bars (TRS) and tensile bars were made by cold compaction technique at 40T and 45T compaction pressures. Sintering for TRS bars was carried out in dilatometer and tensile bars in vacuum furnace at 1100, 1150, 1200 and 1250°C, for 45, 120 and 240 mins. holding times. Combination of these parameters resulted in 24 different process sequences. Effect of augmented sintering using various thermal profiles was studied. Microstructural characterization was carried out involving porosity measurements, EDS analysis, metallography and x-ray diffraction. Mechanical property measurements included transverse rupture strength, tensile strength and micro-hardness. Dilatometric data were used to calculate the activation energy for sintering. These data are being used to develop a model to predict the sintered density of the parts.

**9:20 AM**

**Wear Failure of a Leaded Bronze Bearing: Correlation Between Plant Experience and Laboratory Wear Test Data**: *Qumrul Ahsan*[1]; A. S.M. A. Haseeb[1]; Ehsanul Haque[1]; [1]Bangladesh University of Engineering and Technology, Dept. of Matls. & Metallurgl. Eng., Dhaka 1000 Bangladesh

The present paper describes an investigation on the failure of a large leaded bronze bearing that supports a nine-ton roller of a plastic calendering machine. At the end of the normal service life of a good bearing which lasted for seven years, a new bearing was installed. But the new one failed catastrophically within a few days generating a huge amount of metallic wear debris and causing pitting on the surface of the cast iron roller. Following the failure, samples were collected from both good and failed bearings. The samples were analysed chemically and their microstructures examined. Both samples were subjected to accelerated wear tests in a laboratory type pin-on-disc apparatus. During the tests, the bearing materials acted as pins which were pressed against a rotating cast iron disc. The wear behaviours of both bearing materials were studied using weight loss measurement. The worn surfaces of samples and the wear debris were examined by light optical microscope, scanning electron microscope and energy dispersive x-ray microanalyser. It was found that the laboratory pin-on-disc wear data correlated well with the plant experience. It is suggested that the higher lead content (~18%) of the good bearing compared to 7% lead in the failed bearing helped to establish a protective transfer layer on the worn surface. This transfer layer reduced metal-to-metal contact between the bearing and the roller and resulted in a lower wear rate. The lower lead content of the failed bearing does not allow the establishment of the transfer layer and leads to rapid wear.

**9:45 AM Break**

**10:10 AM**

**Characterisation of Powder Injection Molded 17-4PH Parts Made from a Mixture of Gas and Water Atomised Powders**: *Sedat Özbilen*[1]; M. Dalkiliç[1]; [1]Gazi University, Fac. of Techl. Edu., Metallurgl. Edu. Dept., Teknikokullar, Ankara, Turkey

In this study, the characterisation of 17-4 PH stainless steel parts made by metal powder injection molding (MPIM) were carried out. For this purpose, feedstocks (compounding) containing a pre-determined multi-component wax-polimer binder mix and a blend of fine (dm= 14µm) and spherical gas atomised and coarse (dm= 23.3µm) and irregularly shaped water atomised 17-4 PH stainless steel powders in 0, 70, 80, 90 and 100 wt% ratios were prepared. By using classical MPIM processing cycle such as mixing, molding, solvent extraction and thermal debinding, sintering at 1250, 1280, 1300 ve 1350°C for 1 and 2 hours under hydrogen atmosphere, above mentioned feedstocks were homogenised at 1000°C for 1 hour under argon gas atmosphere. After iced-water quench, the same 17-4 PH stainless steel MPIM parts were aged artificially at 500 and 550°C for four hours under Ar-gas atmosphere. 17-4 PH stainless steel MPIM parts that were produced after all these above mentioned processing steps, were investigated in detail together with as-received powders, molded and debound samples, sintered, heat treated and aged samples by a combination of characterisation methods such as laser powder particulate size and size distribution analysis, optical and scanning electron metallography, SEM fractography and hardness measurements. At the end of this detailed investigation, not only the influence of the chemical composition of the powder blend of as-received powders in the prepared feedstock but also the different methods of debinding techniques, sintering time and temperature, homogenisation and ageing temperature on the microstructure-property relationships of 17-4 PH stainless steel MPIM parts were studied and determined.

**10:35 AM**

**Characterisation of Mechanically Milled 17-4 Ph Stainless Steel Powders**: *S. Özbilen*[1]; T. Findik[1]; C. Çetinkaya[1]; B. Inem[1]; [1]Gazi University, Fac. of Techl. Edu., Metallurgl. Edu. Dept., Teknikokullar, Ankara, Turkey

In this study, a Szegvari type vertical mechanical alloying/milling attritor for research purposes has been designed and constructed. By using this attritor, optimum processing parameters such as milling time, milling speed (rev/min), diameter and the amount of milling balls, milling atmosphere, raw material properties were determined when mechanical milling of gas and water atomised 17-4 PH stainless steel powders were carried out. After the milling, powder size analyses and microscopic studies (optical microscopy and scanning electron microscopy) were utilised for characterisation of as-received & milled powders. It was observed from the results of the present investigation that for 17-4 PH stainless steel powders, optimum properties were obtained when milling was carried out at 500 rev/min in inert atmosphere by using 250g of 10mm diameter balls which will be presented and discussed.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Damage and Fracture

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee

*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

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| Wednesday AM | Room: 216 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Julia Weertman, Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA; Johannes Weertman, Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA

**8:30 AM**

**Damage Evolution and Fracture Mechanisms of Ceramic Matrix Composites with Nondestructive Evaluation (NDE) Techniques**: *Jeongguk Kim*[1]; Peter K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Eng., 323 Dougherty, Knoxville, TN 37996-2200 USA

Fracture behavior of Nicalon fiber reinforced calcium aluminosilicate (CAS) glass-ceramic matrix composites (Nicalon/CAS) was investigated with the aid of nondestructive evaluation (NDE) techniques. Several NDE methods, such as ultrasonic testing (UT), infrared (IR) thermography, and acoustic emission (AE) techniques, were employed for two different types of Nicalon/CAS composites, i.e. cross-ply and unidirectional specimens. Prior to tensile testing, UT was used to characterize the initial defect distribution of Nicalon/CAS samples, with developing ultrasonic C-scans. During tensile testing, AE sensors and an IR camera were used for in-situ monitoring of progressive damages of Nicalon/CAS samples. AE provided the amounts of damage evolution in terms of the AE intensity and/or energy, and the IR camera was used to obtain the temperature changes during the test. UT was conducted on fractured samples after tensile testing to compare progressive damages with the initial defects. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate fracture mechanisms of Nicalon/CAS samples. In this investigation, NDE techniques were used to facilitate a better understanding of fracture mechanisms of Nicalon/CAS composites during tensile testing.

**8:55 AM**

**Effects of Cyclic Loading, Dynamic Strain-Aging and Neutron Irradiation on Fracture Behavior of A516 Grade 70 and Other Steels**: Chang-Sung Seok[2]; *K. L. Murty*[1]; [1]North Carolina State University, Nucl. Eng., Box 7909, Raleigh, NC 27695-7909 USA; [2]Sungkyunkwan University, Mechl. Eng., Suwon, Korea

Ferritic steels commonly used for pressure vessels and reactor supports in light water reactors (LWRs) are known to exhibit radiation embrittlement in terms of decreased toughness and increased DBTT following exposure to neutron radiation. Recent work indicated decreased toughness during reverse-cyclic loading that has implications on reliability of these structures under seismic loading conditions. Moreover, dynamic strain aging (DSA) results in decreased ductility and toughness. We summarize some of our work on these aspects along with synergistic effects, of interstitial impurity atoms (IIAs) and radiation induced point defects, that result in interesting beneficial effects of radiation exposure at appropriate temperature and strain-rate conditions. Radiation-defect interactions were investigated on pure iron, Si-killed mild steel, A516 and reactor vessel steels (A533B). While dips in fracture toughness are observed in A533B steel in the DSA region, A516 steel exhibited at best a plateau. Load-displacement curves during J1c tests on CT specimens did show load drops in the DSA regime. The effect of load ratio (R) on J versus load-line displacement curves for A516 steel indicated decreased J1c as load ratio is decreased. The hardness and the ball-indentation tests were performed to characterize the strain-hardening at the crack tip. In addition, studies on fast vs total (thermal+fast) neutron spectra revealed unexpected results due to the influence of radiation exposure on source hardening component of the yield stress; grain-size of pure iron plays a significant role in these effects.

**9:20 AM**

**Fracture Toughness Behavior of a Heat-Resistant Alloy, Part I Experiment**: L. Chen[1]; *G. Wang*[1]; R. L. McDaniels[1]; S. A. White[1]; J. D. Landes[2]; P. K. Liaw[1]; K. M. Chang[3]; G. Shen[4]; D. Furrer[4]; B. Skillings[4]; [1]University of Tennessee-Knoxville, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [2]University of Tennessee-Knoxville, Mechl. Eng., Knoxville, TN 37996 USA; [3]West Virginia University, Mechl. & Aeros. Eng., Morgantown, WV 26506 USA; [4]Ladish Company, Inc., Cudahy, WI 53110 USA

In this study, tensile and fracture toughness tests on a heat-resistant alloy were conducted in the temperature range from 24 to 816°C in vacuum. Note that the tests at 24°C were done in air. It was found that both the yield strength and ultimate tensile strength of the heat-resistant alloy decreased, but its elongation first increased slightly, then decreased with increasing the test temperature from 24 to 816°C. The scanning electron microscopy (SEM) observations on the fracture surfaces of tension-tested samples revealed that the amount of cleavages dropped, and the amount of dimples increased as the test temperature increased. When increasing the test temperature, fracture toughness increased. At or below 177°C, valid fracture toughness ($K_{Ic}$) tests were obtained. At or above 204°C, it is necessary to use elastic-plastic fracture mechanics in order to evaluate the fracture toughness of the heat-resistant alloy. Based on the load versus displacement curves of the fracture toughness tests, the ductile-to-brittle transition temperature (DBTT) is between 177 and 204°C. This research is sponsored by Ladish Co., Inc.

**9:45 AM**

**The Brittle-to-Ductile Transition in 4H-SiC**: *Ming Zhang*[1]; H. M. Hobgood[2]; Jean-Luc Demenet[3]; *Pirouz Pirouz*[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA; [2]Cree, Inc., 4600 Silicon Dr., Durham, NC 27703 USA; [3]Laboratoire de Metallurgie Physique, CNRS, SP2MI, 86960 Futuroscope, Cedex, France

The behavior of a crystal undergoes a transition over a temperature interval ΔT below which it is brittle and above which it is plastic. In many covalent materials, ΔT has been found to be very narrow and is often represented by a critical value $T_{BDT}$, known as the brittle-to-ductile transition (BDT) temperature. In this work, the technique of 4-point bend test has been used to measure the BDT temperature of pre-cracked single crystal 4H-SiC. The samples were deformed at temperatures from 800°C to 1300°C at four different strain rates, $5.0*10^{-7}$, $1.0*10^{-6}$, $2.6*10^{-6}$, and $4.0*10^{-6}$ $s^{-1}$. The BDT temperature has been found to systematically increase from ~1050°C at $5*10^{-7}$ $s^{-1}$ to ~1200°C at $4.0*10^{-6}$ $s^{-1}$. The results are discussed in terms of a model that interprets $T_{BDT}$ in terms of a competition between the bond-breaking process and dislocation nucleation in the crystal.

**10:10 AM Break**

**10:25 AM**

**On Brittle Fracture in Pure Polycrystalline Fcc-Metal**: *Peter Panfilov*[1]; Alexander Yermakov[1]; [1]Urals University, Inst. of Physics & Appl. Math., Lenin Ave. 51, Ekaterinburg 620083 Russia

Refractory fcc-metal iridium in single crystalline state exhibits high plasticity, but cleave under tension, while its polycrystals behave like usual brittle material. Plane polycrystalline samples have zero plasticity under tension and the type of their fracture surfaces varies from pure brittle intercrystalline fracture (BIF) for galvanoplastic iridium to pure brittle transcrystalline fracture (BTF) for re-crystallized single crystals. 100% BIF means absolute un-workability, whereas metal having 100% BTF can be successfully used for manufacture of iridium containers. On the other hand, iridium wire, having microstructure of severe deformed single crystal, exhibits 10% of elongation prior the failure at 20°C. Re-crystallization of this metal leads to changing the fracture mode, which becomes mixture of BIF and BTF, and its plasticity is diminished up to zero. Inclination to BIF is an inherent property of iridium, which, however, can be reinforced by segregation of impurities on grain boundaries (GBs). Impurity free GB becomes a dangerous place in material when tensile stress is applied along normal direction to it. Therefore, iridium, ever in polycrystalline state, continues to be fcc-metal, which is a plastic substance, and its “brittleness” is either very specific phenomena or “technological” factor. Highest melting point (Tmelt=2443°C) is the cause why this fcc-metal formally meets cleavage criteria. Therefore, an idea that fracture mechanics cannot be applied to fcc-metals is also true for “brittle” polycrystalline iridium. This work is supported by RFBR (01-03-96438a).

**10:50 AM**

**Elevated Temperature Damages in Type 316 Stainless Steel Plate and Weld Metal Under Monotonic and Cyclic Loading**: *Qumrul Ahsan*[1]; John Fedrick Knott[2]; [1]Bangladesh University of Engineering and Technology, Dept. of Matls. & Metallurgl. Eng., Dhaka 1000, Dhaka, Bangladesh; [2]The University of Birmingham, Sch. of Matls. Metall., Edgbaston, Birmingham B15 2TT UK

The present paper describes work carried out to study “creep-fatigue” interactions in Type 316 plate and weld metal. Sequences of tests have been carried out for plate and weld metal at different temperatures (500°C-625°C) in vacuum with the introduction of hold-time cycles associated with holds at Kmax values corresponding to predetermined DK values. Tests involving the introduction of a hold period in fatigue cycles show higher crack growth rates compared with those in cycles without a hold period at all temperatures. It has also been observed from fractographs of plate material that the fracture surfaces in hold-time cycles exhibit predominantly an intergranular failure mode compared with those for no-hold-time cycles where fatigue fracture is fully transgranular. For weld metal, hold-time cycles and no-hold-time cycles both show the transgranular failure mode at 500°C and 575°C but, at 625°C, hold-time cycles generate fracture surfaces with both intergranular and transgranular failure modes. With an increase of temperature and DK, growth of voids occurs around inclusions in the weld metal during hold periods. This is attributed to thermally activated plastic flow and makes an additional contribution to the crack growth rate in hold-time cycles compared with no-hold-time cycles. Stress relaxation as a result of reduction of stress in a strained member is thought to affect the fatigue crack growth behaviour in those tests. The stress relaxation behaviour of Type 316 plate and plate/weldment has been explored at different temperatures (500°C-750°C) under simple tensile loading. The lower the temperature, the smaller the stress drop and the time required to attain a steady-state drop in stress. The stress relaxation behaviour for plate and plate/weldment are identical and stress relaxation is a time- and temperature-dependent phenomenon. The damage during relaxation test is also temperature dependent and is generated by localised effects.

**11:15 AM**

**Scale Effects in Thin Film Deformation and Fracture:** D. P. Adams[2]; N. Yang[1], A. A. Volinsky[3], W. W. Gerberich[4]; [1]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA; [3]Motorola, Mesa, AZ 85202 USA; [4]University of Minnesota, Minneapolis, MN 55455 USA

Abstract Text Unavailable

**11:40 AM**

**Study of Abnormal Effect of Temperature on Impact Toughness by Instrumented Impact Test**: *Wei Zhou*[1]; Qi Zheng[1]; [1]Nanyang Technological University, Sch. of Mechl. & Production Eng., 50 Nanyang Ave., Singapore 639798 Singapore

V-notched specimens were prepared from an austenitic stainless steel and tested over a wide temperature range -196°C to 100°C. Contrary to the usual expectation, the values of impact energy were found to increase considerably with decreasing temperature. Metallographic evidence was obtained to show long rolling bands in the steel. These bands became brittle at low temperatures and formed long delamination running perpendicular to the direction of fracture propagation. The delamination arrested fracture propagation and left the remaining ligament of the specimen unfractured. In this way, the specimen consumed considerably more energy than straight fracture propagation at higher temperatures. Instrumented impact test was carried out to record the load as a function of deflection. The load-deflection curves made it possible to differentiate the fracture initiation and propagation processes. After the fracture propagated at low temperatures, the load is shown to drop much less drastically due to the presence of crack-arresting delamination, further supporting the delamination toughening mechanisms.

**12:05 PM**
**The Effect of Texture, Inclusion Shape, Orientation and Distribution on Ductile Fracture of Rolled Steel Plates**: *Ahmed Amine Benzerga*[1]; Andre Pineau[2]; Jacques Besson[2]; [1]Brown University, Div. of Eng., 182 Hope St., Providence, RI 02912 USA; [2]Ecole des Mines, Centre des Materiaux, BP 87, Evry, Cedex F91003 France

A new approach to ductile fracture in low carbon steel is proposed that accounts for various sources of material-processing induced anisotropy. The anisotropy of plastic flow and yield was characterized through a set of compression and tension tests while the anisotropy of ductility and toughness was analysed using notched bars and compact tension specimens. Standard quantitative metallography techniques were used to identify the main inclusions, their shape and spatial distribution. Up to 7 loading orientations were considered including several off-axes orientations to study cavity rotation. Fractured specimens from load-control tests were analysed. The mechanisms for damage initiation and crack growth were found to be inherently anisotropic. A micromechanical model was formulated that incorporates most relevant microstructural features such as material's texture, void shape, orientation and relative spacing. The model relies on a detailed characterization of the microstructure and damage mechanisms, hence the increased predictive capability. In this paper, we first show how the model is implemented in a finite element code. Next we demonstrate the relevance of the new approach to discussing the physical reasons of slant fracture under plane strain loading and in pressurized cylinders. Then the model is employed to predict fracture anisotropy in round notched bars.

## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Mechanical Properties - I

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Wednesday AM — Room: 214
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Marc A. Meyers, University of California-San Diego, Dept. of Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

**8:30 AM**
**Why Steel Can Be Strong**: *John William Morris*[1]; David Clatterbuck[1]; Daryl C. Chrzan[1]; [1]University of California, Matls. Sci. & Eng., Berkeley, CA 94720 USA

The attractive structural properties of steel reflect the combination of two useful metallurgical characteristics: bcc iron has high inherent strength, and has a nearby almost-stable fcc phase which makes it relatively easy to process steel into useful microstructures. It has recently been recognized that these characteristics are superficially contradictory. The almost-stable fcc should render steel weak and brittle. The fact that it does not is due to the delicate influence of magnetism on the stability of the fcc phase. The rules that govern the elastic stability and, hence, the ultimate strength of iron also suggest simple interpretations for a variety of other interesting features of its behavior, including the influence of Ni on ductility, the prevalence of alternate slip systems and the absence of simple cleavage in fcc alloys.

**9:00 AM**
**Coarsening Kinetics of Muticomponent MC-Type Carbides in High Strength Low Alloy Steels**: *Kaori Miyata*[1]; Tomohiro Omura[1]; Takahiro Kushida[1]; Yuichi Komizo[1]; [1]Sumitomo Metal Industries, Ltd., Corporate R&D Labs., 1-8 Fuso-cho, Amagasaki, Hyogo 660-0891 Japan

The coarsening kinetics of MC-carbide during tempering in V/Nb/Mo-bearing martensitic steels has been investigated. TEM observations show V and Nb stabilize MC-carbide instead of M2C-carbide. The EDX analysis has identified the MC-carbide as a multicomponent system with a continuous solid solution of MoC, VC, and NbC, when the steel is fully solution-treated and quenched. The concentration of V, Nb, and Mo to MC-carbide has controlled coherency between MC-carbide and matrix. The coarsening kinetics of MC-carbide has been equated to a (time)1/5 criteria, which suggests Ostwald ripening of MC-carbide is controlled by pipe diffusion of V, Nb, and Mo. Applying thermodynamic data, the coarsening equation can be expressed as a function of V, Nb, and Mo-content, and the activation energy for pipe diffusion has been estimated as V:Nb:Mo=1:3.9:0.6. This coarsening equation successfully predicts the contribution of precipitation strengthening to CRSS and proposes the optimum composition and heat-treatment for designing HSLA.

**9:30 AM**
**Structural Relaxation, Crystallization, and Embrittlement in Bulk Metallic Glasses**: *Upadrasta Ramamurty*[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore, KA 560 012 India

In this presentation, we shall review our recent efforts to understand the effects of structural relaxation and partial-nanocrystallization on the mechanical behavior of bulk metallic glasses (BMGs). The BMGs investigated include the La-, Zr-, and Pd-, based glasses which are subjected to isochronal and isothermal annealing heat treatments below and above the glass transition temperature. Changes in the structure of the BMG were evaluated by recourse to XRD, DSC, and TEM techniques. Mechanical properties examined include the hardness, elastic modulus, toughness, and indentation response. Results show that the ability to deform through the shear band mechanism decreases significantly with heat treatment whereas the stiffness and the hardness nominally increase. Such a loss in the deformability of the BMG results in a precipitous drop in the toughness with an associated change in fracture morphology. Microscopic mechanisms responsible for these changes in mechanical properties with the annealing treatment will be discussed.

**10:00 AM**
**Cyclic Plastic Strain Behavior of a Secondary Hardening High Strength Steel**: *Raghavan Ayer*[1]; Paul M. Machmeier[2]; Narasimha-Rao Bangaru[1]; Anthony Matuszewski[2]; [1]ExxonMobil Research and Engineering Company, PO Box 998, 1545 Rt. 22 E., Annandale, NJ 08801 USA; [2]Snap-On Tools Corporation, 2801-80th St., Kenosha, WI 53141-1410 USA

Fatigue behavior of high strength, secondary hardening, steels is usually dependent on the steel microstructure. Fully hardened steels, either wrought or cast, can be expected to cyclic strain soften during low cycle fatigue loading. Structures which have been fabricated by joining or subjected to various thermal treatments in the aging range, will yield different microstructures which can impact the fatigue properties. In this study, the effect of aging of a wrought and/or cast secondary hardening steel, after a range of austenization treatments, on cyclic loading in the plastic regime was investigated. The microstructure of the steel is correlated with the resultant toughness and fatigue properties.

**10:30 AM Break**

**10:45 AM**
**Nanomechanics of Materials and Surfaces**: *Subra Suresh*[1]; Krystyn J. Van Vliet[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., Rm. 8-309, 77 Mass. Ave., Cambridge, MA 02139 USA

Experimental and computational results are presented on the nucleation of defects at surfaces of crystalline materials subjected to nanoscale contact, with the objective of elucidating the nanomechanics of contact for a wide variety of structural and functional applications. In particular, the experiments entail controlled nanoindentation studies of monocrystalline and polycrystalline face-centered cubic materials and model experiments involving indentation of a soap bubble raft. The computational studies involve full three-dimensional simulations of nanoindentation of face-centered cubic materials. The experimental results are compared with computations with the purpose of identifying the conditions for the nucleation and progression of defects during nanoscale contact.

**11:15 AM**
**Nanoscopic Approaches to Materials Design**: *William W. Gerberich*[1]; William M. Mook[1]; [1]University of Minnesota, Chem. Eng. & Matls. Sci., 151 Amundson Hall, Minneapolis, MN 55455 USA

In keeping with the theme of microstructural design, we are determining the mechanical response of the smallest possible building blocks of nanocrystalline composites. These composites, fabricated from hypersonic plasma particle deposition, are made up of nanoparticles in the 2 to 100 nm diameter particle regime and can be Si, Ti, TiC, TiN, etc. Initial evaluations of single silicon nanospheres demonstrate unique behavior ranging from superhardnesses of up to 50 GPa to work hardening rates of 500 GPa, more than an order of magnitude greater than those ever measured in bulk materials. We will briefly discuss how such particles can be deposited, found and measured by nanoindentation and atomic force microscopy. Mostly, however, we will discuss properties and the potential of such nanospheres for revolutionizing material science approaches to structure/property correlations associated with mechanical behavior.

**11:45 AM Invited**
**Where are the Carbon Atoms in Steel?**: *George Smith*[1]; [1]Oxford University, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

Carbon is the dominant strengthening element in steels, and is also one of the chief causes of problems such as strain ageing. The study of the distribution of carbon in steel at the atomic level is therefore of fundamental scientific and engineering importance. However, the study of the distribution of carbon atoms within complex steel microstructures is very challenging, because of the exceptional difficulty in identifying this species by conventional micro- or nano-analytical techniques. The atom probe field ion microscope (APFIM) is capable of detecting single carbon atoms by a mass spectrometric method, and therefore provides a unique tool for the investigation of this problem. The presentation will describe a number of important new insights obtained by this method, including the measurement of carbon segregation to twin interfaces and grain boundaries, the investigation of Cottrell atmospheres, the measurement of the carbon content of retained austenite, and the observation of carbon atom redistrib ution during the tempering of plain carbon and alloy steels. The role of alloy elements in controlling the temper resistance of steels will also be discussed.

## Microstructure/Property Relationships in Alpha/Beta Titanium Alloys: Strain Rate and Second Phase Effects

*Sponsored by:* Structural Materials Division, SMD-Titanium Committee
*Program Organizers:* Patrick L. Martin, US Air Force, AFRL/MLLM, WPAFB, OH 45433 USA; James Cotton, The Boeing Company, Seattle, WA 98124-2499 USA

Wednesday AM — Room: 220/221
October 9, 2002 — Location: Columbus Convention Center

*Session Chairs:* Mike Loretto, University of Birmingham, Dept. of Matls. Sci. & Tech., Birmingham B15 2TT UK; Henry J. Rack, Clemson University, Dept. of Ceram. & Matls. Eng., Clemson, SC 29634-0907 USA

**8:30 AM Invited**
**Influence of High-Strain Rate and Impact Loading on the Structure/Property Behavior of Ti-6Al-4V**: *George T. Gray*[1]; Shuh-Rong Chen[1]; Robert S. Hixson[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

The high-strain-rate stress-strain response of titanium alloys is receiving renewed interest related to systems calculations for crash-worthiness, foreign-object damage, high-rate machining, and ballistic applications. Interest in building physically-based predictive constitutive models to describe these processes requires a knowledge of the coincident influence of temperature and strain rate on mechanical response, particularly under impact conditions. In this paper, the effect of strain rate, temperature, and shock loading on the substructure evolution and mechanical response of Ti-6Al-4V is presented. The constitutive response of Ti-6Al-4V is reviewed and examples of how its mechanical behavior under dynamic loading can be modeled using Taylor impact testing is illustrated. The shock-loading and equation-of-state response of Ti-6Al-4V is discussed in light of the $\alpha$-$\omega$ pressure-induced phase transformation observed in pure Ti. The defect generation, rate sensitivity, and shock-loading response of Ti-6Al-4V are discussed as a function of strain rate and temperature.

**9:10 AM**
**An Investigation into the Response of Ti Alloys to High Strain Rate Deformation**: *M. Zakaria*[1]; Xinhua Wu[1]; W. Voice[2]; M. H. Loretto[1]; [1]University of Birmingham, IRC in Matls., Edgbaston B15 2TT UK; [2]Rolls-Royce, PO Box 31, Derby DE24 8BJ UK

Samples of Ti64, Ti 550 and Ti-15-3 have been subjected to strain rates from $10^{-3}s^{-1}$ to $10^{3}s^{-1}$ and the relative energy absorption during deformation compared. Transmission electron microscopy is being carried out to understand the response of these alloys to the different strain rates. In grains stressed in directions close to <10-10> and <11-20) there is a high density of dislocations which have c-components and at lower strain rates a high density of a- dislocations is observed in these grains. At high strain rates the density of c-component dislocations is again high, but because of the high internal stresses present in these samples the visibility of the a-component dislocations is greatly reduced. Detailed observations of the dislocations and of the level of internal stress will be reported for samples stressed over a wide range of strain rates and these observations correlated with the efficiency of energy absorption during straining.

**9:35 AM**
**The Relationships Between Microstructure and Fracture of Ti-6Al-2Sn-2Zr-2Mo-2Cr-0.15Si Sheet**: *Terryl A. Wallace*[1]; Edgar A. Starke[2]; [1]NASA Langley Research Center, Metals & Thermal Structures Branch, MS 188A, 2 W. Reid St., Hampton, VA 23681-0001 USA; [2]University of Virginia, 116 Engineer's Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

Ti-6Al-2Sn-2Zr-2Mo-2Cr-0.15Si (Ti-6-2222) is a candidate alloy for advanced aerospace vehicles in high-strength, thin-gage applications due to its improved properties compared to conventional Ti alloys. The purpose of this work was to study the formation of $Ti_3Al$ in alpha-beta solution treated Ti-6-2222 sheet and to establish processing-microstructure-property relationships. Samples of 0.050-in thick Ti-6-2222 sheet were solution treated at 1685°F and then aged 1200°F for times up to 1000h to produce various sizes of $Ti_3Al$ precipitates. Tensile properties were determined at room temperature, -65°F, and 350°F. Microstructures and deformation modes were characterized using scanning electron microscopy and transmission electron microscopy, and correlated with mechanical properties. Results showed that strength decreased with aging time. Microstructural changes with increased aging were found to include coarsening of the alpha and beta plates in the transformed beta regions, growth of the $Ti_3Al$ precipitates in the primary alpha grains, increased formation of silicides, and increased tendency for planar slip.

**10:00 AM Break**

**10:15 AM**
**Synthesis and Properties of Discontinuously Reinforced Titanium Composites with TiB Reinforcements**: *K. S. Ravi Chandran*[1]; Krutibas Panda[1]; [1]University of Utah, Metallurgl. Eng., 135 South 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

Discontinuously reinforced Titanium (Ti)-Titanium Boride (TiB) composites with alpha and beta phases in the matrix were synthesized in-situ by reaction sintering of TiB2 and Ti with elemental alloy powders. The goal was to examine the nature of TiB whisker formation in three different kind of powder mixtures. The effects of powder packing and relative locations of powders on the morphological changes in TiB whisker formation and their growth were studied at various processing temperatures. It is shown that the morphology, size and the distribution of whiskers were found to be influenced by the initial packing conditions that existed in the powder mixtures. Tensile tests and fractographic investigations were carried out on these composites. The evolution of composite microstructure, the influence of powder packing variables and the morphology and the growth of TiB whiskers and their effect on mechanical properties are discussed.

**10:40 AM**
**Hot Working Behavior of a 20 vol.% TiB Reinforced Ti-6Al-4V Composite–A Study Using the Processing Map Approach**: Daniel B. Miracle[1]; *Radhakrishna B.V. Bhat*[2]; [1]Air Force Research Laboratory, Sr. Scientist, Matls. & Mfg. Direct., Bldg. 655, WPAFB, Dayton, OH 45433-7817 USA; [2]Systran Federal Corporation, Sr. Scientist, 4027 Col. Glenn Hwy., Ste. 210, Dayton, OH 45431-1672 USA

Discontinuously reinforced Titanium matrix (DRTi) composites have superior specific strength & stiffness, as compared to the respective matrix alloys. They are also amenable to conventional deformation processing unlike the continuous fiber reinforced composites. However, the introduction of particulate reinforcement affects the hot working behavior of the matrix alloys and the optimal hot working parameters for the composite are not the same as those for the respective matrix alloys. Processing maps based on the Dynamic Materials Model is an established technique for studying the hot working behavior of materials. A 20 vol.% TiB reinforced Ti-6Al-4V matrix composite, produced by powder metallurgy, has been subjected to hot compression tests at strain rates ranging from 0.001 to 10 sec-1 and temperatures ranging from 950 to 1200°C. The flow stresses obtained have been used to generate a processing map. The deformation mechanisms corresponding to the various domains in the processing map, the microstructures developed at these processing conditions and their implications on the composite properties will be presented and discussed.

**11:05 AM**
**Probing the Strength of Individual Phases in Ti Alloys Using Nanoindentation**: *G. B. Viswanathan*[1]; Enhua Lee[1]; R. Banerjee[1]; H. L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210 USA

In this study, nano-indentation has been applied to the assessment of the strength of individual phases in Ti alloys containing an alpha/beta two-phase microstructure. Specifically, nanohardness measurements were obtained from individual alpha and beta phases in these alloys. TEM samples were carefully prepared beneath specific indentations using a dual-beam Focused Ion Beam (FIB) instrument and the deformation structures from these samples have been characterized in detail in the transmission electron microscope. The nature of the defect structures combined with orientation information gained from orientation imaging microscopy (OIM) has been correlated with the values obtained from the nano-indentation experiments. In the case of the deformation occurring in the beta-ribs, this phase contains refined distributions of secondary alpha-Ti, as expected from the conventional heat-treatment of these materials. The excising of foils from beneath the nano-indents permits direct observations of the interactions of dislocations in the beta phase with the finely dispersed secondary alpha-Ti.

This research has been supported in part by funding of a Focused Research Group program by the National Science Foundation, Division of Materials Research, with Dr. K. Linga (KL) Murty as Program Director.

**11:30 AM**

**Crystallographic and Morphological Aspects of Alpha Laths in Alpha/ Beta Ti Alloys**: *D. Bhattacharyya*[1]; G. B. Viswanathan[1]; H. L. Fraser[1]; D. Furrer[2]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 N. College Rd., Columbus, OH 43210 USA; [2]Ladish Company, Inc., PO Box 8902, Cudahy, WI 53110-8902 USA

Texture is an important factor determining the properties of metallic forgings, and especially so in Ti alloys because of the anisotropy of the alpha phase. The crystallography of the alpha and beta phases and the Burgers orientation relationship commonly observed between these phases play a prominent role in the microtexture of these alloys. While macrotexture is important to understand the flow characteristics of the forging process, microtexture is crucial in determining local mechanical properties. To determine both macro- and microtexture, orientation imaging microscopy (OIM) studies were conducted on samples from Ti-6246 alloy forgings. The results indicate that in the beta forged condition there are alpha/beta colonies, having close crystallographic relationship but which are morphologically very different, present both within and across prior beta grain boundaries. Also, it was found that when two adjacent beta grains had a common {110} pole, the alpha laths growing into these two different grains seemed to prefer their {0001} poles to be parallel to this particular {110} pole. Moreover, if these two adjacent beta grains are rotated by ~10.5° about the common {110} pole, then the alpha laths growing into these two grains, in two different directions, may have the same orientation, while maintaining the Burgers relationship with both beta grains. Thin foils have been excised from these boundaries using a dual-beam Focused Ion Beam (FIB) instrument and these have been characterized in the transmission electron microscope. This research was supported in part by the Ladish Company, an industrial partner of the Center for the Accelerated Maturation of Materials.

## Modeling the Performance of Engineering Structural Materials: Fracture and Fatigue - I & Fracture and Fatigue - II

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee

*Program Organizers:* Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday AM — Room: 215
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Peter Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., 427- Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; Vikas Prakash, Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., 10900 Euclid Ave., Cleveland, OH 44106-7222 USA

**8:30 AM**

**Probabilistic Aspects of Brittle Fracture**: *John Frederick Knott*[1]; [1]The University of Birmingham, Metall. & Matls., Elms Rd., UK

The paper examines statistical treatments of local brittle fracture stress and fracture toughness at three levels: the micro-scale, the macro-scale and the meso-scale. It traces developments from the RKR/Curry-Knott models, through various forms of other "local" approaches, to the need to estimate credible lower-bound properties for safety assessments. A comparison is made between Weibull analysis of data and the use of a normal distribution. The dangers of employing simple (non-physically-based) extrapolation for materials which are spatially "heterogeneous" is emphasised and guidelines for analysing such data are suggested. Comments are made on the "Master Curve". Application to the brittle fracture of silicon carbide helps to clarify the effects of variation in fracture toughness vs. those of "inherent" defect distribution.

**8:55 AM**

**Dynamic Fracture of Nickel Based Superalloy**: *Vikas Prakash*[1]; Naoto Utsumi[1]; [1]Case Western Reserve University, Dept. of Mechl. & Aeros. Eng., 10900 Euclid Ave., Cleveland, OH 44106-7222 USA

Inconel 718 has proved that it has excellent creep and fatigue resistance at elevated temperatures. However, safety considerations require engine blade containment upon fracture. In view of this, an experimental and theoretical investigation is being conducted at CWRU to understand the dynamic fracture of Inconel 718 in both the annealed and precipitation-hardened state. In order to determine the dynamic fracture toughness and crack propagation characteristics, pre-cracked specimens are subjected to dynamic three-point bend loading utilizing a modified split Hopkinson pressure bar apparatus. Force and load-point displacement are obtained by using strain measurement at a single strain gage station. The history of crack initiation and rapid crack propagation are measured by high-speed photography. The results provide a comparison between the annealed and hardened conditions of Inconel 718, which will be very helpful to the design of engine fan-blade containment systems.

**9:20 AM**

**Dynamic Fracture of Medium Density Polyethylene**: *Vikas Prakash*[1]; Carla Matins[1]; [1]Case Western Reserve University, Mechl. & Aeros. Eng., Cleveland, OH 44106-7222 USA

The objective of this study is to understand the correlation between impact velocity and dynamic fracture toughness in medium density polyethylene. A novel three point bend technique utilizing a modified SHPB is employed. The impact velocity is varied from 1.0 to 7.0 m/s. Strain at a single strain gage placed on the incident SHPB is used to obtain the load-point force and displacement history. The load-point force versus displacement plot is used to obtain the energy absorbed during the dynamic fracture process. Along with these measurements, extensive optical and scanning electron microscopy is carried out to understand the micro- and macro- damage mechanisms operative during dynamic fracture of polyethylene.

**9:45 AM**

**Elevated-Temperature Crack Growth in Nickel-Base HAYNES® 230® Alloy**: *Yulin Lu*[1]; L. J. Chen[1]; P. K. Liaw[1]; G. Y. Wang[1]; R. L. McDaniels[1]; S. A. Thompson[2]; J. W. Blust[2]; P. F. Browning[2]; A. K. Bhattacharya[2]; J. M. Aurrecoechea[2]; D. L. Klarstrom[3]; [1]The University of Tennessee, Dept. of Matls. Sci. & Eng., Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The crack growth behavior of nickel-based HAYNES 230 superalloy was investigated under fatigue, creep, and hold time test conditions at 816 and 927°C. It was noted that both increasing temperature and the introduction of a hold time at the maximum load led to an increase in the crack growth rate. It was also found that the elevated-temperature crack growth rate increased with increasing hold time. Close examination of the crack growth rate versus hold time behavior revealed that crack propagation was cyclicly-dependent at shorter hold times. For long hold times, the crack growth rates became creep-dependent, especially at higher temperatures. For intermediate hold times, the crack growth rates exhibited a creep-fatigue interaction mode. Using the stress intensity factor range as a correlating parameter, the crack growth rates were predicted by linear summation of pure fatigue and creep data. Crack growth rate predictions reproduced most of the characteristics observed experimentally.

**10:10 AM Break**

**10:25 AM**

**Physically-Based Models for Predicting Fatigue Life Variability in Ni-Base Superalloys**: *Kwai S. Chan*[1]; Michael P. Enright[1]; Ronald L. Bagley[2]; David L. Davidson[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA; [2]University of Texas-San Antonio, Dept. of Mechl. Eng., San Antonio, TX 78249 USA

Physically-based fatigue models have been developed for treating the variability of fatigue life due to microstructural variations. Both fatigue crack initiation and growth processes have been modeled in terms of the accumulation of dislocations in a material volume representative of the underlying microstructure. The representative material volume is located at the crack tip for crack growth, but is embedded in an optimally oriented grain for crack initiation. Explicit relationships between fatigue resistance and material parameters such as Young's modulus, dislocation barrier spacing, yield strength, and fatigue ductility have been established. Based on these deterministic models, probabilistic models are developed in which the variability of the material parameters is expressed in terms of appropriate dimensionless random variables. The application of the fatigue models to predict fatigue life variability in Ni-based superalloys will be evaluated against experimental data from the literature. Work supported by the AFOSR through Contract No. F49620-01-1-0547, Dr. Craig S. Hartley, Program Manager.

**10:50 AM**

**Modeling the Crack Growth in Inhomogeneous Materials**: Narendra K. Simha[2]; Dieter F. Fischer[3]; *Otmar Kolednik*[1]; Changrong Chen[4]; [1]Erich Schmid Institute of Materials Science, Austrian Acad. of Scis., Leoben A-8700 Austria; [2]University of Miami, Coral Gables, FL 33124-0642 USA; [3]Institute of Mechanics, Montanuniversität Leoben, Leoben A-8700 Austria; [4]Materials Center Leoben, Leoben A-8700 Austria

The investigation deals with the computation of the effect of material inhomogeneities on the effective crack driving force in elastic-plastic bodies. If the material properties vary in the direction of the crack extension, the effective crack driving force becomes different from the nominally applied far-field driving force. The material inhomogeneities induce an additional crack-driving force term, called the material inhomogeneity term, Cinh, which leads to a crack tip shielding or anti-shielding effect. Cinh is derived from a theoretical material forces model. It is evaluated by integrating the gradient of the stored energy density (which has an explicit dependence on the reference coordinates) in the direction of crack growth. To demonstrate the ability of the model, it is applied to a bimaterial specimen with a gradient interlayer. Different cases are considered: (i) a variation of only the Young's modulus, (ii) the yield stress, (iii) the hardening exponent, and (iv) the simultaneous variation of all the material parameters. Cinh is computed by a simple post-processing procedure to a conventional finite element analysis. The model is checked by computing independently Cinh and the difference between the J-integral on a contour close to the crack tip and the J-integral on a contour in the far-field.

**11:15 AM**

**Modeling the Low Cycle Fatigue Behavior of a Particle Reinforced 7034 Aluminum Alloy Based Metal Matrix Composite**: *T. S. Srivatsan*[1]; Meslet Al-Hajri[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

A study has been made to understand the cyclic stress response, cyclic stress-strain response and cyclic strain resistance characteristics; fatigue life and fracture behavior of aluminum alloy 7034 discontinuously-reinforced with silicon carbide particulates. Specimens of the metal matrix composite were cyclically deformed under total strain amplitude control at both ambient and elevated temperatures for both the underaged and peak aged microstructural conditions. Under total strain-amplitude control, specimens of the composite exhibited combinations of hardening and softening to failure. In this presentation, the cyclic stress response and stress-strain response characteristics, cyclic strain resistance, fatigue life and fracture behavior of the composite for both the underaged and peak aged microstructures, at the two temperatures, will be compared and observed differences rationalized in light of the competing and mutually interactive influences of cyclic strain amplitude and resultant response stress, cyclic stress amplitude, intrinsic microstructural effects, deformation characteristics of the composite constituents and macroscopic aspects of fracture.

**11:40 AM**

**The High Cycle Fatigue and Fracture Behavior of an Al-Cr-Fe Alloy Consolisted by the Technique of Plasma Pressure Compaction**: *T. S. Srivatsan*[1]; S. Givens[2]; Meslet Al-Hajri[1]; R. Radhakrishnan[3]; J. Hutchins[3]; T. S. Sudarshan[3]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA; [2]Bridgestone/Firestone Corporation, Akron, OH, USA; [3]Materials Modification, Inc., 272l-D, Merrilee Dr., Fairfax, VA 22031 USA

Powders of an Al-7Cr-1Fe alloy were prepared by the technique of Gas Atomization and Reaction Synthesis (GARS) at the Ames Laboratory, Ames, Iowa. A pre-alloyed stock of the aluminum alloy was melted and atomized in an inert environment. The atomized particles were classified to less than 45 micron sized powders. The powder particles were then consolidated in a vacuum environment using the technique of plasma pressure compaction (P2CTM). The samples were pulsed at 150°C for 10 minutes and subsequently consolidated at 550°C under a pressure of 40 MPa for 10 minutes. In this presentation, the high cycle fatigue response and final fracture behavior of the aluminum alloy will be highlighted. The cyclic stress amplitude-controlled fatigue properties and fracture characteristics of the aluminum alloy will be highlighted for a range of cyclic stress amplitudes and at two different load ratios. The influence of test temperature and load ratio on stress amplitude versus fatigue life stress response will be rationalized. Also an attempt will be made to elucidate the intrinsic mechanisms governing stress response and fatigue fracture characteristics in light of the competing and mutually interactive influences of microstructural effects, deformation characteristics of the material, cyclic stress amplitude, load ratio and test temperature. Research partially supported by: Air Force Materials Laboratory (USAF), Wright Laboratories (Dayton, Ohio), and The University of Akron (Akron, Ohio) and Dept of Energy.

## Pb-Free and Pb-Bearing Solders - IV

*Sponsored by:* Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA

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| Wednesday AM | Room: 211 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Hans Conrad, North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA; K. N. Tu, University of California-Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095-1595 USA

**8:30 AM Invited**

**Electromigration in Solder Joints and Solder Lines**: Hua Gan[1]; Woojin Choi[1]; *King-Ning Tu*[1]; [1]University of California-Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095-1595 USA

Electromigration has been recognized to be a reliability issue in flip chip solder joints. Eutectic solder is a two-phase alloy, so its electromigration behavior is different from that in Al or Cu interconnects. Besides, a flip chip solder joint has a build-in current crowding configuration to enhance electromigration failure. To have a fundamental understanding of electromigration in SnPb and Pb-free solder alloys, we prepared solder lines in v-grooves etched on (100) Si. Using Cu wires as electrodes for the lines, we found that Pb is the dominant diffusing species tested at 150ºC, but Sn is the dominant diffusing species at room temperature. Interestingly, the rates of Cu-Sn intermetallic compound formation at the cathode and anode are very different. There is a polarity effect on interfacial reactions and it is much faster at anode. In addition, the failure mode of flip chip solder joints will be presented.

**9:00 AM Invited**

**Analysis of Thermal Stress Evolution in Electroless Ni-P Films**: Jaeyong Song[1]; *Jin Yu*[1]; [1]KAIST, Ctr. for Elect. Pkgg. Matls., Matls. Sci. & Eng., 373-1, Kusong-dong, Yusong-gu, Taejon 305-701 Korea

In the low cost flip chip technology, electroless plated Ni-P films have been widely used as under-bump-metallurgy(UBM) to solders. However, high tensile stresses in the films associated with the amorphous to crystalline phase transformation and concomitant precipitation of Ni3P intermetallic compound often cause reliability concerns. In the present work, thermal cycling and isothermal aging experiments were conducted for the Ni-P UBM with varying P content. Then, evolutions of film stress were measured using in-situ laser curvature method. Abrupt stress increases arising from amorphous to nanocrystalline phase transformation and precipitation of Ni3P phases were observed in the amorphous and crystalline Ni-P films, respectively. Microstructural changes during the thermal cycling and aging experiments were analyzed using the XRD, DSC, and TEM, and correlated with the film stress changes. Also, the stress evolution during the solid state reaction between Ni-P and Sn was observed and analyzed with the formation of intermetallic compounds.

**9:30 AM**

**Electrochemical Corrosion Behavior of Lead-Free Sn-Ag Solders**: *Mario F. Arenas*[1]; Ramana G. Reddy[2]; [1]University of Alabama, Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487 USA; [2]University of Alabama, Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487 USA

Due to environmental concerns, a variety of Pb-free solder alloys have been proposed to replace the conventional Pb-37Sn as a general use solder. Sn-Ag alloys are strong candidates for that purpose. Implementation of these solder alloys requires detailed knowledge of the corrosion resistance. However, data on the corrosion properties of Sn-Ag alloys is lacking. In this study the corrosion resistance of various binary and ternary base Sn-Ag alloys were investigated using electrochemical techniques such as corrosion potential measurements and potentiodynamic polarization in $Na_2SO_4$solution. Results were compared against the conventional Pb-Sn alloy. The corrosion products formed during the tests were characterized using scanning electron microscopy and X-ray diffraction.

**9:55 AM**

**The Effects of Both the Cooling Rate and Pb Additions on the Microstructure and Solidification Behavior of the Near-Eutectic Sn-Ag-Cu Solder**: *Adam R. Zbrzezny*[1]; [1]University of Toronto, Dept. of Matls. Sci. & Eng., 184 College St., Wallberg Bldg., Toronto, Ontario M5S 3E4 Canada

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Several solidification regimes, ranging from a very slow unidirectional solidification to quenching, were used to investigate the microstructure and freezing characteristics of the Sn-3.5Ag-0.9Cu and Sn-3.5Ag-0.9Cu with 1-10%Pb solders. It was found that the cooling rate had a strong influence on the resulting microstructure. In the case of lead-free solder, fast cooling rates produced a highly dendritic microstructure with primary ?Ò-Sn dendrites, very fine interdendritic ternary eutectic, and no faceted intermetallic phases. Moderate cooling rates tended to produce a mixture of two binary eutectics (Sn-Cu6Sn5 and Sn-Ag3Sn), regions with ternary eutectic (Sn-Cu6Sn5-Ag3Sn), and sizable Cu6Sn5 intermetallic compounds, often of the shape of hollow hexagonal prisms. Very slow cooling rates yielded big faceted intermetallic compounds in the Sn matrix. The addition of Pb resulted in a development of the dispersed regions of Pb-rich phase and Sn-Pb-Ag ternary eutectic. A significant pasty range, observed with DSC, was a direct effect of Pb-addition.

**10:20 AM Break**

**10:40 AM Invited**
**Effect of an Electric Field on the Phase Distribution in 60Sn40Pb Solder Joints**: *Hans Conrad*[1]; Kang Jung[1]; [1]North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

The phase size distributions (PSDs) of the Sn, Pb and combined Sn and Pb phases was determined for as-reflowed 60Sn40Pb solder joints and following annealing at 50-150ºC. The PSDs in all cases had a positive skew, which was approximated in accord by a log-normal function, and were time invariant in accord with the Bitti-Nunzio model. The eutectic coarsening kinetics for E=0 was of the form Dn-Don=Ko exp(-Q/RT)t where Do=1.0µm, n=4.1 x 0.15, Q=39.8 ± 8 kJ/mole and Ko=(1 ~ 4.5) * 10-23m4/s. An electric field E=5 kV/cm retarded the rate of coarsening at 150ºC of both the Sn and Pb phases.

**11:10 AM Invited**
**Conductive Anodic Filament (CAF) Formation: An Historic Perspective**: *Laura J. Turbini*[1]; [1]University of Toronto, Ctr. for Microelect. Assembly & Pkgg., 184 College St., Rm. 150B, Toronto, ON M5S 3E4 Canada

Conductive Anodic Filament (CAF) is a failure mode in printed wiring boards (PWBs) which occurs under high humidity and high voltage gradient conditions. The filament, a copper salt, grows from anode to cathode along the epoxy-glass interface. First identified by Bell Labs in 1976, this failure mode had been investigated by Dr. Charles Jennings of Sandia who termed it a "punch through" phenomenon. Early studies of CAF were confined to unprocessed PWBs, but in the 1990's Jachim identified the effect of solder fluxes in enhancing this failure mode. This presentation will review the history of CAF from its identification in the 1970's, to the statistical analysis of its failure mode and the factors that enhance its formation.

**11:40 AM**
**Synchrotron Radiation and Focused Ion Beam Study of Tin Whisker Growth on Pb-Free Surface Finish**: *Woojin Choi*[1]; Taekyeong Lee[1]; King-Ning Tu[1]; Nobumich Tamura[2]; R. S. Celestre[2]; A. A. MacDowell[2]; Y. Y. Bong[3]; L. Nguyen[3]; J. T.T. Sheng[4]; [1]University of California at Los Angeles, Dept. of Matls. Sci. & Eng., 6532 Boelter Hall, Box 951595, Los Angeles, CA 90095-1595 USA; [2]Lawrence Berkeley National Laboratory, Adv. Light Source, Berkeley, CA 94720 USA; [3]National Semiconductor Corporation, Santa Clara, CA 95051 USA; [4]Macronix International Company, Hsinchu, Taiwan

We have performed synchrotron radiation micro-diffraction analysis of Sn whiskers grown on the lead-frame finishes of eutectic SnCu and pure Sn to measure the local stress level and the orientation of the grains around the whisker. Also, we examined the grain boundary intermetallic compound formation and the surface oxide on the solder finishes by focused ion beam. Cross-section transmission electron microscopy analysis was carried out. Many more grain boundary precipitates of Cu6Sn5 were found in the eutectic SnCu finish than in the pure Sn finish. The formation of these grain boundary precipitates provides the driving force of whisker growth. The effect of surface oxide is to control the localized and pencil-like growth of whiskers. The mechanism of Sn whisker growth will be reviewed.

**12:05 PM**
**Tin Whisker Growth Driven by Electrical Currents**: *Chih Chen*[1]; Sue-Hong Liu[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., Hsinchu 300 Taiwan

Lead-free solders are replacing traditional SnPb solders gradually due to environmental concern in microelectronic packaging industry. In most lead-free solders, tin is the richest element and weighs over 95% of the solders. Therefore, tin whisker growth driven by electrical currents would be one of the most crucial reliability issues in microelectronic packaging. In this study, we use Blech structure to investigate the electromigration effect in pure tin, in which tin thin film strips of 5000 Å are deposited on titanium thin films of 700Å. Current density of 8×104 A/cm2 is applied in the Blech sample. We observe tin whiskers and hillocks on the anode side and voids on the cathode side. The average growth rate of tin whiskers is measured as a function of current density. Also, to investigate temperature effect, samples are stressed at room temperature and 50°C respectively. The average growth rate of tin whisker as a function of time, current density and temperature will be presented. Furthermore, the growth mechanism of tin whiskers driven by electrical currents will be discussed.

## Rapid Prototyping of Materials: Rapid Prototyping Processes and Process Integration

*Sponsored by:* Materials Processing & Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector; MPMD-Powder Materials Committee, SMD-Composite Materials Committee-(Jt. ASM-MSCTS)

*Program Organizers:* Fernand D.S. Marquis, South Dakota School of Mines & Technology, Department of Materials & Metallurgical Engineering, Rapid City, SD 57701-3901 USA; Rick V. Barrera, Rice University, Mechanical Engineering & Materials Science Department, Houston, TX 77005 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Meisha L. Shofner, Rice University, MEMS, Houston, TX 77005 USA

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| Wednesday AM | Room: 212 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chair:* Enrique V. Barrera, Rice University, Mechl. Eng. & Matls. Sci., Houston, TX 77005 USA

**8:30 AM**
**Evaluation of Microstructure-Property Dependence on Building Rate for Laser Engineered Powder Metal Deposition**: *Pierrette Gorman*[1]; John E. Smugeresky[2]; D. M. Keicher[1]; [1]Optomec Design Company, Albuquerque, NM 87123 USA; [2]Sandia National Laboratories, Livermore, CA 94551 USA

Using the Laser Engineered Net Shaping (LENS™) process, structural materials can be simultaneously synthesized, and formed while microstructure is controlled by appropriate control of process parameters. No tooling, molds, and minimal machining is required to achieve a useful part. This paper attempts to understand the underlying principles that govern the process. By using principles of materials science coupled with what is known about the mechanism of laser based powder metal deposition, a wide range of materials with unique microstructures can be engineered into the forming process. Because LENS™ does not depend on deformation to achieve useful shapes, the process offers potential to enable the use of many advanced material systems. Examples discussed in this paper will focus on 300 Series Stainless to obtain a range of properties dependent on process parameters and composition. Prior work has established that it is possible to obtain a range of tensile yield strengths between two to three times that of conventionally processed annealed material with little change in ductility. At double the yield strength of annealed material, there is no loss in ductility. The results of our microstructure analysis will be discussed to show how both grain refinement and dislocation density affect the mechanical properties. The focus of current work is to determine the robustness of the process, and identify the process window which will guarantee superior material properties. Corresponding work performed to understand other material systems will also be discussed. This work was supported by the US Department of Energy under contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy.

**9:00 AM**
**Solidification and Fusion of Discrete Layers in Precision Droplet Manufacturing: Simulation and Experiment**: *B. Matthew Michaelis*[1]; Jun Zhu[1]; Melissa Orme[1]; [1]University of California-Irvine, Mechl. & Aeros. Eng., 4200 Engineering Gateway, Irvine, CA 92697-3975 USA

Arbitrary metallic components can be synthesized with controlled deposition of nano-liter molten droplets generated by capillary stream break-up and deposited at rates on the order of 10,000 drops/second. This additive precision droplet manufacturing (PDM) process allows objects to be "grown" without the use of a mold or machining. Since objects are formed from discrete drop splats, robust inter-splat unions are desired. We seek to remove the intra-splat boundaries and thus achieve a high quality component by having newly arriving drops remelt a thin layer of the previously deposited and solidified material. This remelting encourages mixing at the splat boundaries and yields a more uniform microstructure. A numerical model, which simulates the solidification-remelting-solidification scheme, is used to understand and guide the process development. Specific effects of substrate temperature, drop arrival temperature and deposition frequency on component build quality are evaluated. An experimental study is conducted to corroborate the simulation results.

**9:30 AM**

**Surface Roughness Determination of As-Received and Laser Polished Indirect-SLS Parts by Means of Line Profilometry**: *Karmen Lappo*[1]; Jeremy Murphy[1]; *Jorge A. Ramos*[1]; Kristin Wood[1]; David L. Bourell[1]; Joseph J. Beaman[1]; [1]University of Texas at Austin, Mechl. Eng., MC C2200, Austin, TX 78712 USA

During laser surface polishing of indirect-SLS metal parts a proficient method for measuring the surface roughness improvements is desired. Currently the ASME B46.1 Surface Texture testing standard does not specify a method for determining a representative Ra value for an area. Laser surface polishing of metallic parts is a promising technique that improves the surface roughness not attributed to waviness. By multiple single line measurements and choosing cutoff filter wavelengths, which correspond to the wavelength due to the waviness of each sample, a representative Ra value can be determined for textured surfaces. Geometry of the precursor material used in fabricating the part (i.e. particle size and shape) as well as its surface morphology can be accounted for when determining a representative Ra surface roughness, as these provide a linkage among the Ra values obtained by line profilometry. Surface roughness results of as-received and laser polished indirect-SLS specimen surfaces are presented.

**10:00 AM Break**

**10:20 AM**

**Modeling the Surface Roughness Enhancement of Indirect-SLS Metal Parts after Laser Surface Polishing**: *J. A. Ramos*[1]; D. L. Bourell[1]; [1]University of Texas, Mechl. Eng., MC C2200, Austin, TX 78712 USA

An analytical model has been developed that predicts the surface texture of indirect-SLS metal (60 wt% 420 stainless steel, 40 wt% brass) parts after laser surface polishing. The surface of a modeled SLS part consists of semi-spherical caps having a diameter distribution similar to that of the precursor powder. The polishing mechanism is shallow surface melting, in which a rastering laser beam causes surface peak melting. Molten material flows from the cusp of the spherical caps into the surface valleys driven by a surface tension unbalance at the triple lines. The model is used to predict surface roughness values as a function of laser power, scan speed and precursor powder particle size with an error not greater than 15%.

**10:50 AM**

**Integration of Rapid Prototyping in Design and Manufacturing**: *Daniel F. Dolan*[1]; Chenoa Jensen[1]; Michael A. Langerman[1]; Fernand D.S. Marquis[2]; [1]South Dakota School of Mines and Technology, Dept. of Mechl. Eng., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Eng., Rapid City, SD 57701 USA

Up to recent years designers have been limited in what they can produce and to adjust their design goals in order to enable the component part or assembly to be manufactured using a specific process or a combination of processes. The introduction of Rapid Prototyping incorporating 3D CAD and 3D CAM made it possible for many of these challenges to be removed, since not only it is not necessary to withdraw the part from the mould but it possible to have re-entrant shapes with easy manufacturing and unlimited geometry capability. This design approach brings new responsibilities to the designer and allows the introduction of multi-materials, multi-functionality and aesthetics into the manufacture of the component part or assembly. These new approaches to design and manufacturing made it possible to fabricate complex custom components at very low prices, bringing with it considerable profit making opportunities. This paper will present how Rapid Prototyping is being introduced into senior capstone design courses and will discuss successful case studies of the application of Rapid Prototyping in design and manufacturing.

**11:20 AM**

**Intelligent Laser Fabrication and Processing**: *Fernand D.S. Marquis*[1]; William J. Arbegast[2]; Michael A. Langerman[3]; [1]South Dakota School of Mines and Technology, Dept. of Matls. & Metallurgl. Eng., Rapid City, SD 57701 USA; [2]South Dakota School of Mines and Technology, Adv. Matls. Procg. Ctr., Rapid City, SD 57701 USA; [3]South Dakota School of Mines and Technology, Dept. of Mechl. Eng., Rapid City, SD 57701 USA

Considerable advances have been observed in intelligent laser processing during the last five years. The South Dakota School of Mines and Technology (SDSMT) has recently acquired state of the art equipment for intelligent laser processing using a 3KW Nd:YAG laser mounted on a Fanuc 16i robot. SDSMT will conduct advanced research on rapid prototyping, solid free form fabrication of tri-dimensional shapes during direct laser deposition and in-situ repair of reactive and refractory metallic structural components. The paper will discuss the state of the art facilities recently acquired and characteristics of planned and on going research.



## Characterization and Representation of Material Microstructures in 3-D: X-Ray Methods

*Sponsored by:* Structural Materials Division,
*Program Organizers:* Mike Uchic, Air Force Research Laboratory, Dayton, OH 45433-7817 USA; Dennis M. Dimiduk, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Milo V. Kral, University of Canterbury, Department of Mechanical Engineering, Christchurch, New Zealand; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375 USA

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| Wednesday PM | Room: 222/223 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chairs:* Dennis M. Dimiduk, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA; Andy Geltmacher, Naval Research Laboratory, Multifunctl. Matls. Branch (Code 6352), Washington, DC 20375 USA

**2:00 PM Invited**

**Non-Destructive 3 Dimensional Microscopy by High Energy Synchrotron X-Rays**: *Dorte Juul Jensen*[1]; Henning Friis Poulsen[1]; [1]Risø National Laboratory, Matls. Rsrch. Dept., Dk-4000, Roskilde, Denmark

A so-called 3DXRD (3D x-ray diffraction) microscope has been developed and is installed at the materials beamline ID11 at the European Synchrotron Radiation Facility in Grenoble, France. It is the only one of its kind in the World. It typically operates at 50-100 keV, which leads to penetration depths in the range mm-cm in most metals and alloys. It allows non-destructive determination of crystallographic orientation and of elastic strain from each diffracting volume within the sample. Volumes as small as 0.5µm can easily be detected and maps of the structure can be resolved with a spatial resolution of about 5µm. The 3DXRD microscope has been designed for fast measurements, and the time resolution is sufficient for a whole range of in-situ kinetics investigations, e.g. recrystallisation, grain growth and phase transformation. The 3DXRD microscope is presented and various applications are used to illustrate its potentials. These include investigations of 3 dimensional grain structures, phase transformation and recrystallisation kinetics.

**2:30 PM Invited**

**Three-Dimensional X-Ray Diffraction Microscopy: Shining a Six-Dimensional Light on Internal Bulk Microstructures**: *Robert M. Suter*[1]; Changshi Xiao[1]; Daniel Hennessy[1]; [1]Carnegie Mellon University, Dept. of Physics, Pittsburgh, PA 15213 USA

Three-Dimensional X-ray Diffraction Microscopy (3DXDM) is a new non-destructive probe of internal microstructure and microstructure dynamics in bulk materials. The technique uses focused, high energy synchrotron X-rays that penetrate millimeters to centimeters of material. We use imaging and ray-tracing of diffracted beams from individual grains to determine grain geometry in three dimensions and crystallographic orientation in three-dimensional Euler space. Spatial resolution is on the micron scale; time resolution can be on the order of minutes. A cubic millimeter of microstructure can be mapped in an hour. In this talk we will describe the technique, its capabilities, and data analysis requirements. Experimental facilities at the European Synchrotron Radiation Facility will be described as will recent developments at the Advanced Photon Source at Argonne National Laboratory. Examples will be drawn from data taken at both laboratories.

**3:00 PM Invited**

**3D Characterization of Damage Morphology in HY100 Steel Using X-Ray Computed Microtomography (XCMT)**: *A. B. Geltmacher*[1]; R. K. Everett[1]; K. E. Simmonds[1]; C. Young[2]; D. A. Koss[2]; [1]Naval Research Laboratory, Multifunctional Matls. Branch (Code 6352), Washington, DC 20375 USA; [2]Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

Improved 3D characterization of void initiation, growth and coalescence behavior is necessary for the development of more accurate ductile failure models. X-ray computed microtomography (XCMT) is a new tool for studying the internal structure of organic, metallic, and ceramic materials. Just as CAT scans have revolutionized medical imaging, XCMT provides a unique and exciting look into the microstructure of advanced engineering materials. Three-dimensional, high-resolution micro- and meso-structural images can be determined with 1-2 mm precision providing improved insight and understanding of the mechanisms involved in materials damage and failure. In the case of HY100 steel, voids are known to nucleate at very small strains along MnS inclusions. Previous research has shown that these inclusions are non-uniformly distributed and occur in banded microstructures, which are susceptible to coalescence by a "void sheeting" process under high triaxial stress states. In the present research, XCMT has been used to examine the morphology of damage in notched tensile specimens that have been strained to 69% and 87% of the failure strain. The XCMT results highlight complex damage morphologies developed in this material. The images show the preferential void growth in the bands parallel to the rolling direction and linking by void sheeting between the bands. The voxel data have been extracted and used to create a 3D finite element model of two voids. The simulations indicate rapid accumulation of strain in the region between the two voids. The XCMT data provide an unprecedented level of detail of the 3D damage morphology and it has been used to develop a new damage model for HY100 steel.

**3:30 PM Break**

**3:50 PM Invited**
**USAXS Imaging: A New Probe of Materials Microstructure**: *Gabrielle G. Long*[1]; Lyle E. Levine[1]; [1]NIST, Matls. Sci. & Eng. Lab., 100 Bureau Dr., Stop 8520, Gaithersburg, MD 20899-8520 USA

Ultra-small-angle X-ray scattering imaging (USAXS imaging) is a new transmission X-ray imaging technique that makes use of USAXS as the contrast mechanism. Until now, images of bulk structures within materials have relied on X-ray absorption contrast and/or X-ray phase contrast, and, in the case of single crystals, X-ray diffraction contrast. As a distinct imaging technique, the USAXS contrast mechanism offers: extremely high sensitivity to density variations; general applicability to single-crystal, polycrystalline and amorphous materials; and its USAXS-derived capability to extract quantitative microstructural information from images acquired at different scattering vectors. Remarkably, this kind of information becomes available in the USAXS images even when the scattering objects are below the spatial resolution in size. Three-dimensional (3-D) information on the sizes, shapes and spatial arrangements of structures are available from stereo-USAXS image pairs. A goal of future development is the implementation of full 3-D USAXS tomography.

**4:20 PM Invited**
**The 3D X-Ray Crystal Microscope: A New Tool for the Study of Materials**: *G. E. Ice*[1]; B. C. Larson[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Rm. B260, MS 6118, One Bethel Valley Rd., Oak Ridge, TN 37831-6118 USA

A new class of X-ray diffraction instrumentation has been made possible by the availability of intense polychromatic synchrotron radiation combined with advances in X-ray optics and X-ray area detectors. These instruments are distinguished by the ability to study layered and true polycrystalline materials with submicron spatial resolution both transverse to and along the probe beam. A prototype 3D microscope at the Advanced Photon Source has recently been commissioned and has begun experiments. This instrument uses polychromatic radiation to obtain a full Laue pattern from every subgrain that intercepts the nondestructive and penetrating hard X-ray beam. Single-crystal-like information can be recovered from every subgrain by the use of energy scanning and differential aperture methods. Not only is the microscope sensitive to phase, subgrain crystallographic orientation and morphology, but it can also map subgrain distributions of the plastic and elastic strain tensor. The ability to nondestructively map 3D distributions of stored plastic deformation energy, elastic strain and the detailed 3D local crystallographic environment of buried grains is certain to advance our understanding of mesoscale structural evolution in materials.

**4:50 PM**
**Computed Microtomography Studies to Characterize Microstructure Property Correlations in Ceramic Coatings**: *A. Kulkarni*[1]; A. Goland[1]; S. Sampath[1]; H. Herman[1]; J. Ilavsky[2]; A. Allen[2]; G. Long[2]; [1]State University of New York, Stony Brook, NY, USA; [2]National Institute of Standards and Technology, Gaithersburg, MD, USA

A quantitative multidisciplinary approach towards materials characterization is being undertaken for Processing-Microstructure-Property correlations in plasma sprayed and Electron-Beam Physical-Vapor-Deposited (EB-PVD) ceramic coatings. High-resolution X-ray microtomography is being explored for the first time, with emphasis on quantitative characterization of imperfections (porosity, pore size distribution, pore connectivity and morphology) in these coatings and on the relative changes in microstructural features upon thermal cycling. Two dissimilar coating microstructures, anisotropic layered structure in case of plasma sprayed coating and unique columnar pore morphology in EB-PVD coating system are principally studies materials. Some of the complex relationships have been explored between the observed microstructural features and their correlation to thermal conductivity and elastic modulus of the coatings.

**5:20 PM**
**Measuring the 3-D Grain Topology of a Polycrystal Using Absorption-Contrast Tomography**: *Carl E. Krill III*[1]; Kristian Döbrich[1]; Christoph Rau[2]; [1]Universität des Saarlandes, FR 7.3 Technische Physik, Postfach 151150, Geb. 43B, D-66041 Saarbrücken, Germany; [2]ESRF, BP 220, F-38043 Grenoble, Cedex, France

Characterizing the topology of grain-boundary networks in polycrystalline materials is a crucial step in the modeling of properties and phenomena that depend on the grain microstructure. For example, the coarsening rate of individual grains in a polycrystal is thought to be affected by correlations in the sizes of neighboring grains induced by the process of grain growth itself. In the past, most topological studies of bulk polycrystalline materials have been carried out on single planar sections, which at best provide access to average topological quantities. In contrast, local topological information can be obtained only by a three-dimensional characterization method, such as serial sectioning. For some classes of samples, x-ray tomography represents a potentially faster and more accurate alternative route to the same information, as we have demonstrated for specimens of Al doped with up to 3 at.% Sn. In this material, the tin atoms segregate to the grain boundaries, thereby imparting a strong contrast in attenuation that can be reconstructed tomographically. Common to both serial sectioning and tomography is the challenge of extracting quantitative microstructural information from the resulting three-dimensional images. In the case of Al-Sn, we applied an iterative region-growing algorithm followed by a novel grain-boundary-network optimization step (based on a phase-field simulation of grain growth) to obtain 3-D topological information for 5000 Al grains, including a first measurement of the nearest-neighbor size correlation function in this material. The resulting information has been incorporated into a non-mean-field theory for grain growth, the accuracy of which was evaluated by comparing its predictions with the observed microstructure of the Al-Sn samples.

## Forming and Shaping of Light Weight Automotive Structures: Processing & Manufacturing - I

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

Wednesday PM    Room: 210
October 9, 2002    Location: Columbus Convention Center

*Session Chair:* Mahmoud Demeri, Ford Motor Company, Mfg. Sys., Dearborn, MI 48121 USA

**2:00 PM Invited**
**Structural Cast Magnesium Development**: *Richard Osborne*[1]; [1]General Motors Corporation, Matls. Ctr., MC 480-205-314, 30007 Van Dyke, Warren, MI 48090-9065 USA

This presentation will discuss the critical technical and manufacturing issues that limit the large-scale application of structural cast magnesium automotive components. The presentation will feature project activities that focus on science and technology development necessary to implement front and rear magnesium structural cradles. This project is also closely aligned with a concurrent OEM chassis program, which will provide a validation test bed for the technology and tools developed. Current project challenges, task activity status and participants will also be highlighted in the presentation.

**2:30 PM Invited**
**Advances in Tube and Sheet Hydroforming for Lightweight Automotive Structures**: *Taylan Altan*[1]; [1]Ohio State University, ERC/NSM, Rm. 339, 1971 Neil Ave., Baker Systems Bldg., Columbus, OH 43210 USA

Reduction in vehicle weight while maintaining size and comfort may be achieved by a) design innovations, i.e. new configurations that offer increased stiffness to weight, or b) use of materials with high strength to weight ratios. Design innovations include the use of tubular materials. tailored welded, tailored rolled, or textured sheet while light weight materials include the use of alloys from aluminum, magnesium and high strength steels. Both alternatives require new methods of product and process design and detailed knowledge of material properties under deformation conditions. Furthermore, with the increase use of new forming methods the use of FEM simulation for process optimization becomes an absolute requirement. This paper will review a) methods for determining material properties needed for process design, b) the latest advances in tube and sheet hydroforming, c) elevated temperature forming of aluminum and magnesium alloys, d) stamping of HSS, tailored welded and tailored rolled sheet, and e) advances in process simulation such as adaptive simulation and optimization techniques.

**3:00 PM**
**Robust Trimming of Aluminum Panels**: *Sergey Fedorovich Golovashchenko*[1]; [1]Ford Motor Company, Mfg. Sys. Dept., Scientific Rsrch. Lab., MD3135, 2101 Village Rd., Dearborn, MI 48124 USA

Experimental and numerical results on trimming of aluminum body parts will be presented. Mechanism of fracture and slivers generation in conventional trimming process will be explained based on laboratory experiments and numerical simulation highlighting the effect of bending. The new trimming process, eliminating slivers and creating robust trimming conditions will be discussed. The simulation of the trimming process was done using a numerical solution of the solid mechanics equations of motion, assuming the blank deformation to be plane strain. The numerical integration procedure is based on a modified Wilkins explicit method with triangular grids. The cumulative damage theory was used to predict the material fracture at every element in the blank. The reduction in ductility, which was calculated for every time interval, was determined using cumulative damage fracture criterion dependent upon the mean stress.

**3:25 PM**
**Roll Hemming of Aluminum Closure Panels for Improved Process Robustness**: *P. A. Friedman*[1]; [1]Ford Research Laboratory, 2101 Village Rd., MD 3135, Dearborn, MI 48121 USA

In an effort to reduce vehicle weight, the automotive industry has switched to aluminum sheet for many high volume closure panels. The current trend for these applications has been the use of a heat-treatable Al-Mg-Si alloy (AA6xxx). While these materials possess ample formability for stamping, they often have insufficient bendability to achieve a flat hem around thin inner reinforcement panels. In automotive production, two main types of hemming are typically used: press-action where the entire flange is bent inwards at the same time and roll hemming where a wheel traverses the perimeter of the panel while bending the metal inwards. In this talk, the distinct advantages of roll hemming over more traditional press-action hemming for aluminum closure panels will be discussed.

**3:50 PM Break**

**4:00 PM**
**Hybrid Electromagnetically Assisted Stamping**: *Jianhui Shang*[1]; Vincent J. Vohnout[1]; *Glenn Steven Daehn*[1]; [1]Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

One of the central problems in stamping is that strain is non-uniformly distributed in formed structures. This takes place because friction produces a situation where forces and hence strains necessarily vary from place to place. We will outline a very general solution to this problem–electromagnetic actuators are embedded in rather standard stamping tools. When a large impulsive current is run through these actuators, eddy currents are induced in the work piece. This produces a pressure pulse that can be controlled in magnitude and location. This pulse can be used to impart strain in a desired pattern in the sheet metal. An advancing tool can then be used to set the shape of the forming part. This process can be repeated an arbitrary number of times. Both a section analysis of this process as well as experimental data for simple geometries showing the effects of key parameters will be presented.

**4:25 PM**
**Structural High Velocity Crimping for Lightweight Structural Fabrication**: *Peihui Zhang*[1]; Glenn Steven Daehn[1]; [1]Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Crimping, the creation of mechanical joints by a combination of bending and possibly an interference fit has been practiced for many years for many types of applications. Despite the ubiquity of this joining method, there are few to no careful studies in the engineering literature to enable the fabrication of structural crimp joints. Moreover, if crimping is carried out at high velocity (as can be enabled by electromagnetic forming) an interference fit can be naturally accomplished. This presentation will provide a combination of dynamic finite element simulations and experimental results that shed light on the following questions: 1) How and why does high velocity forming naturally develop an interference fit? 2) How does one optimize the design of such high velocity crimp joints? 3) What kinds of strength can be expected from such joints? and 4) How might structural crimping enable lightweight high-strength automotive structures?

**4:50 PM**
**Springback Reduction Through Post-Stretching**: *Z. C. Xia*[1]; Feng Ren[1]; [1]Ford Motor Company, Scientific Rsrch. Labs., 2101 Village Rd., MD3135/ SRL, PO Box 2053, Dearborn, MI 48121 USA

Springback is a major problem for stamping as aluminum and other light-weight materials are increasingly used. A technique to reduce springback through post-stretching is presented, which is most effective for drawing of open channel type of structures such as rails and frames. In this technique, a second set of drawbeads is activated near the end of the drawing operation, effectively locking metal flow in the binder and forcing the draw wall into stretching mode. Numerical simulations for a U-channel drawing are used to analyze the process. After an examination of deformation profile after drawing and wall curl as a result of springback, various magnitude of post-stretch amount is simulated and their deformation history is traced in order to understand the mechanism of this technique. It is found that a post-stretch strain around 2% almost completely eliminates wall curl. Experimental work with a complex rail is also discussed.

## General Abstracts – Surface Engineering

*Sponsored by:* TMS
*Program Organizers:* TMS, Warrendale, PA 15086 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Wednesday PM — Room: 224/225
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Sudipta Seal, University of Central Florida, AMPAC, Orlando, FL 32826 USA

**2:00 PM**
**Microstructure Development in Raney™ Type Cobalt Catalysts**: *Mi-hee Cho*[1]; Kevin P. Trumble[1]; [1]Purdue University, Sch. of Matls. Eng., 1289 Matls. & Electl. Eng. Bldg., W. Lafayette, IN 47907-1289 USA

Raney™ type catalysts are well known as high-surface materials. They are usually made from aluminum-transition metal alloys by selective leaching of aluminum in a caustic solution. The leaching leaves the transition metal in a highly porous microstructure having high surface area. Compared to the more common Raney™ Ni alloys, the Al-Co binary system has many more high-aluminum intermetallic phases, which have yet to be thoroughly investigated for catalyst applications. As-cast and annealed microstructures of the full range of high-aluminum Al-Co binary alloys have been characterized by optical microscopy, SEM and EDS, XRD, and quantitative stereology. Preliminary results on microstructure development in leaching will also be discussed.

**2:25 PM**
**Fracture Study of Aluminum Composite Coatings Produced by the Kinetic Spray Method**: *Thomas H. Van Steenkiste*[2]; Alaa A. Elmoursi[1]; Daniel Gorkiewicz[1]; Bryan Gillispie[1]; [1]Delphi, Delphi Rsrch. Labs., M/C 483.478.101, 51786 Shelby Pkwy., Shelby Township, MI 48316 USA; [2]Delphi, Delphi Rsrch. Labs., M/C 483.478.105, 51786 Shelby Pkwy., Shelby Township, MI 48316 USA

The kinetic spray process is a low temperature method for producing coatings. This process allows one to formulate various mixtures of metals, ceramics, polymers, etc. and combine them with a ductile material (as the matrix), to produce composite coatings. The low temperatures involved result in little oxidation or reaction between the parent materials of the composite coating. This report deals with the study of fractured surfaces of aluminum composites using a matrix of aluminum with reinforcing particles of diamond, tungsten, molybdenum, silicon carbide or aluminum nitride. Fracture surfaces were examined with the SEM microscope for evidence of bonding.

**2:50 PM**
**Flow and Temperature Profiles Near the Crystal-Solution Interface During Industrial Hydrothermal Growth of Quartz Single Crystals**: *Hongmin Li*[1]; [1]The University of Akron, Dept. of Mechl. Eng., 302 Buchtel Mall, Akron, OH 44325 USA

High quality single quartz crystals can be obtained with hydrothermal growth. Crystal plates grow when the nutrient molecules precipitate onto seed surfaces and join the crystal lattice from the surrounding aqueous solution. Crystal growth is a complicated physical and chemical process near the seed-solution interface. The surface shapes of the crystals grown in industry hydrothermal autoclaves imply that local flow and temperature profiles near the crystal-solution interface have critical effects on the surface process. In this study, convective flow and heat transfer in an industry size autoclave was numerically simulated. Flow and temperature profiles near the crystal-solution interface were obtained. Local variations of temperature and velocity gradient were presented. The effects of the local distributions on the growth at the crystal seed surfaces were analyzed. A heater arrangement to obtain a better growth uniformity on crystal surfaces was proposed.

**3:15 PM**
**Analysis of Tantalum Coatings Produced Using the Kinetic Spray Process**: *Thomas H. Van Steenkiste*[1]; [1]Delphi Research Laboratories, Mfg.,

51786 Shelby Pkwy., Shelby Township, MI 48316 USA

Tantalum coatings have been produced using a relatively new process, kinetic spray, (similar to the cold spray process). Tantalum starting powders (having particle diameters greater than 50 microns) are injected into a de-Laval-type nozzle, entrained in a supersonic gas stream, and accelerated to high velocities due to drag effects. The particle's kinetic energy is transformed into plastic deformation, strain and heat on impact with the substrate surface. Particles are not thermally softened or melted, producing low oxide, reduced residual stress, high adhesion and low porosity coatings. Analysis of the mechanical and physical properties of these tantalum coatings will be presented. Comparison between kinetic sprayed tantalum coatings and tantalum coatings produced using conventional coating methods will be discussed.

**3:40 PM Break**

**4:05 PM**
**Deposition of Protective Coatings by the Kinetic Spray Process**: *Zhibo Zhao*[1]; Bryan A. Gillispie[1]; Thomas H. Van Steenkiste[1]; John R. Smith[1]; [1]Delphi Research Laboratories, 51786 Shelby Pkwy., Shelby Township, MI 48315 USA

This paper describes deposition of metallic coatings using the kinetic spray process. This coating process is a relatively new process that involves using high velocity metallic particles to generate surface coatings. In this coating process, metallic particles (>50 mm) are injected into a de-Laval nozzle and propelled to high velocities (>400 m/s) by a supersonic gas stream before they impinge onto a substrate. The coatings are formed subsequently as the metallic particles are severely deformed plastically and bonded to both the substrate and one another. The process can generate the coatings with little oxidation, low residual stress, low porosity and a wide range of coating thickness (microns to centimeters), along with rapid coating buildup. In this study, the kinetic spray process is utilized to deposit a number of protective coatings, including metal coatings, alloy coatings and composite coatings. Several fundamental aspects of the coating process, including coating formation, mass loading behavior and microstructure of coatings are investigated.

**4:30 PM**
**Metalorganic Chemical Vapor Deposition of Ti-Oxynitride Thin Film Using Titanium Isopropoxide and Ammonia**: *Siddhartha Kumar Pradhan*[1]; Phillip J. Reucroft[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA

Ti-oxynitride is a hard coating material suitable for wear resistance and protective coating applications. Titanium isopropoxide (TIP), a metal-organic precursor, and ammonia are used as source materials to deposit $TiO_xN_y$ films in a conventional CVD reactor. At 500°C anatase ($TiO_2$) film is deposited. At higher deposition temperature the film is mostly TiO and above 600°C highly oriented $TiO_{0.7}N_{0.3}$ film is deposited. Studies on morphology and microstructure of these films will be presented to elucidate film growth behavior.

**4:55 PM**
**Mechanics of Cogwheel Pair Based on Alumina-Chrome Carbide Composites**: *Maksim Kireitseu*[1]; [1]Institute of Machine Reliability, Mech. of Composites, Lesnoe 19-62, Minsk 223052 Belarus

Cogwheels usually operate in strong conditions of complex load, thermal and mechanical stresses, few are less forgiving in the event of a failure. Thus, one of the considerations in the design of cogwheel pairs is the reliable work and durability of the system. For many years, the automotive cogwheels have been manufactured from low-carbon steel and other steels. On the other hand, an advantage that aluminum has over steel is high corrosion resistance, low weight as well as the manufacturing cost. For many companies this means looking for better alternative in technologies that are used to coat aluminum with hard alumina layer, a coating technique is under increasing pressure of an environmental-protection standpoint. Alumina is one or coatings that are more superficial applied to improve wear and corrosion resistance, load rating and thermal stability of aluminum parts. Although coating composition has changed in the best mode since the original anodized alumina layer to modern hard alumina formed by microplasmic or micro arc oxidizing process, coating application remain a problem. In particular, this paper will consider the aspects in the mechanics of cogwheel pair based on alumina composite on aluminum and alumina hardened by chrome carbide composites. Summarizing results, it has been revealed the following: (a) alumina-based composites improve load rating, wear rate of the aluminum substrate and vibroacoustic characteristics of aluminum cogwheel pairs in contrast to steel one. Alumina coatings on the cogs of the wheel decrease vibration and noise of the pair at 3-4 dB in some frequencies between 0.1-7 kHz of frequency band. However, the achieved effects depend on speed of rotation and applied moment at the pair. (b) major vibrations and noise generated by cogwheels and rolling bearings are damped by the alumina composite during work of the cogwheels. Researches revealed that an effect of damping is increased by the multilevel and inhomogeneous structure of the composite. Tests of porosity disclosed that aluminium with porosity up to 15% and more and oxide ceramics allow to damp vibrations and noise in multistage and discrete mode. (c) to provide durability, reliability and overall performance, optimal parameters of alumina-based composites (thickness of the layers, hardness and porosity) have been revealed. (d) Alumina hardened by chrome carbide layer has the lowest coefficient of friction then that of alumina, whereas contact fatigue strength of the pair may be decreased. (e) The combination of polymer layer and layer of aluminium or its alloy as a good noise dampers reduced vibrations and noise of the gearbox.

**5:20 PM**
**Durability and Reliability of Composite System: Chrome Carbide–Hard Anodic Oxide Ceramics–Aluminium**: *Sergey Yerakhavets*[1]; [1]Institute of Machine Reliability, Mech. of Composites, Lesnoe 19-62, Minsk 223052 Belarus

The reliability and durability of Al-Al2O3-CrC composite ceramic coatings in the view of corrosion resistance, service life and dynamic load rating have been investigated. An effect of CrC layer on durability and service life of composite coating based on alumna ceramics was discussed. Summarizing the results, they revealed the following. (1) Chrome carbide layer has an advanced corrosion resistance than alumna. Visual observations of tested samples with CrC did not have any peeled off particles or corrosion on surface viewed by optical microscope with 500x resolution. The coating provides desired performance after 700 hours of tests in strong mix of sulfuric and salt acids in contrast to steel or alumna parts. Durability and load rating of the Al-Al2O3 coating in strong aggressive acid environment can be improved in two times by deposition of chrome carbide layer. (2) The results of structure investigation revealed that an optimal combination of mechanical properties of materials used in composite provide improved durability and reliability of the Al-Al2O3-CrC composite coating. As the researches discovered sub layer of oxide aluminum ceramic bears significant level of applied loads as result of its high strength, microhardness and about 300µm thickness. CrC layer provides both low level of stresses in the coating and high tribological parameters. Due to a little thickness CrC layer has low level of internal stresses. Particles of chrome carbide penetrate into the pores of oxide ceramic layer that improves both adhesion and load rating of the composite. In other words, CrC layer strength oxide ceramic. In result, this load rating and overall reliability of the coating exceed the same parameters of separated layer of CrC or alumna. (3) Control of technological parameters revealed that opened on surface pores of alumna and pressure of vapors during deposition of CrC particles effect on adhesion, load rating of the coating. The hardest coating have pores of 1-4µm in diameter; pressure of vapors during pyrolitic deposition of CrC particles ranges in 3-8 Pa. (4) The coating can replace many parts such as sliding and thrust bearings, engines, turbines, etc. that are made of expensive steels working in aggressive environment Particular application depends on real loads in machine and speed of rotation of moving parts. Nevertheless, the coating has an advanced load rating and corrosion resistance. Along with the aluminum, the hard oxide layer can be formed on titan, tantalum and other light-weighted and strong metals, that might eliminate some revealed disadvantages in particular case.

## Mechanisms and Mechanics of Fracture: Symposium in the Honor of Professor J. F. Knott: Fatigue of Materials

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee

*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Wednesday PM Room: 216
October 9, 2002 Location: Columbus Convention Center

*Session Chairs:* Sami El-Soudani, Boeing Company, Phantom Works, Long Beach, CA 90807-5309 USA; Yakichi Higo, Tokyo Institute of Technology, P&I Lab., Yokohama 226-8503 Japan

**2:00 PM**
**Thermography Detection of the Fatigue Behavior of Reactor Pressure Vessel (RPV) Steels**: *Bing Yang*[1]; Peter K. Liaw[1]; Hsin Wang[2]; Liang Jiang[1]; Lijia Chen[1]; D. Fielden[1]; J. Y. Huang[1]; R. C. Kuo[1]; J. G. Huang[3]; [1]University of Tennessee-Knoxville, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA;

[3]Taiwan Power Company, Taipei, Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 0.5 Hz and 20 Hz fatigue testing. Temperature oscillation during fatigue, temperature evolution during low-cycle fatigue is discussed. The relationship among temperature, stress-strain state, and fatigue behavior is investigated. Both thermodynamics and heat-transfer theories are applied to model the observed temperature variation during fatigue. Temperature evolution of fatigue specimen with different geometries are compared and analyzed.

**2:25 PM**

**Critical Examination of Fatigue Thresholds**: *K. Sadananda*[1]; Ronald L. Holtz[1]; A. K. Vasudevan[2]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375 USA; [2]Office of Naval Research, Arlington, VA 22217 USA

The Unified Approach developed by the authors indicates that fatigue involves two load parameters, Kmax and deltaK. This requirement manifests as two thresholds needed for a fatigue crack to grow. Crack arrest occurs if either one is not met. At low R-ratios, Kmax is the controlling parameter since threshold deltaK is generally met. At high R-ratios deltaK becomes the controlling parameter since Kmax threshold is met due to high-applied Kmax values. Requirement of cyclic plasticity insures that reversibility of plastic flow should govern the cyclic threshold and criteria for crack opening or breaking of crack-tip bonds should govern the Kmax threshold. Dislocation models will be examined in the light of these requirements. Effects of environment, internal stresses, role of crack closure on the two thresholds will be examined. The models will be compared with the experimental results.

**2:50 PM**

**Advances in Ultra-High Cycle Fatigue**: *Melanie Jean Kirkham*[1]; Peter K. Liaw[1]; [1]University of Tennessee, Matls. Sci. & Eng., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA

Traditional fatigue analysis identifies in some metals a fatigue limit, which is the stress limit below which the metal will not fail after an infinite number of cycles. The calculation of traditional fatigue limits is based upon measurements to a maximum of around 106 cycles. In the past, this approach worked well because fatigue lives of machinery were below 106 cycles. However, more modern applications, for example in the automobile and aerospace industries, can require fatigue lives over 108 cycles. Recently, research has begun to extend S-N curves into the gigacycle range, also called Ultra-High Cycle Fatigue (UHCF). Failure at stresses below traditional fatigue limits has been discovered. Extended S-N curves exhibit a stair-step shape with a second drop in stress in the UHCF range. Investigation into crack initiation suggests that while failure below 107 cycles is primarily due to surface-initiated cracks, failure in the UHCF range is primarily due to internally initiated cracks. This paper reviews the research being conducted on Ultra-High Cycle Fatigue. This work is supported by the National Science Foundation, the Division of Design, Manufacture, and Industrial Innovation, under Grant No. DMI-9724476.

**3:15 PM**

**Fatigue Crack Growth in Ti-10V-2Fe-3Al Beta Titanium Alloy–Role of Microstructure and Crack-Wake Asperity Contact**: *Sushant K. Jha*[1]; K. S. Ravi Chandran[2]; [1]Systran Federal Corporation, 4027 Col. Glenn Hwy., Ste. 210, Dayton, OH 45431-1672 USA; [2]University of Utah, Metallurgl. Eng., 135 S., 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The observations of effects of microstructure and the unusual crack closure responses occurring during fatigue crack growth in different microstructures of the metastable ß-titanium alloy, Ti-10V-2Fe-3Al are presented. It is shown that microstructural unit sizes of the order of a few microns can produce measurable crack closure responses that can be ordered in terms of the microstructure size scale. The development of crack closure during the decreasing and increasing $\Delta K$ tests were different, due to the nature of roughness development in the crack-wake. A simple hinge-type model showed a good correlation between the predicted fracture surface asperity height created during near threshold fatigue crack growth, with the primary alpha particle spacing in the microstructures of this alloy. Fractographic studies as well as crack path observations were also found to be consistent with the predictions.

**3:40 PM Break**

**3:55 PM**

**Temperature Evolution During Fatigue of HASTELLOY® C-2000® Alloy**: *T. A. Saleh*[1]; B. Yang[1]; P. K. Liaw[1]; R. A. Buchanan[1]; D. L. Klarstrom[2]; [1]University of Tennessee, Matls. Sci. & Eng., 434 Dougherty Hall, Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

Infrared (IR) thermography was employed as a non-destructive evaluation technique to evaluate the thermal behavior of HASTELLOY® C-2000® alloy during fatigue testing. Four stages of temperature evolution were observed: (1) an initial increase in temperature followed by a temperature decrease to (2) an equilibrium (steady-state) temperature, which held until (3) a rapid temperature increase and (4) a temperature drop after specimen failure. Modeling techniques were used to predict the temperature profile from the stress and strain data. Additionally, using the temperature recorded by the IR camera, the stress and strain results in the sample were back-calculated. Relationships between the temperature evolution and geometry effects of round-bar specimens were explored. This research is supported by the National Science Foundation Integrative Graduate Education and Research Training (IGERT) program with Drs. W. Jennings and L. Goldberg as contract monitors, and Haynes International, Inc.

**4:20 PM**

**Fatigue-Induced Shape Change in Marble**: *Ulf Lindborg*[1]; [1]National Heritage Board, Conservation Dept., PO Box 5405, Stockholm 114 84 Sweden

Marble sometimes displays a strange, gradual change of shape during normal outdoor exposure without a mechanical load. This is a severe problem for many marble-clad buildings where facade panels bow visibly after 5-20 years and require replacement. The present work links marble bowing to a fatigue process, caused by stresses from variations in temperature and humidity. Fatigue results in the formation of microcracks providing local volume expansion causing the observed shape change. Laboratory experiments prove that combined moisture-and-temperature cycling aggravates fatigue. A model is put forward, focussing on crack closure during the compressive part of a cycle. The presence of water in a microcrack delays or prevents closure, thus providing a set of microcracks that are able to grow during a subsequent tensile half-cycle. The model also explains that shape changes are observed only for certain marbles and limestones but not for granites, high-porosity limestones or sandstones.

**4:45 PM**

**Short and Long Fatigue Crack Growth in Ti-6242: A Comparison of Dwell and Cyclic Fatigue Crack Growth**: *W. O. Soboyejo*[1]; W. Shen[1]; R. Anderson[1]; D. F. McBagonlurinuuri[1]; E. Akpan[1]; C. Mercer[1]; [1]Princeton University, Princeton Matls. Inst. & The Dept. of Mechl. & Aeros. Eng., Olden St., Princeton, NJ 08544 USA

This paper presents the results of a combined experimental and computational study of short and long fatigue crack growth in Ti-6242. The underlying mechanisms of sub-surface fatigue crack nucleation and short crack propagation are elucidated by benchmarking techniques. The crack/microstructure interactions associated with short crack growth are also explored using edge-cracked specimens. Following the initial focus on short crack growth, a comparison between long fatigue crack growth and long dwell crack growth is presented. Dwell crack growth is shown to be associated with a higher incidence of static fracture modes. The differences between the measured dwell and fatigue crack growth rates are also presented. The implications of the results are then discussed for the prediction of fatigue and dwell crack growth.

**5:10 PM**

**A New Fatigue Life Extension Method Using Nano-Sized Martensites in Austenitic Stainless Steels**: *Yakichi Higo*[1]; Tomonari Inamura[1]; Masayuki Shimojo[1]; Kazuki Takashima[1]; [1]Tokyo Institute of Technology, P&I Lab., 4259 Nagatsuta-cho, Midori-ku, Yokohama, Kanagawa 226-8503 Japan

A new fatigue life extension method is proposed in this study. Increase in tensile strength is not always effective on fatigue life extension. Fatigue of metallic materials is mainly attributed to the accumulation of irreversible motion of dislocations. Pinning dislocations at their intersection using nano-sized obstacles is considered to be effective for fatigue strengthening, as the motion of pinned dislocations would be bowing and the accumulation of fatigue damage should be retarded. In austenitic stainless steels, nano-sized martensite particles are considered to be formed at intersections of dislocations when the temperature is controlled at a certain cryogenic temperature above Ms. These nano-sized martensites are expected to act as pinning centers of dislocations. Austenitic stainless steels were cryogenically treated at 195K to form nano-sized martensites and fatigue life tests were conducted. As results, effectiveness of this method on fatigue life extension was confirmed in 304 and 316-type austenitic stainless steels.

**5:35 PM**

**The Effect of Banded Structure on the Fracture Behavior of a Low Carbon 10% Ni Steel**: *Xian Jie Zhang*[1]; Ernest J. Czyryca[1]; [1]Naval Surface Warfare Center, Carderock Div., 9500 MacArthur Blvd., W. Bethesda, MD 20817 USA

Banded structure is a common inhomogeneity in steel plates. It originates from dendritic solidification, develops in hot or cold working during plate processing, and affects the final microstructure and mechanical properties, especially fracture behavior, of high strength steels. This type of inhomogeneity is persistent and can not be totally eliminated even after a prolonged homogenizing heat treatment. In this study, the banded structure of a low carbon 10% Ni steel displayed unique fracture characteristics, where alternating bands showed a pattern of two different fracture modes, brittle and ductile, on the fracture surfaces of Charpy V-notch impact samples with a broad range of heat treatments and testing temperatures. Analytical techniques, especially stereo-section fractography (SSF1), were employed to relate this fracture pattern to the banded structure of the steel. A direct correspondence between the banded fracture surface characteristics and the microstructural banding underlying the fracture surface was established. The effect of the banded structure on the fracture mode and the ductile-to-brittle transition behavior of the low carbon 10% Ni steel are discussed. 1. X. J. Zhang, R. W. Armstrong, and G. R. Irwin, “Stereo (SEM) Section Fractography of Isolated Cleavage Regions in Nuclear Vessel Steels,” Metall. Trans., 20A (1989), pp. 2862-2866.

## Microstructural Design of Advanced Materials: A Symposium in Honor of Professor Gareth Thomas: Mechanical Properties - II

*Sponsored by:* Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
*Program Organizers:* Marc Andre Meyers, Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093-0411 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

Wednesday PM — Room: 214
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Subra Suresh, Massachusetts Institute of Technology, Matls. Sci. & Eng., Cambridge, MA 02139 USA

**2:00 PM**
**On the Development of ABC-Silicon Carbide: Processing, Microstructure, Toughness, Creep and Fatigue**: *R. O. Ritchie*[1]; L. C. DeJonghe[1]; X. F. Zhang[1]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., Berkeley, CA 94720 USA

The development of a high-toughness SiC is described, based on the notion of in situ toughening through the growth of plate-like, elongated grains in the presence of Al, B and C sintering additives during hot pressing. The fully controllable microstructure and phase composition allows for development of optimized recipes in response to different mechanical requirements. Although the use of commercial SiC has been severely limited to date by its very poor fracture resistance, ABC silicon carbide is shown to have significantly higher fracture toughness at ambient temperatures. Moreover, due to structural changes in the microstructure above approximately 1100ºC, associated with the in situ crystallization on amorphous grain boundary films, this material combines such good lower temperature toughness with excellent elevated temperature strength and creep resistance. The mechanisms of such crack growth resistance, associated with fracture, creep and fatigue crack growth, are discussed in light of the microstructural evolution.

**2:30 PM**
**The Onset of Twinning in Plastic Deformation and Displacive Transformations**: *Marc Andre Meyers*[1]; *Matthew S. Schneider*[1]; Otmar Voehringer[2]; [1]University of California-San Diego, Dept. of MAE, La Jolla, CA 92093 USA; [2]University of Karlsruhe, Inst. for Matls. Rsrch., Karlsruhe, Germany

It was first shown by G. Thomas (Acta Met.13,1965,1211; Met. Trans. 2,1971,2373) that slip and twinning are competing deformation mechanisms and that they have a profound effect on the mechanical properties of martensitic steels. A constitutive description will be presented that quantitatively predicts the criterion postulated by Thomas. The temperature and strain-rate sensitivities of slip are much higher than those for twinning, rendering twinning a favored deformation mechanism at low temperatures and high strain rates. Constitutive equations for slip and twinning are presented and applied to a variety of metals and alloys. They enable the prediction of twinning initiation domains. This constitutive description is extended to the martensitic transformation in steels; the lath/plate morphology change is interpreted in terms of the two modes of deformation. The effects of material (stacking-fault energy, grain size, composition) as well as external (temperature, strain rate) parameters are successfully incorporated. The constitutive description is applied to the shock compression regime, where the shock front thickness (and, consequently, strain rate) is related to the peak pressure by the Swegle-Grady relationship. Predictions will be compared to seminal shock loading work by Johari and Thomas (Acta Met., 12,1964,1153) and Nolder and Thomas (Acta Met., 12, 1964,227).

**3:00 PM**
**Crystal Imperfections Seen by X-Ray Diffraction Topography**: *Ronald W. Armstrong*[1]; [1]AFRL/MNME, 2306 Perimeter Rd., Eglin AFB, FL 32542-5910 USA

Microscopic-to-macroscopic crystal strains are directly imaged in X-ray diffraction topographs that are matched at essentially unit magnification on a point-by-point basis with real crystal sizes. The several relatively low-imperfection density transmission and reflection techniques produce sensitive diffraction contrast results that overlap at a narrow size scale with higher defect density observations made with the transmission electron microscope, as exemplified in the latter case by the many defect characterization/property measurement systems investigated by Professor Gareth Thomas and colleagues and students. For the X-ray case, individual dislocations are favorably studied in molecular (energetic) crystals and near-perfect covalent (semiconductor) crystals. Otherwise, cumulative dislocation interactions within subgrain boundaries and local plastic deformation zones, produced by dislocation pile-ups or at microindentations, are favorably studied in all crystals, and in special instances, in polycrystal grains.

**3:30 PM Break**

**3:45 PM**
**Structural Armor by Design**: *Kenneth S. Vecchio*[1]; [1]University of California-San Diego, Dept. of Mech. & Aeros. Eng., 9500 Gilman Dr., MC-0411, La Jolla, CA 92093-0411 USA

Metal-Intermetallic Laminate (MIL) composites based on Ti-Al3Ti can be designed for structural use to optimize the unique properties and benefits of the constituent components, resulting in materials which have the high strength and stiffness of the intermetallic phase and the high toughness of the metal. There is excellent microstructural control and microstructural variability as layer thickness can be chosen with great precision, and the foil stacking sequence and foil materials can be varied within the thickness to yield graded structures and microstructures optimized for specific applications. These materials are fabricated layer-by-layer, it is possible to incorporate additional microstructural features within each layer, enhancing the applicability of these composites, including: embedded thermal and electrical insulators; embedded metal wires for strain and damage sensors; through-thickness wires and tubes for surface sensing, while simultaneously providing through-thickness strengthening. Processing strategies have been developed to fabricate MIL composites with embedded particle damping.

**4:15 PM**
**Taylor Hardening at Elevated Temperatures**: *M. E. Kassner*[1]; [1]Oregon State University, Dept. of Mechl. Eng., Rogers Hall, Corvallis, OR 97331-6001 USA

Previous work on aluminum and stainless steel show that the density of dislocations within the subgrain interior influences the flow stress at a given strain rate and temperature. The hardening in stainless stress is consistent with the Taylor relation if a linear superposition of ‘lattice’ hardening (or the stress necessary to cause dislocation motion in the absence of a dislocation substructure) and the dislocation hardening is assumed. It is shown that constant stress creep behavior where the total dislocation decreases during primary (hardening stage) creep, is consistent with Taylor hardening. The temperature dependence of the alpha ‘constant’ in the Taylor equation is discussed.

**4:45 PM**
**Nanostructures and Their Effects on Titanium Processed by ECAP and Cold Rolling**: *Yuntian T. Zhu*[1]; Jianyu Huang[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanostructured Ti was produced by warm Equal Channel Angular Pressing (ECAP) followed by cold rolling. In this talk, we’ll present the defect structures, including grain and subgrain structures, dislocation cells, dislocation distributions, grain boundaries, and the hierarchy of these structural features, which were observed using transmission electron microscopy (TEM). The effects of these nanostructures on mechanical behaviors of Ti will be discussed.

**5:05 PM**
**Microstructural Design of 7X50 Aluminum Alloys for Fracture and Fatigue**: *Fernand D.S. Marquis*[1]; [1]South Dakota School of Mines and Technology, Matls. & Metallurgl. Eng., 501 E. St. Joseph St., Rapid City, SD 57701 USA

This paper focuses on the microstructural design of the 7050 and 7150 aluminum alloys for the control of the recrystallization, fracture, fatigue crack initiation and fatigue crack growth of these alloys. An investigation of the evolution of the microstructure during primary, secondary and intermediate thermo-mechanical processing has been carried out. The study of the recrystallization behavior, the grain morphology and the sub-grain morphology has been carried out. Other microstructural features such as the morphology of the constituent particles, as the formation of primary hydrogen porosity and its evolution during intermediate thermo-mechanical processing have also been studied. The paper discusses the effect of these microstructural features on the fracture and fatigue behavior of these alloys.

**5:25 PM**
**Composition Dependence of Elastic Constants of Single Phase Cubic Alloys**: *Craig S. Hartley*[1]; [1]Air Force Research Laboratory, AFOSR/NA, 801 N. Randolph St., Rm. 732, Arlington, VA 22203 USA

A computationally efficient, physics-based technique is described for calculating the composition dependence of single crystal elastic constants of disordered, single-phase alloys with cubic Bravais lattices. Alloys are modeled as virtual crystals in which the energy of representative atom pairs is approximated by a virtual potential constructed from the pair potentials of component pairs based on a quasi-chemical approximation. Using the method of long waves, second-order elastic constants are calculated from first and second neighbor axisymmetric force constants obtained from the virtual potential. Since only elastic constants are modeled, the form of the potential employed contains only parameters that describe the slope and curvature in the vicinity of the first and second nearest neighbors. Examples are presented for several binary alloy systems differing in solubility characteristics and crystal structures of the pure solutes.

## Microstructure/Property Relationships in Alpha/Beta Titanium Alloys: Chemistry and Microstructure Effects on Slip

*Sponsored by:* Structural Materials Division, SMD-Titanium Committee
*Program Organizers:* Patrick L. Martin, US Air Force, AFRL/MLLM, WPAFB, OH 45433 USA; James Cotton, The Boeing Company, Seattle, WA 98124-2499 USA

Wednesday PM — Room: 220/221
October 9, 2002 — Location: Columbus Convention Center

*Session Chairs:* M. Ashraf Imam, Naval Research Laboratory, Washington, DC 20375-5000 USA; Jim D. Cotton, The Boeing Company, Seattle, WA 98124 USA

**2:00 PM Invited**
**The Structure and Slip Transmission Properties of Alpha/Beta Interfaces in Single Colony Titanium Alloy Crystals**: Joseph Tatalovich[1]; Michael F. Savage[2]; Gopal Babu Viswanathan[1]; Michael D. Uchic[3]; *Michael J. Mills*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 478 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]NIST, Matls. Sci. & Eng. Lab., 100 Bureau Dr., Gaithersburgh, MD 20899-8553 USA; [3]Air Force Research Laboratory, MLLM, 2230 Tenth St., WPAFB, OH 45433-7817 USA

The Widmanstatten colony is a common microstructural constituent in commercial titanium alloys after slow cooling from above the beta transus temperature. The Burgers orientation relationship is typically observed between the alpha and beta phases, which is thought to promote easy slip transfer through the beta laths, so that the relevant microstructural length scale is the colony size rather than the lath spacing. In this investigation, the structure of the alpha/beta interfaces for several "near-alpha" alloys, including the aeroengine alloy Ti-6242, has been determined using diffraction contrast and high resolution TEM. In addition, the processes of slip transmission, for both basal and prism slip planes in the alpha phase, has been studied in detail via TEM investigation of single colony crystals that have been oriented and deformed to activate particular slip systems in the alpha matrix. The strong anisotropy of the constant strain rate and creep properties of these colony crystals will be rationalized in terms of the observed mechanisms of slip transmission.

**2:40 PM**
**Atomic Mobility and Growth/Dissolution of Globular Alpha in the Ti-Al-V System**: *Qing Chen*[1]; Kaisheng Wu[1]; Yunzhi Wang[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Computer simulation of diffusion-controlled microstructure evolution needs not only thermodynamic database but also atomic mobility values. The experimental data on the diffusivities of Ti, Al, and V in the bcc (beta) and hcp (alpha) phases in the Ti-Al-V and its constituent binary systems is assessed by means of the CALPHAD procedure using DICTRA. A set of atomic mobility parameters consistent with most of experimental information is obtained. With available thermodynamic database and assessed atomic mobility data, the growth/dissolution rates of globular alpha in the Ti-6Al-4V alloy are predicted and compared using both DICTRA and the phase field method. The work is supported by AFRL under MAI contract.

**3:10 PM**
**Strain Rate Recovery During Low Temperature Creep in an Alpha Titanium Alloy**: *Matthew C. Brandes*[1]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Recently, titanium-aluminum alloys were discovered to experience significant recovery of strain hardening during low-stress ambient-temperature creep. This phenomenon occurs in specimens that had been loaded in compression, crept to some level of plastic strain, unloaded and allowed to rest at room temperature for a period of time, and reloaded to the initial stress level. The rate of creep immediately following reloading is found to be up to several orders of magnitude greater than that preceding unloading. In this study a Ti-6 wt % Al alloy has been used to represent a model, single-phase alpha system. The strain rate recovery has been measured as a function of both annealing temperatures, between 25 and 150°C, and annealing time. Analyses of surface slip traces were used to monitor the microstructural locations associated with recovery.

**3:40 PM Break**

**3:55 PM**
**High Temperature Creep Behavior of Binary Ti-6Al Alloy with Trace Amounts of Ni**: *Junho Moon*[1]; Gopal B. Viswanathan[1]; Karthikeyan Subramanian[1]; Robert W. Hayes[2]; S. P. Fox[3]; Michael J. Mills[1]; [1]The Ohio State University, The Dept. of Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]Metals Technology, Inc., 19801 Nordhoff, Northridge, CA 91324 USA; [3]Timet, Inc., N. America Techl. Lab., W. Lake Mead & Atlantic Ave., Henderson, NV 89015 USA

The high temperature creep behavior of two binary equiaxed Ti-6Al alloys with varying trace amounts of Ni (0.002 and 0.005 wt%) was studied. Creep tests were performed in uniaxial compression over the temperature range 538 to 621°C at applied stress levels ranging from 131 to 279MPa. Stress and temperature jump tests were performed to obtain the stress exponents and the creep activation energy. The results show that increased amounts of Ni increased the minimum creep rates at all stress levels. Stress exponent values of ~5.0 was obtained for both samples. Detailed TEM analysis of the deformation structure was performed on samples crept monotonically at 200MPa up to 1% plastic strain. The results are explained with reference to the recently reported trends associated with lattice self-diffusion in alpha-titanium in the presence of fast diffusing impurities. Analysis of the dislocations structures along with the possible creep mechanism is presented.

**4:25 PM**
**RX2™ Titanium: A New Damage-Tolerant Microstructure for Alpha-Beta Titanium Alloys**: *Sami El-Soudani*[1]; [1]The Boeing Company, C078-0533, Long Beach, CA, USA

RX2™ titanium is the improved alpha-beta titanium alloy unique microstructure produced by a heat treat process that substantially enhances both mechanical properties and fracture resistance of mill-processed alpha+beta titanium alloys. This innovative method of heat treating has demonstrated that the alpha+beta microstructure is transformed into an alpha+alpha2+beta microstructure, preferably containing no silicides. Such improvements have been demonstrated on two commercial titanium alloys, namely Ti-6242S and Ti-62222, both in sheet and extruded product forms. The optimization steps involve a sequence of thermomechanical and special solution and aging steps, causing improvement in the mechanical properties including: (a) tensile strength; (b) fracture toughness; (c) creep resistance; (d) elastic stiffness; (e) thermal stability; (f) resistance to hydrogen embrittlement; (g) fatigue; and (h) resistance to cryogenic temperature embrittlement. The microstructure contains equiaxed alpha phase strengthened with alpha2 precipitates coexisting with lamellar alpha-beta phase. The alpha2 precipitates are confined totally to the equiaxed primary alpha phase. This process and the resulting microstructure/property relationships will be described.

**4:55 PM**
**The Formation of Uniform Fine-Grain Microstructures in Two-Phase Ti-6Al-4V Titanium Alloy During Hot and Warm Deformation**: *S. V. Zherebtsov*[1]; G. A. Salishchev[1]; R. M. Galeyev[1]; O. R. Valiakhmetov[1]; [1]Russian Academy of Science, Inst. for Metals Superplasticity Problems, Khalturina str. 39, Ufa 450001 Russia

Two-phase titanium alloys with uniform fine-grained microstructures exhibit uniform mechanical properties. The effects of the initial microstructure, temperature, and rate of deformation on the formation of uniform fine-grained microstructures in Ti-6Al-4V will be discussed. During beta deformation and following beta anneals both continuous and discontinuous recrystallization is observed. Discontinuous recrystallization leads to the formation of large beta grains having similar crystallographic orientation. These areas cause non-uniform subsequent alpha+beta deformation and preservation of microtexture, that leads to high ultrasonic noise. Deformation temperature defines many aspects of the final microstructure and its effect will be discussed. Dynamic continuous recrystallization takes place over a wide temperature range within the alpha+beta region. The conversion of alpha/beta phase-boundaries from a semi-coherent state into an incoherent one controls the transformation of lamellar microstructure into a globular microstructure. Production of a uniform fine-grained microstructure with minimal microtexture is possible using the correct combination of alpha+beta deformation and recrystallization anneals.

## Modeling the Performance of Engineering Structural Materials: Composites & Processing and Resulting Properties

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee

*Program Organizers:* Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

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| Wednesday PM | Room: 215 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chair:* Meslet Al-Hajri, The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA; T. S. Srivatsan, The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

**2:00 PM**

**Computer Simulation of Dislocation Motion in Fine-Scale Multilayered Composites**: *Qizhen Li*[1]; Evan Andrew Sperling[1]; Peter M. Anderson[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

A classic method of strengthening materials to plastic deformation is to refine the microstructure, so that barriers to dislocation motion occur more frequently. A limiting example of this approach is to decrease the individual layer thickness in multilayered composites to several nanometers. We simulate dislocation motion in such materials with a cellular automaton approach, in which slip planes are discretized into triangular patches that slip according to an energetic driving force. The results show that composite strength can be increased by decreasing the layer thickness, so that dislocations are confined to smaller volumes of material. However, a critical layer thickness arises, below which the composite cannot be strengthened significantly by further refinement. The simulations help one to visualize the confinement process. They also help define the role of lattice parameter mismatch and interfacial dislocation content in controlling the critical layer thickness and associated plastic strength of the composite.

**2:25 PM**

**Interfacial Sliding in Multi-Material Systems and its Mechanism**: *Indranath Dutta*[1]; [1]Naval Postgraduate School, Ctr. for Matls. Sci. & Eng., Dept. of Mechl. Eng., 700 Dyer Rd., Monterey, CA 93943 USA

Large shear stresses may develop at interfaces between dissimilar materials during thermo-mechanical excursions when there is a significant difference in the coefficient of thermal expansion (CTE) between them. Most typically, these shear stresses are confined to the extremities of one of the components, such as fiber-ends in a composite, or the edges of a thin film on a substrate. However, when the dimensions of the phases is small, these shear stresses may prevail over a large area fraction of the total interface. Under appropriate thermal-mechanical conditions, the interface may slide via diffusional processes, thereby accommodating relative dimensional changes between the phases. This phenomenon has been noted in metal-matrix composites, and in thin-film interconnects on Si. In this paper, we will present experimental and modeling approaches for deriving the mechanism of this phenomenon, and to study its effect in (1) continuous fiber reinforced metal-matrix composites and (2) high density interconnect structures on microelectronic devices during thermal cycling. Supported by NSF grant no. DMR 0075281 and NCCOSC Graduate Student Fellowship.

**2:50 PM**

**The Strain Amplitude Controlled Fatigue Response of 2009 Aluminum Alloy–SiC Particulate Metal-Matrix Composite**: *Meslet Al-Hajri*[1]; T. S. Srivatsan[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

In this technical presentation the influence of discontinuous ceramic particulate reinforcements on low cycle fatigue response and final fracture behavior of 2009 aluminum alloy metal-matrix composites will be highlighted. The cyclic stress amplitude-controlled fatigue properties and fracture characteristics of the 2009 aluminum alloy metal matrix discontinuously-reinforced with silicon carbide particulates will be highlighted for a range of cyclic strain amplitudes and at two different temperatures. The influence of test temperature and strain amplitude on stress response versus fatigue life characteristics will be rationalized. Also an attempt will be made to elucidate the intrinsic mechanisms governing stress response and fatigue fracture characteristics in light of the competing and mutually interactive influences of composite microstructural effects, deformation characteristics of the composite constituents, cyclic strain amplitude, and test temperature.

**3:15 PM**

**Nanoindentation of Alumina-Chrome Carbide Composite**: *Maksim V. Kireitseu*[1]; Sergey G. Yerakhavets[1]; [1]Institute of Machine Reliability, Mech. of Composites, Lesnoe 19-62, Minsk 223052 Belarus

The work revealed the possibility to improve physical, mechanical properties of alumina-based ceramic composite by CrC-based thin coatings produced by the metalorganic chemical vapor deposition. The CrC coatings heal various surface defects of the alumina that strengths and toughs the composite. It is found to be high microhardness and fracture resistance regarding initiation and propagation of Palmquist cracks during Vickers indentation. The nanoindentation hardness and abrasive wear resistance by dimple grinding test of the coatings were compared between the sample series and related to the microstructure, phase composition, and residual stress obtained by transmission electron microscopy, x-ray diffraction, and substrate curvature technique, respectively. All films exhibited a dense and columnar microstructure. Nanoindentation test showed high hardness for CrC alumina multilayer and homogeneous alumina in the range of 20-26 GPa. The absence of superlattice hardening in the polycrystalline alumina nanostructured multilayer is suggested to be due to micro cracking at pores and grain boundaries under the indenter tip. CrC layer filled the defects and pores might enhance crack propagation under overloaded indenter. For both series, fracture toughness was lower for the multilayer than the corresponding homogeneous films. The abrasive wear resistance of films between sample series showed a negative correlation with grain size. Composite exhibits higher scratch wear resistance compared to the alumina.

**3:40 PM**

**Hard Oxide Ceramics Modified by Ultra Dispersed Diamonds**: *Maksim V. Kireitseu*[1]; [1]Institute of Machine Reliability, Mech. of Composites, Lesnoe 19-62, Minsk 223052 Belarus

Alumina-diamonds composite coatings have been investigated by in suite experiments that reveal microstructure, wear rate and fatigue behavior of the coatings. A model to predict behavior of the coating under loading has been developed. Failure mechanics of the coating and some technological aspects to be applied have been discussed. Alumina-based hard coatings are very popular in industrial engineering as result of its valuable mechanical properties to be used for quality and performance of different machines. However, basically ductile aluminium substrate used as the base for alumina coatings produced by micro arc oxidizing or electroplating technologies significantly decrease load rating, fatigue life and fracture resistance of the coatings. Previously it has been shown that alumina layer might be strengthen by different hard particles of metals like chrome carbide, SiN, SiC, etc. that fill the pores and surface defects of ceramic coatings. However, obtained results have shown that load rating and wear rate of new composites are improved. To improve fracture resistance, load rating and overall strength of alumina coatings produced on aluminum or its alloy by micro arc oxidizing technology they have been hardened by particles of ultra dispersed diamonds. An average size of clusters was 60 angstroms. The particles of diamonds were dislocated into pores of alumna as well as in matrix structure of the top hard layer. The diamonds were synthesized in strong non-equilibrium conditions of a detonation surge. Form of diamonds was like isometric fragments. To compare an effect of other hard particles on mechanical properties of the coatings we have compared hardness and load rating of several samples hardened by diamonds and chrome carbide. Summarizing the experimental results it has been revealed that (a) structure of the coating consists of up to 70% hard alumina phases and conglomerated particles of diamonds that might exhibit multi-layered matrix of the composite. The structure of the sub-layers consists of alpha and gamma phases of alumna glasses phases of metal and hard particles of diamonds or CrC. In general, the alumina phases envelop hard particles in strong space (like shell) that presents main matrix of the coating. (b) Particles might diffuse in the alumina matrix and in the soft substrate. (c) Hardness of the coating improves up to 25 GPa. In result, it gives as low as 0.00004-0.00006 mm/km wear rate at 15-20 GPa contact pressure. Especially, it has to be stressed improved wear behavior of the coatings under dry friction because the diamonds can be a lubricant. (d) Fatigue behavior investigate by indentation technique have revealed improved fatigue toughness in contrast to the alumina hardened by CrC. Crack arrested regions appear in the coating however, diamonds deflects the cracks effecting on its length and direction. (e) Rheological model including revealed fatigue related aspects of strength degradation has been developed to predict mechanical behavior of the coating under static loading.

**4:05 PM Break**

**4:20 PM**
**Simulation of Microstructures and Mechanical Properties of a Cast 319 Aluminum Alloy Component**: *Mei Li*[1]; Ravi Vijayaraghavan[1]; John Allison[1]; Xinyan Yan[2]; [1]Ford Motor Company, Matls. Sci. Dept., Dearborn, MI 48121-2053 USA; [2]University of Wisconsin, Matls. Sci. & Eng. Dept., Madison, WI, USA

Cast aluminum alloys, particularly 319-type, are increasingly being utilized by automotive industry for manufacturing engine blocks and cylinder heads to reduce vehicle weight and consequently increase fuel economy and reduce emissions. Predictions of microstructure and the corresponding mechanical properties are important for estimating in-service performance of these components and for simultaneous optimization of both the component geometry and the casting/heat treatment process. This talk describes the simulation of microstructure evolution during casting and subsequent heat treatment processes and yield strength after heat treatment of a typical complex component. A micromodel is used to predict the fraction of as cast $Al_2Cu$ phase and secondary dendrite arm spacing (SDAS). A solution treatment model is developed to calculate the fraction of dissolved $Al_2Cu$. This result is then used in conjunction with a precipitation model to predict amount of $Al_2Cu$ precipitated in the aging process and the corresponding yield strength. Finally, the predicted yield strength in a complex engine block is compared with experimental data.

**4:45 PM**
**An Assessment of In-Service Stress Relaxation of a Work-Hardened Al-Mg Alloy**: *Lihua Zhu*[1]; Armand J. Beaudoin[1]; Hamid Garmestani[2]; Stuart R. MacEwen[3]; [1]University of Illinois, Mechl. & Industl. Eng., 1206 W. Green St., MC-244, Urbana, IL 61801 USA; [2]FAMU-FSU College of Engineering, Dept. of Mechl. Eng., Ctr. for Matls. Rsrch. & Tech., Ctr. for Nonlinear Nonequilibrium Aeroscis., Rm. 229, 2525 Pottsdamer Rd., Tallahassee, FL 32310-6046 USA; [3]Alcan International, Ltd., 945 Princess St., Kingston, Ontario K7L 5L9 Canada

Time-dependent relaxation processes continue after forming of sheet metal components. Mechanical properties and even the shape of the part may evolve with time. Beverage can ends, made of an aluminum-magnesium alloy, provide one example of relaxation in a metal product. Ends are manufactured in a series of forming operations, and the industry standard requires the end buckle pressure to meet a minimum buckle strength. It has been established that buckle pressure decreases with time in service. In this work, we outline a simple bending test to study relaxation at stress levels well below the usual 0.2% offset yield stress. The evolution of stress and development of plastic strain with time are assessed through a simple analysis of springback. The microplastic processes that lead to permanent deformation of the bend beam are characterized by a model developed by Garmestani and Hart.

**5:10 PM**
**Modeling the Evolution of Strength During Wire Drawing**: *Donald Lesuer*[1]; Chol Syn[1]; Oleg Sherby[2]; [1]Lawrence Livermore National Laboratory, L-175, Livermore, CA 94551 USA; [2]Stanford University, Dept. of Matls. Sci., Stanford, CA 94305 USA

Extensive plastic deformation during wire drawing is commonly used to produce steel wires with very high strengths. Typically these steels are eutectoid and hypereutectoid steels and drawing strains up to 4 are used during processing. The resulting materials can have tensile strengths in excess of 4000 MPa. The evolution of microstructure and the strengthening mechanisms resulting from wire drawing have been studied for eutectoid and hypereutectoid steels. Strength has been shown to be a function of pearlite colony size, interlamellar spacing and the size of the stable dislocation cells that are produced during wire drawing. The results have been used to model the evolution of strength during wire drawing. Model predictions for the evolution of tensile strength with drawing strain show excellent agreement with data derived from a number of eutectoid and hyperectectoid steels as a function of drawing strain. Work performed under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48.

**5:35 PM**
**Numerical Simulation of the Temperature and Electric Fields in Electric Current Aided Sintering**: *Jing Zhang*[1]; Antonios Zavaliangos[1]; Martin Kraemer[2]; Joanna Groza[2]; [1]Drexel University, Matls. Eng. Dept., 3141 Chestnut St., Philadelphia, PA 19104 USA; [2]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA

Sintering aided by pulsed electrical current offers the advantage of accelerated densification in a variety of powder. In this work we employ numerical simulations in order to understand the distribution of current into the punch/die/compact assembly, and scale-up. A thermoelectrical model is employed for the conduct in punches and the die with thermal and electrical properties that vary with temperature. For the sintering compact, a first order sintering model is employed to describe the densification and the associated changes in the macroscopic resistivity. Parametric studies on the contact resistance between the punches and the die show, that for a low contact resistance, a substantial amount of the current is diverted around the specimen, and the specimen is heated indirectly through conduction from the heating punches. For low contact resistance or with increasing conductivity of the sintering compact, direct heating of the specimen is achieved.

**6:00 PM**
**Electromagnetic Sensors for Monitoring Phase Transformation in Steels–Experimental Results and Modelling Simulations**: *Mayorkinos Philip Papaelias*[1]; Claire L. Davis[1]; Martin Strangwood[1]; Anthony J. Peyton[2]; [1]University of Birmingham, Sch. of Metall. & Matls., Elm Rd., Edgbaston, Birmingham, W. Midlands B15 2TT UK; [2]Lancaster University, Eng. Dept., Lancaster LA1 4YR UK

Obtaining the derived mechanical properties (particularly strength) in dual and triple strip steels depends upon controlling the exact phase balance (i.e. ferrite and pearlite/bainite/martensite). This is achieved by controlling the cooling processes after hot rolling. Optical pyrometers are currently employed by steel manufacturers to monitor temperature of the strip, however their accuracy is affected by factors such as water droplets and dust which are present in the run-out table area. Consequently alternative techniques (X-rays, ultrasonic and electromagnetic sensors) for monitoring phase transformation are being investigated. The technique presented in this paper is based on the magnetic properties of the phases present in steel. Austenite is paramagnetic whilst ferrite and pearlite are ferromagnetic below the Curie temperature, which is around 770°C. Hence, electromagnetic sensors are capable of detecting the changes in the magnetic properties of steel as it cools down. Results are presented for a medium carbon steel (C 0.45 wt %), which during continuous cooling transforms below 770°C, in the form of impedance-temperature diagrams. It is shown that the electromagnetic sensor can successfully detect the phase transformation as it progresses and can differentiate between the ferrite and pearlite transformation. However, it is also shown that the change in impedance is not linearly proportional to the change in ferrite fraction in the steel and is effected by the prior austenite grain size. Finite element simulations conducted to determine the behaviour of the electromagnetic field during transformation showed that the electromagnetic signal is strongly affected by the distribution of the ferromagnetic phase parallel to the magnetic lines of flux.

## Opportunities and Issues in Homeland Security

*Sponsored by:* TMS, Public & Governmental Affairs Committee
*Program Organizer:* Toni Marechaux, National Research Council, National Materials Advisory Board, Washington, DC 20418 USA

Wednesday PM — Room: D131-132
October 9, 2002 — Location: Columbus Convention Center

*Session Chair:* Toni Marechaux, National Academies of Science and Engineering, National Materials Advisory Board, Washington, DC 20418 USA

**2:00 PM Opening Remarks**

**2:10 PM**
**Fifty Years of Defending the Nation at Home and Abroad**: *A. D. Romig*[1]; John C. Cummings[1]; [1]Sandia National Laboratories, MS 0513, Albuquerque, NM 87185 USA

Since WW II, US Department of Energy laboratories have played key roles in defending our nation. The National Nuclear Security Administration (NNSA) laboratories (Sandia, Los Alamos, and Lawrence Livermore National Laboratories) were scientific and technical cornerstones of the US success in prevailing during the Cold War. Recently these labs have been major players in the nation's efforts to combat terrorism and secure our homeland. This lecture will present unclassified activities within the NNSA laboratories, especially Sandia. Laboratory technology in the Afghanistan theater has enabled locating, tracking, and incapacitating Taliban and Al Qaeda facilities and forces. At home, technology has been deployed to detect chem-bio agents. Other technologies have been developed to mitigate these agents in the event of their use. For example, working with the US Postal Service, Sandia deployed technology to decontaminate mail in Washington, DC, and a Sandia-developed foam was used to treat several facilities contaminated with anthrax. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

**2:35 PM**
**Development of the Fortress™ Door–Cockpit Security**: *David R. Williams*[1]; [1]Alcoa, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

Alcoa has been an historic leader in the development and processing of aerospace metallics, primarily aluminum, from the beginning of manned flight through the current generation of commercial and military aircraft and space vehicles. The presentation will provide an overview of how Alcoa has continued to expand its global materials development, concurrent design, test and characterization and advanced manufacturing capabilities through both acquisition and internal growth. The talk will further discuss how Alcoa has brought those capabilities to bear on the critical issue of domestic security, and the new markets and new opportunities that Alcoa is pursuing and developing. The talk will concentrate on a specific example: the development of the Fortress™ door, a reinforced flight deck door for the commercial aerospace industry. It will explore the product development challenges and issues, and the organizational challenges Alcoa faced including: a market in which Alcoa had not participated; new customers; government regulatory requirements; business unit sponsorship; significant time constraints; and, resources and capabilities that were diverse, scattered, and unaccustomed to working together.

**3:00 PM Break**

**3:20 PM**
**Counterterrorism and Materials Research and Engineering: Planning, Challenges, and Opportunities**: *Lewis Sloter*[1]; [1]US Department of Defense, ODUSD(S&T)/Weapons Sys., 1777 N. Kent St., Ste. 9030, Arlington, VA 22209-2110

Current plans and planning efforts associated with counterterrorism in the Federal sector are reviewed with emphasis on the positive role that materials research and engineering can play. Generic challenges associated with inspection and detection of materials and devices and broad opportunities for materials research and engineering contributions are discussed. The need for systems and system engineering approaches to the problem and the challenges that this poses to the materials community are highlighted and general approaches suggested.

**3:45 PM**
**The Role of Universities in Homeland Defense**: Todd Stewart[1]; *Jim Williams*[1]; [1]Ohio State University, 142 Hitchcock Hall, 2070 Neil Ave., Columbus, OH 43210-1178 USA

The events of September 11, 2001 have created a heightened interest and need for innovative ways to protect the people of the US and, concurrently, minimize the degree to which their personal freedom is compromised. Achieving these two objectives simultaneously will require true innovation because new approaches to achieving security will be necessary. This begs the question, how will we meet his challenge and where will this innovation originate? A possibility is that a and major source of non-aligned technical and scientific talent is represented by faculty students in the research universities of this country. Engaging this talent pool in a productive manner to achieve the desired innovation creates some interesting challenges, many of which are non-technical. Included will be handling intellectual properties, restriction on access to sensitive data and information and the multi-national character of the talent pool at all universities. This talk will describe some of these challenges and offer some suggestions regarding ways to proceed.

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## Pb-Free and Pb-Bearing Solders - V

*Sponsored by:* Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
*Program Organizers:* K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA

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| Wednesday PM | Room: 211 |
| October 9, 2002 | Location: Columbus Convention Center |

*Session Chairs:* John William Morris, University of California-Berkeley, Dept. Matls. Sci. & Eng., Berkeley, CA 94720 USA; Indranath Dutta, Naval Postgraduate School, Dept. of Mechl. Eng., Monterey, CA 93943 USA

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**2:00 PM Invited**
**The Influence of Temperature on the Creep of Tin-37 Lead Solders**: *William John Plumbridge*[1]; Colin Richard Gagg[1]; [1]The Open University, Matls. Eng., Walton Hall, Milton Keynes, Buckinghamshire MK7 6AA UK

Service conditions experienced by interconnections are becoming increasingly arduous. Temperature ranges are expanding, and upper limits are expected to exceed 0.9 of the melting temperature in K. To provide information for design and life prediction, the creep behaviour of a Sn-37Pb alloy between 160 and -50°C has been investigated. Significant creep occurs at temperatures as low as -50°C, and the effect of temperature on creep life is profound. For example, at an applied stress of 15MPa, the ratio of rupture times at -50 and 75°C is 2x104. Differences in minimum strain rate are even greater, with a variation of 106 between these temperatures. Creep strengths at 1000h are 33 and 1.5 MPa respectively. The stress sensitivities of rupture time and minimum strain rate diminish slightly with falling temperature. The relationship between applied stress and minimum creep rate behaviour is best described by a power law equation although the steady state domain generally occupies less than 30% of life. Bi-linearity exists in the Monkman-Grant expression, with a transition strain rate at 10-4h-1.

**2:30 PM Invited**
**Microstructural Coarsening and Creep Response of Microelectronic Solder Joints**: *Indranath Dutta*[1]; Sunglak Choi[1]; Neff Anastasio[1]; [1]Naval Postgraduate School, Ctr. for Matls. Sci. & Eng., Dept. of Mechl. Eng., 700 Dyer Rd., Monterey, CA 93943 USA

Microelectronic solder joints are typically exposed to aggressive thermo-mechanical cycling (TMC) conditions during service. During prolonged TMC, the solder microstructures can undergo significant strain-enhanced coarsening. Solder joint life is dictated largely by the creep strain range, which depends on microstructural scale, which in turn depends on both the imposed hydrostatic constraint and the local inelastic strain within the joint. Because of this interdependence of microstructure, and hence mechanical behavior, on the loading conditions, it is imperative to develop (a) an understanding of how local stress/strain states affect coarsening, and (b) how this coarsening affects creep behavior. Both these issues will be addressed in this paper. First, the development of stresses/strains and their effects on microstructural scale in flip-chip joints of eutectic Pb-Sn solders will be addressed. Then, a methodology for the development of a closed-form creep law incorporating microstructural coarsening in Sn-Ag solders will be discussed, along with some experimental creep data.

**3:00 PM**
**Characterization of Creep Deformation of Single Shear Lap Sn-Ag Eutectic and Composite Solder Using Orientation Imaging Microscopy**: *A. U. Telang*[1]; T. R. Bieler[1]; J. G. Lee[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Single shear lap eutectic and composite solder joints with a joint area of 1 $mm^2$ and 80-160 micron thickness on copper substrate were creep deformed at elevated and room temperature to a strain of about 0.3. Orientation Imaging Microscopy (OIM) studies were carried out before and after creep deformation on a number of joints made with eutectic paste and in-situ composite solder. The effect of reinforcements on solidification microstructure is described. Changes in the crystallographic orientation and grain size and morphology due to creep deformation at different temperatures are characterized using OIM in an effort to ascertain the deformation mechanisms and modes of failure that are possible. This work is supported by the National Science Foundation under grant NSF DMR-0081796.

**3:25 PM Break**

**3:45 PM Invited**
**Creep Behavior in Pb-Free Solders**: *John William Morris*[1]; Ho Geon Song[1]; Fay Hua[2]; [1]University of California, Dept. Matl. Sci. & Eng., 555 Evans Hall, Berkeley, CA 94720 USA; [2]Intel Corporation, Matls. Tech., 3065 Bowers Ave., Santa Clara, CA 95054 USA

The Pb-free solders of greatest current interest in microelectronic technology can be divided into two categories: Sn-rich compositions based on Sn-Ag or Sn-Cu binary and ternary near-eutectics, and balanced, multiphase compositions based on Sn-Bi, Sn-In and Sn-Au. The present paper will review the microstructures and properties of these solders with particular emphasis on their creep behavior. Not surprisingly, the creep behavior of the Sn-rich solders is dominated by the Sn phase. These solders exhibit the anomalous thermal behavior of creep in pure Sn itself; the stress exponents for steady-state creep change significantly near room temperature with the consequence that the conventional constitutive equations for steady-state creep are not strictly applicable. The balanced, multiphase compositions show a greater variety of behaviors. The behavior of eutectic Sn-Bi and Sn-In-Cu solders will be specifically discussed.

**4:15 PM Invited**
**Creep and Fatigue of Sintered Solder Joints**: *Dimitar Dostinov*[1]; *Mark A. Palmer*[1]; [1]Kettering University, IMEB, 1700 W. Third Ave., Flint, MI 48504 USA

Sintered lead-free composite solder joints with reduced flux have been prepared by adding copper powder to solder pastes based on Sn-Ag-X eutectics. Prior work has shown that solder joints prepared by this method have shear strengths between 18 and 25 MPa and room temperature creep resistance at 50% of the yield strength. We will continue our investigation of sintered solder joints by examining the elevated temperature creep and isothermal fatigue resistance. This work was supported by the National Science Foundation DMII 0086726.

**4:45 PM**
**Creep of the Lead-Free Solder 'Sn-3.8Ag-0.7Cu' at 75°C**: *Shellene Cooper*[1]; William J. Plumbridge[1]; [1]The Open University, Matls. Eng. Dept., Walton Hall, Milton Keynes, Buckinhamshire MK7 6AA England

Electronic equipment is facing the challenge of both miniaturisation and the need to replace lead in interconnections. In service, interconnections generally fail by thermomechanical fatigue, and this behaviour is strongly affected by the creep process. This paper compares the creep behaviour of Sn-37Pb and Sn-3.8Ag-0.7Cu at 75°C. It is found that the creep resistance of the ternary lead-free alloy is superior to Sn-37Pb. However, the lead-free alloy is less ductile but generally possesses strains to failure above 10% in comparison to the 25 to 50% ductility of Sn-Pb. Over the region tested, the creep behaviour is best described by the Norton power law constitutive equation. The stress exponent for Sn-Ag-Cu is 14 at 75°C and indicates that a dispersion-strengthened mechanism is dominant in the creep process. The activation energy for Sn-3.8Ag-0.7Cu is approximately 120kJ/mol and falls in the region similar to that observed for the self-diffusion of tin.

**5:10 PM**
**Effect of Dwell Time on Thermomechanical Fatigue of Sn-Ag Based Solder Joints**: *J. G. Lee*[1]; A. Telang[1]; K. N. Subramanian[1]; T. R. Bieler[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Thermomechanical fatigue (TMF) due to the mismatch in coefficients of thermal expansion between solder and substrate gradually degrades the mechanical properties of solder joints during service. Solder joints fabricated with eutectic Sn-Ag, and Sn-Ag solders with Cu and/or Ni, were TMF'ed between -15ºC and +150ºC with hold times of 115 min. at high the temperature extreme and 20 min. at the low temperature extreme. Characterization of the surface damage and residual mechanical strength of these solder joints was carried out after 0, 250, 500, 1000, and 1500 TMF cycles. Results obtained from this study were compared with those obtained with longer dwell time at lower temperature extreme so as to gain an understanding of the role of combined effects of temperature and dwell time on the damage accumulation in Sn-Ag based solder joints during TMF. Acknowledgment: Work supported by the National Science Foundation under grant NSF DMR-0081796.

## Characterization and Representation of Material Microstructures in 3-D: Stereology & Quantitative Characterization

*Sponsored by:* Structural Materials Division,
*Program Organizers:* Mike Uchic, Air Force Research Laboratory, Dayton, OH 45433-7817 USA; Dennis M. Dimiduk, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Milo V. Kral, University of Canterbury, Department of Mechanical Engineering, Christchurch, New Zealand; George Spanos, Naval Research Laboratory, Physical Metallurgy Branch, Washington, DC 20375 USA

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*Session Chairs:* Hamish L. Fraser, The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210 USA; Arun M. Gokhale, Georgia Institute of Technology, Matls. Sci. & Eng., Atlanta, GA 30332-0245 USA

**8:30 AM Invited**
**Efficient, Unbiased, and Assumption-Free Estimation of Number Density in Opaque Material Microstructures Using Large-Area Disector (LAD)**: *Arun M. Gokhale*[1]; Asim Tewari[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332-0245 USA

In a three-dimensional microstructure, a test-probe of dimension k (≤3) can yield an unbiased estimate of a d-dimensional microstructural characteristic, if and only if, k ≥ [3 -d]. Estimation of number density essentially involves counting number of points (particle centers) in three-dimensional space (i.e., d = 0), and therefore, a three-dimensional volume probe (k = 3) is essential for unbiased estimation of number density in three-dimensional microstructures. Consequently, in general, the number density (i.e., number of particles/grains/voids/inclusions, etc. per unit volume) is one vital piece of microstructural information that cannot be obtained from measurements performed on random independent two-dimensional metallographic planes. However, a detailed serial sectioning and 3D image reconstruction are NOT required for an unbiased estimate of the number density! The number density can be efficiently estimated in an unbiased and assumption-free manner using a large area dissector (LAD) that essentially consists of two montage serial sections separated by a small distance. This new technique utilizes a combination of digital image analysis and unbiased dissector sampling procedures. The applications of this technique are discussed together will comparison of the experimental number density data obtained by the LAD technique and the detailed serial sectioning involving large number (~100) serial sections.

**9:00 AM Invited**
**Multi-Scalar Analysis of Area Fractions: The MSAAF Technique Applied to Discontinuously-Reinforced Aluminum (DRA) Microstructures**: *Jonathan E. Spowart*[2]; Robert W. Bush[3]; Daniel B. Miracle[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Southern Ohio Council of Higher Education, 3171 Research Blvd., Ste. 141, Dayton, OH 45420 USA

The MSAAF technique has been developed for the characterization of the spatial distribution of second-phase particles in 2-dimensional distributed multi-phase systems. This analysis is based on the combination of simple image processing procedures and is able to probe a wide range of microstructural length scales from the size of the second phase particles up to the size of the specimen itself. In this paper, MSAAF is used to compare the different levels of microstructural heterogeneity found in experimental 6061/SiC/25p DRA materials processed using established P/M techniques. Each material contained F-600 grade SiC particles (median diameter, $d_{50}$ = 13.4 µm) however, the median matrix particle size was varied in a controlled manner, by careful screening of the 6061-Al powder stock. The range of matrix particle sizes that were chosen (26.4 µm, 42.0 µm and 108.6 µm) introduced increasing levels of spatial heterogeneity into the DRA microstructures, as quantified using MSAAF. Also reported are the results of mechanical (tensile) testing carried out on the different DRA materials, both in the as-fabricated and heat-treated (T6) conditions. Excursions from the expected mechanical behavior are related to the levels of microstructural heterogeneity observed in each specimen, and suggestions are made towards a universal microstructural figure-of-merit, based on these results.

**9:30 AM**
**Quantitative Characterization and Representation of Microstructure in α/β Ti Alloys Using Rigorous Stereological Procedures**: *Eunha Lee*[1]; G. Babu Viswanathan[1]; Dhriti Bhattacharyya[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

The mechanical properties of α/β Ti alloys are very sensitive to microstructural features such as volume, size, morphology and distribution of individual phases, which in turn, largely depend on the processing and thermo-mechanical treatment procedures. Therefore, it is imperative that any models attempting predict mechanical properties in these alloys have to take into account the critical microstructural features. Introduction of microstructural variables into quantitative models requires the development of rigorous procedures for the quantification of these variables. Using concepts derived from three-dimensional stereology, a set of such procedures for quantifying specific microstructural features on interest in α/β Ti alloys have been developed and will be discussed in this paper. These features include prior β-grain size, width of grain boundary α, primary equiaxed α, primary Widmanstätten α, scale and orientation of secondary α, volume fractions of α and β, colony size factor, β-rib size, texture and local orientations. Comparisons have been made between results obtained using previously accepted methods of microstructural quantification and the new stereological procedures, and these will be discussed.

**9:55 AM Break**

**10:15 AM**
**Microstructural Anisotropy and Grain Strain**: *Craig S. Hartley*[1]; [1]Air Force Research Laboratory, AFOSR, Arlington, VA 22203 USA

The quantitative description of anisotropic microstructures is described in terms of a tensor quantity that expresses the spatial variation of the mean linear intercept with the direction of the test line. This representation can be expressed as a second rank tensor, the Microstructural Anisotropy Tensor, whose principal axes define directions along which the mean linear intercepts are extrema. In deformed, single phase metals, the principal axes of the deformed microstructure are aligned with the principal axes of deformation, and the change in principal values can be used as a measure of local plastic strain. This procedure is demonstrated for rolling, extrusion and simple tension tests. The definition of "grain strain" in terms of the changes in the principal axes with deformation is developed and derived and the application to analysis of deformed plastically anisotropic metals is discussed.

**10:40 AM**
**Applications of Stereology in Materials Research**: *B. R. Patterson*[1]; S. Wu[1]; M. J. Papo[1]; X. Deng[1]; [1]University of Alabama at Birmingham, Birmingham, AL 35294 USA

This presentation gives examples of several types of stereological measurements that have proved valuable in studies of materials phenomena. These measurements include nucleation and growth rates, new methods for grain and particle size distribution measurement and characterization of crack or fracture path in metal and ceramic matrix composites. The examples of nucleation and growth rate measurements are from aluminum recrystallization studies and involve measurement of the number of recrystallized grains per volume, $N_V$, and the amount of growing surface area of recrystallizing grains, $S_V$, per unit volume. Examples of crack path characterization and the relationship to fracture toughness are given for ceramic and metal matrix composites. Metal matrix composite strength and toughness is also related to the mean free path between and through the reinforcing particles. Support received from National Science Foundation Grant No. DMR-9904352, Alcoa Technical Center, Smith International, Inc. and UAB Graduate School.

**11:05 AM**
**Quantification of Interface Energy Anisotropy Through 3-D Equilibrium Shape Measurement**: *Ralph E. Napolitano*[1]; Shan Liu[2]; Rohit Trivedi[1]; [1]Iowa State University, Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA; [2]Iowa State University, Inst. for Physl. Rsrch. & Tech., Ames, IA 50011 USA

The orientation dependent crystal-melt interfacial free energy is directly related to the equilibrium shape of discrete noninteracting particles through the Wulff construction. In many metallic systems, the amplitude of interface energy anisotropy is very low and the 3-D Wulff plot can be uniquely and fully determined through measurement of the equilibrium shape. For systems which are more anisotropic, the equilibrium shape may exhibit missing orientations or even facet planes. In these cases, the Wulff plot can only be determined for those orientations present in the shape. In this work, the anisotropy of solid-liquid interface energy is investigated in the Al-Cu system. Multiple-plane sectioning, serial-sectioning, and micromilling techniques are employed in the characterization of 3-D equilibrium shapes. Reconstructed 3-D shapes are generated and used to explicitly determine the Wulff plot. Results are fitted with both "in-plane" and "Kubic harmonic" representations of interfacial energy orientation dependence. This work was supported by the Office of Basic Energy Science, Division of Materials Science, US Dept. of Energy, under contract No. W7405-Eng-82.

**11:30 AM**
**Primary Phase Clustering in Porous Ceramics**: *Ian Nettleship*[1]; Richard J. McAfee[1]; [1]University of Pittsburgh, Dept. of Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Many methods used to characterize the lengthscale of homogeneity in complex microstructures are not practical for high volume fractions. This presentation will show how image processing techniques can be used to define clustering of grains in porous ceramics. The resulting partitioned structure was then unfolded into three dimensions to reveal the population of clusters. The implications for our understanding of creep and densification of porous ceramics will be discussed.

## Forming and Shaping of Light Weight Automotive Structures: Processing & Manufacturing - II

*Sponsored by:* Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
*Program Organizers:* Dan Zhao, Johnson Controls, Inc., Plymouth, MI 48170 USA; Prabir Chaudhury, Intercontinental Manufacturing, Garland, TX 75046 USA; Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Department, Northville, MI 48167 USA; Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA

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*Session Chair:* Zhe Jin, Alcoa Technical Center, Thermomechl. Procg., Alcoa Center, PA 15069 USA

**8:30 AM Invited**
**Laser Assisted Arc Welding for Ultra-High Strength Steels**: *David P. Pace*[1]; Kevin L. Kenney[1]; [1]INEEL, 2525 N. Fremont, Idaho Falls, ID 83415 USA

An innovative hybrid welding technique, combining laser and gas metal arc welding, was developed and investigated as a solution to the challenges of joining ultra high strength steels. The joint strength, joint formability, gap tolerance and welding speed were used to evaluate this process on two ultra high strength steels and compared to both laser and gas metal arc welding. Investigations into an industrial application will also be presented.

**9:00 AM**
**Production Robotic Friction Stir Welding: A Reality**: *Christopher B. Smith*[1]; John F.F. Hinrichs[1]; R. J. Heideman[1]; N. J. Scott[1]; W. A. Crusan[1]; [1]Friction Stir Link, Inc., W227 N546 Westmound Dr., Waukesha, WI 53186 USA

With the advent of robots with significantly larger payloads than previously available, production robotic friction stir welding (FSW) for automotive and other high volume applications is feasible. The use of a robotic FSW solution allows for manufacturing cost reductions versus other joining technologies (e.g. GMAW, riveting, etc.) that would not otherwise be possible in many high volume applications. The use of a robot also adds flexibility, improves the robustness of the process in production and expands the range of applications where the joining process is applicable. In this paper, the history of robotic FSW development will be discussed, explaining why robotic FSW is the right choice for many applications. In addition, several high volume applications will be discussed where robotic FSW provides significant cost and/or manufacturing advantages.

**9:25 AM**
**Hot Forging of Light Weight Structural Parts Using Stock Produced by Equal Channel Angular Extrusion/Pressing (ECAE/P)**: *Prabir K. Chaudhury*[1]; Gabriel Regalado[1]; [1]Intercontinental Manufacturing, Metall., 1200 N. Glenbrook Dr., Garland, TX 75046 USA

Ultrafine grain material produced by Equal Channel Angular Extrusion/Pressing (ECAE/P) has been shown to have superior strength properties at low temperatures. This investigation evaluated use of very fine to ultrafine grain material as a forging stock in hot forging of light metal structural parts. Two aluminum (AA 6061) and magnesium (ZK60) alloy parts were hot forged using conventional stock materials and ECAE/P processed materials at various temperatures. The quality of the forgings in terms of die fill, cracks, laps, folds, and other defects were examined. The forgings with very fine to ultrafine grain material were successfully produced at much lower temperatures and with reduced stock size. The results show that the forging temperatures and the forging loads can be reduced considerably as a result of finer grains in these alloys. This presentation will discuss the applicability of the ECAE/P produced material in light weight structural part forgings in terms of alloy system and grain refinement.

**9:50 AM**
**Magnesium Alloy Forgings for Light Weight Applications**: *Prabir K. Chaudhury*[1]; [1]Intercontinental Manufacturing, 1200 N. Glenbrook Dr., Garland, TX 75046 USA

Wrought magnesium alloys exhibit specific mechanical properties that are comparable to high strength Al and Ti alloys. The magnesium alloy forgings can meet the strength and weight requirements of many lightweight applications such as those in aerospace and automotive industries. However, the use of magnesium alloy forged structural products is limited to specific aerospace applications only, although they have been proven to function very reliably in many structural applications. Very few wrought alloys have been developed and most structural forgings use AZ80A and ZK60A. This is mainly because of the lack of knowledge in terms of their service properties and processing data. This paper will show examples of various forgings produced for aerospace industry and provide some of the engineering and processing data. The presentation will also discuss some of the recent advances in magnesium alloy forgings leading to safer processing conditions.

**10:15 AM Break**

**10:25 AM**
**Reactive Thermo-Mechanical Processing for the Low-Energy Production of Aluminide Structural Intermetallics**: *Khaled Morsi*[1]; Jose Rodriguez[1]; James Wall[1]; Sherif O. Moussa[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA

Aluminide intermetallics are of significant technological importance. These aluminum containing materials offer desirable materials properties with potential weight savings. Major drawbacks, which have so far restricted the use of these materials, are the high-energy usage in synthesis and processing and difficulty in fabrication into the final component shape. The proposed work explores a new near net shape, low-energy approach which can be used to process a range of important aluminide intermetallics and their composites (e.g. titanium aluminides, nickel aluminides, iron aluminides and niobium aluminides), having wide ranging applications within the automotive sector. Reaction synthesis (RS) is a process that makes effective use of the chemical energy generated during a reaction to process structural intermetallics with minimal external energy inputs. Although RS presents great advantages in terms of cost and energy savings, the resultant microstructure often includes unwanted porosity, with negative impact on mechanical properties. The work presented represents the first application of thermomechanical processing (extrusion and forging) during the high temperatures achieved in reaction synthesis to form and simultaneously shape aluminide products. The effect of varying processing parameters on the developed microstructure and properties is presented. Results confirm the feasibility of this new approach to process these materials at operating temperatures ~400°C lower than conventionally used in the extrusion and forging of these materials with potentially considerable energy and cost savings.

**10:50 AM**
**Retrogression Heat Treatments in AA6111 and Their Effect on Formability**: *Paul E. Krajewski*[1]; Edward F. Ryntz[1]; [1]General Motors, R&D, 30500 Mound Rd., MC 480-106-212, Warren, MI 48090 USA

Retrogression heat treatments have been shown to significantly improve the formability of age-hardenable aluminum alloys. This technology has been successfully used to make aluminum ladders and other structures. The present study investigates retrogression heat treatments in AA6111 with the goal of improving press formability. The kinetics of retrogression heat treatments were characterized, and their effect on aging kinetics as well as formability studied in detail. Retrogression heat treatments significantly increase the fracture curve for aluminum alloys, which allows for improved bending and local forming. Specific application of retrogression heat treatments to automotive forming problems such as hemming and trimming will be discussed.

**11:15 AM**
**Preferential Softening of 6063-T6 Aluminum Alloy Utilizing a High Density Infrared (HDI) Plasma Arc Lamp**: *Ronald Derek Ott*[1]; Craig A. Blue[1]; Adrian S. Sabau[1]; Tsung-Yu Pan[2]; Armando M. Joaquin[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS-6063, Oak Ridge, TN 37831-6063 USA; [2]Ford Motor Company, Mfg. Sys. Dept., Scientific Rsrch. Lab., 2101 Village Rd., Dearborn, MI 48124 USA

Oak Ridge National Laboratory has a very unique rapid heating capability with its high-density infrared (HDI) plasma arc lamp. Power densities up to 3.5 $kW/cm^2$ are achievable over an area of 35 cm by 3.175 cm. The power is continuously variable and can be pulsed ranging from 1.5% to 100% and power changes can take place in less than 20 milliseconds. Processing temperatures up to 3,000°C can be obtained in varying environments, making it a very robust manufacturing resource. Recently this capability has been used to investigate the selective softening, i.e. hardness reduction, of extruded 6063-T6 aluminum alloy. By changing the amperage and exposure, or pulse, time the percent reduction in hardness and softened zone size can be varied. In the current experiments a 50% reduction in hardness has been achieved and confirmed by microstructural examination, which showed the $Mg_2Si$ precipitates have gone back into solution.

## Mechanisms and Mechanics of Fracture: A Symposium in the Honor of Professor J. F. Knott: Advanced Materials

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA; R. O. Ritchie, University of California-Berkeley, Department of Materials Science & Engineering, Berkeley, CA 94720 USA

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*Session Chairs:* Rob Ritchie, University of California-Berkeley, Berkeley, CA 94720 USA; K. L. Murty, North Carolina State University, Nucl. Eng., Raleigh, NC 27695-7909 USA

**8:30 AM**
**Fracture and Damage in Particle Reinforced Metal Matrix Composites**: *Bhaskar S. Majumdar*[1]; [1]New Mexico Tech, Matls. Dept., 801 Leroy Place, Socorro, NM 87801 USA

In order to predict the toughness of particulate based aluminum composites, we have developed a model that owes its origin partly to Knott's model of shear-dominant fracture of high-strength alloys. The model predicts lower toughness with increased strength, unlike most predictions. We have also been investigating damage mechanisms in composites. Metallography reveals particle fracture and localized matrix slip as two important damage/inelasticity processes. Insitu neutron diffraction measurements indicate regular load transfer during the initial stages of plasticity. However, at large strains, the SiC particles exhibit a stress saturation, although only 20% of particles are cracked. The matrix, on the other hand, exhibits a reloading behavior. We interpret this anomalous reverse load-transfer to a synergistic network of plastically deforming matrix. The interpretation is backed with FEM analysis, and provides insight into the inelastic processes leading to final composite failure.

**8:55 AM**
**The Effects of Crystal Orientation and Misorientation on Crack Propagation and Arrest in a Near-Gamma TiAl Alloy**: *Boon-Chai Ng*[1]; Tom R. Bieler[1]; Martin A. Crimp[1]; [1]Michigan State University, Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824 USA

Understanding how and why cracks arrest may provide insight for strategies to improve the ductility of structural materials. In this study, near gamma TiAl alloy was examined using electron channeling contrast imaging (ECCI) to investigate dislocation and twin activity in regions where facile fracture or crack arrest occurred. In-situ notched 4-point bend tests of bulk samples were performed to assess crack configurations in relation to micro and substructural morphologies. Upon loading, cracks propagated in a zig-zag manner from the notch and was successfully arrested. Conditions that facilitate or frustrate crack propagation were quantitatively correlated with operative slip and twinning systems, crystal orientations, misorientations with neighboring grains, and the efficiency of deformation transfer across boundaries.

**9:20 AM**
**Tensile Microcrack Initiation at Gamma-Gamma Boundaries in TiAl**: *B. A. Simkin*[1]; T. R. Bieler[1]; M. A. Crimp[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., 2527 Engineering, E. Lansing, MI 48824-1226 USA

Although gamma titanium aluminide alloys are beginning to find application, their potential is hampered by low room temperature ductility. In the present study, microcrack nucleation is examined on the tensile surface of Ti-48Al-2Cr-2Nb four point bend specimens that were deformed but not fractured. A systematic search for microcracks showed that most occurred by decohesion of gamma-gamma grain boundaries in the alloy. The nature of the deformation defects present in the grains associated with a grain boundary are related to the fracture or non-fracture of that grain boundary. Direct imaging of the active twinning and dislocation systems was accomplished using electron channeling contrast imaging (ECCI). Grain orientations were determined by means of selected area channeling patterns (SACPs) in the SEM, which allows the unambiguous determination of the crystal orientations for the tetragonal gamma-TiAl, rather than the pseudo-cubic orientation determination typically obtained by electron backscatter diffraction (EBSD) methods. The relationships between active slip systems and crack nucleation is discussed in relation to Schmid factors and a compatibility factor for deformation transfer using the observed active systems. This work was supported by the Air Force Office of Scientific Research under grant #AFRL NO F49620-01-1-0116.

**9:45 AM**
**The Fracture Behavior of Discontinuously Reinforced Aluminum (DRA)**: *Nik Chawla*[1]; [1]Arizona State University, Mechl. Behavior of Matls. Fac., Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

The fracture processes in composite materials are quite different from that of their monolithic counterparts. In this talk, the tensile and fatigue fracture behavior of discontinuously reinforced aluminum (DRA) will be presented. In tension DRA exhibits planar fracture of the brittle reinforcement particles and ductile microvoid growth and coalescence in the aluminum matrix. The effect of particle strength, strength of the particle/matrix interface, and matrix strength on tensile behavior of the material will be discussed. Fatigue fracture in the composites is controlled by surface defects, particle clusters, and rogue-inclusions, particularly when these defects are larger than the reinforcement particle size. The implications of defects on fatigue fracture and fatigue life will also be discussed. Research sponsored by the United States Automotive Materials Partnership and the Office of Naval Research.

**10:10 AM Break**

**10:25 AM**
**Effects of Structure on the Interfacial Fracture of Thin Ductile Films**: *Neville R. Moody*[1]; D. P. Adams[2]; N. Yang[1]; A. A. Volinsky[3]; W. W. Gerberich[4]; [1]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA; [3]Motorola, Mesa, AZ 85202 USA; [4]University of Minnesota, Minneapolis, MN 55455 USA

Interfaces are critical factors in controlling the reliability of hybrid microcircuits. These devices contain sub-micron-thin films with nanoscale structures and properties that vary markedly with changes in processing. As a consequence, we have begun a study of the relationship between interface structure and properties in 200-nm-thick sputter deposited gold films using two substrate preparation procedures. High Resolution TEM shows these two procedures create dramatically different structures. One procedure creates 5-nm-thick epitaxial twins in the gold film along a well-defined film-substrate interface while the other creates a disordered interface and a gold film with random in-plane texture. Although the properties were similar, the interfacial fracture energies varied significantly with the twinned microstructure exhibiting very low interfacial fracture resistance. When combined with fracture models, the results suggest that the epitaxial twins inhibit dislocation emission from interfacial cracks leading to low measured fracture energies. This work supported by USDOE Contract DE-AC04-94AL85000.

**10:50 AM**
**The Quasi Static and Cyclic Fracture Behavior of 2009 Aluminum Alloy Reinforced with Silicon Carbide Particulates**: *T. S. Srivatsan*[1]; Meslet Al Hajri[1]; [1]The University of Akron, Dept. of Mechl. Eng., Akron, OH 44325-3903 USA

Development of discontinuously-reinforced metal-matrix composite materials has certainly provided the much desired impetus for a thorough and systematic understanding of microstructural effects on quasi-static and cyclic deformation and fracture characteristics, in order to develop a framework for predicting their behavior when subjected to a spectrum of loading conditions. This technical presentation will focus on rationalizing using principles of materials science and mechanics of solids the extrinsic and intrinsic mechanisms and micromechanisms governing the quasi-static and cyclic fatigue fracture characteristics of a silicon carbide particulate (SiCp)-reinforced 2009 aluminum alloy metal-matrix composite. The emphasis will be on understanding composite microstructural effects on fatigue performance and associated failure modes. The damage mechanisms will be rationalized in light of concurrent and mutually interactive influences of nature of loading, test temperature, microstructural condition, and composite fracture mode.

**11:15 AM**
**Plasticity Length Scales and Stress-Life Behavior in LIGA Nickel MEMS Structures Under Cyclic Loading**: W. O. Soboyejo[1]; *Jun Lou*[1]; Pranav Shrotriya[1]; Seyed Allameh[1]; T. Buchheit[2]; [1]Princeton University, Princeton Matls. Inst. & the Dept. of Mechl. & Aeros. Eng., Olden St., Princeton, NJ 08544 USA; [2]Sandia National Laboratories, Modlg. & Reliability Dept., Albuquerque, NM 87123 USA

This paper presents the results of a study of cyclic deformation and fatigue fracture in LIGA nickel MEMS structures. A modeling framework is presented for the extraction of plasticity length scale parameters from cyclic micro-bend experiments. The measured plasticity length scale parameters are then related to underlying dislocation substructures. Following the initial emphasis on plasticity length scale parameters, a micro-fatigue test set-up is described before presenting initial stress-life data obtained from micro-fatigue experiments. The underlying microstructural changes associated with micro-fatigue are then described before discussing the relative contributions from crack nucleation and growth components to the total fatigue life.

**11:40 AM**
**An Investigation of Fatigue and Fracture in Bulk Amorphous Alloys**: *Christopher Mercer*[1]; R. Cirincione[1]; P. Shrotriya[1]; P. Anglin[1]; W. O. Soboyejo[1]; [1]Princeton University, Princeton Matls. Inst. & the Dept. of Mechl. & Aeros. Eng., Olden St., Princeton, NJ 08544 USA

This paper presents the results of an investigation of deformation, crack nucleation and propagation in bulk amorphous alloys. Hertzian indentation techniques are used to identify the stress states associated with shear band nucleation and crack nucleation under monotonic and cyclic loading. The mechanisms of fatigue crack nucleation and propagation are also explored in smooth and notched bend specimens. The results show that fatigue crack growth in amorphous metals occurs in three distinct regimes, as in ductile metals and their alloys. The underlying mechanisms of fatigue fracture are elucidated before analyzing the shielding contributions from micro-cracking.

**12:05 PM**
**Effect of Constitutive Relations on Plastic Deformation Localization under Kinematic Hardening Rule**: *Su-Jin Yun*[1]; [1]Agency for Defense Development, Tech-4-4, Yuseong-ku PO Box 35, Daejon 305-600 Korea

It is attempted to simulate the plastic deformation under simple shear loading using rate-independent constitutive relations, elastic-plastic flow theory, deformation theory and rigid plasticity. The stress rate is corotated with spin of substructure obtained using plastic spin. First, the finite difference analysis for a homogeneous deformation is carried out with the constrained boundary conditions. Then, it is followed by inhomogeneous analysis by introducing a small perturbation, which is corresponding to the initial geometric defects in the work piece. A similar finite difference scheme is employed to investigate the effects of constitutive relation on the shear band formation. The thermal energy due to plastic work is ignored so that the distinct shear localization behavior stems from the nature of each constitutive description. The rigid plastic material results in more severe shear band than the elastic-plastic material. Moreover, it is noted that when elasto-plasticity used, deformation theory yields lower shear localization than flow theory.

**12:30 PM**
**Microstructural Banding and Biaxial Fracture Toughness Tests in a Specially Heat-Treated Reactor Pressure Vessel Steel:** Randy K. Nanstad[1]; Mikhail A. Sokolov[1]; Philip J. Maziasz[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6151 USA

The Heavy-Section Steel Technology (HSST) Program at Oak Ridge National Laboratory (ORNL) includes a task to investigate the effects of constraint on the cleavage initiation fracture toughness of reactor pressure vessel (RPV) steels using relatively large cruciform fracture toughness specimens under varying degrees of biaxial loading. During characterization testing of the specially heat treated plate of A533B steel, some relatively low fracture toughness values were observed and subsequent metallography revealed the presence of varying degrees of dark bands in the microstructure. An investigation was subsequently conducted to evaluate the whether the fracture toughness results obtained from the biaxially loaded specimens were influenced by the steel microstructure in a biased manner. Various metallurgical tools, including metallography, microhardness testing, scanning electron fractography, electron microprobe analysis, and analytical electron microscopy were used to characterize the nature of the bands and evaluate the potential effects on the fracture toughness results. The main conclusions are presented and discussed.

## Modeling the Performance of Engineering Structural Materials: Modeling Accounting for Microstructure

*Sponsored by:* Structural Materials Division, SMD-Structural Materials Committee
*Program Organizers:* Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; T. Srivatsan, University of Akron, Department of Mechanical Engineering, Akron, OH 44325-3903 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Thursday AM — Room: 215
October 10, 2002 — Location: Columbus Convention Center

*Session Chairs:* Yu Xiang, University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA; Chi-Sing Man, University of Kentucky, Dept. of Math., 715 Patterson Office Tower, Lexington, KY 40506-0027 USA

**8:30 AM**
**Effects of Texture and Microstructure on Plastic Anisotropy of Aluminum Alloy Sheet**: *Yu Xiang*[1]; *Chi-Sing Man*[2]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA; [2]University of Kentucky, Dept. of Math., 715 Patterson Office Tower, Lexington, KY 40506-0027 USA

Man recently derived expressions which show explicitly how crystallographic texture affects the angular dependence of the q-value and the uniaxial flow stress $\sigma$ of sheet metals. These expressions were corroborated by experimental data on an AA5xxx hot band and its O-temper counterpart. Here we report on the findings of a further study on samples of the same aluminum alloy which resulted after the hot band was annealed at 500°F for 5 hours. Optical microscopic and X-ray analyses indicated that neither the grain size and shape nor the texture of the hot band changed after this heat treatment. Results of uniaxial tension tests showed that the angular dependence of the q-value also remained unchanged, but the directional properties of $\sigma$ were substantially different before and after the annealing. These findings suggest that, whereas texture may suffice in accounting for the anisotropy of the plastic potential, some additional microstructural factor(s) other than grain size and shape must affect the anisotropy in the yield function of this alloy. TEM and EBSD were employed to seek the microstructure(s) that further contributed to the flow stress anisotropy.

**8:55 AM**
**Application of Nanoindentation Measurements and 3D Finite Element Simulations for Calculation of Local Constitutive Behavior of a Magnesium Alloy**: *Arun M. Gokhale*[1]; *Zhaohui Shan*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., 771 Ferst Dr., Love Bldg., Atlanta, GA 30332-0245 USA

Cast Magnesium alloys invariably contain a layer of fine-grained structure (so called skin) at the external surfaces and a relatively coarse grained microstructure in the interior that also contains significant amount of porosity. To model the mechanical response of such castings, the local constitutive behaviors corresponding to the fine-grained skin region and the pore-free interior are required. These data obviously cannot be obtained from standard mechanical tests performed on a bulk alloy specimen. In this study, nanoindentation technique and Finite Element simulation have been applied to compute the local mechanical properties of different regions in an AM60 Mg-alloy die-casting. The results show that the elastic properties (Young's modulus) of the AM60 alloy are the same in the skin and interior regions, and they are almost the same as those of the bulk specimen measured by standard tensile test. However, the plastic properties (yield stress and strain hardening exponent) of the AM60 alloy at the skin and the interior regions are different, and significantly higher than those of the bulk specimen measured by tensile test.

**9:20 AM**
**Microstructure-Based Modeling of Thermal and Elastic Behavior of Materials Using Object Oriented Finite Element Analysis (OOF)**: *N. Chawla*[1]; D. T. Vonk[1]; *K. K. Chawla*[2]; B. Patel[2]; M. Koopman[2]; B. R. Patterson[2]; [1]Arizona State University, Mechl. Behavior of Matls. Fac., Dept. of Cheml. & Matls. Eng., Tempe, AZ 85284 USA; [2]University of Alabama at Birmingham, Dept. of Matls. Sci. & Eng., Birminghan, AL 35294 USA

The microstructure of a material inherently controls its properties. Thus, it becomes important to model the material behavior using a microstructure-based approach. Object Oriented Finite Element Analysis (OOF) is a useful tool for modeling the microstructural features of a material, which are typically not incorporated in traditional finite element analysis typically utilizing a unit cell approach. This talk will present an overview of OOF as a tool for modeling elastic and thermal problems. Several examples of the modeling of elastic and thermal properties, using OOF, will be described. The material systems studied include: SiC particle reinforced Al matrix composites, "double cemented" WC reinforced Co composites, and Sn-Ag solder/Cu joints. It will be shown that OOF, through incorporation of the actual material microstructure, can provide for a powerful tool for modeling and understanding of microstructural features and prediction of material behavior. Research supported by the National Science Foundation and the Office of Naval Research

**9:45 AM**
**A Neural Network Analysis of HSLA Steels**: *Edward A. Metzbower*[1]; [1]Naval Research Laboratory, Code 6320, Washington, DC 20375-5320 USA

A neural network analysis has been performed on a data set from programs that the US Navy funded in order to determine the composition and properties of HSLA steels. The input variables were the chemical elements, plate thickness, and tempering temperature. The outputs consisted of the yield and ultimate tensile strength, elongation, and Charpy V-notch values at either -1 or -51 C. The neural network technique used a Bayesian framework and allowed the estimation of error bars that warn when the data are sparse or locally noisy. After the neural network was trained, a best model was determined. The single best model sometimes generalizes poorly on unseen data. A committee of best models was used to make predictions that are more reliable than the single best model. Several compositions were evaluated at different thicknesses and strengths to assess the effect of changes in composition on the properties of the plate.

**10:10 AM**
**Microstresses and Plasticity Mechanisms in High-Volume Fraction Superalloys**: Shuwei Ma[1]; Partha Rangaswamy[2]; Donald Brown[2]; *Bhaskar S. Majumdar*[1]; [1]New Mexico Tech, Matls. Dept., 801 Leroy Place, Socorro, NM 87801 USA; [2]Los Alamos National Laboratory, Los Alamos, NM, USA

High volume fraction superalloys exhibit strength and creep resistance well in excess of the individual phases. For efficient alloy development and life prediction, it is important to model and validate deformation mechanisms at the local level. We conducted insitu neutron diffraction measurements to probe the deformation behavior of a polycrystalline superalloy, CM247LC. Of particular interest was the onset of plastic deformation, as influenced by elastic anisotropy and Schmid factor, and by the geometric constraint arising out of the relatively large (~300nm) gamma-prime (gp) phase surrounded by narrow gamma channels. We used dislocation models to correlate the CRSS for the different orientations with the experimental results. For example, the highly constrained [100] grains were modeled based on the APB energy requirement for producing superpartials in the gp phase. Penetration of the gp phase was not required for the [110] orientation. Comparisons between model results and experimental findings will be provided.

## Notes

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