

Technical Program

WEDNESDAY AM

DAMAGE PROCESSES IN ADVANCED MATERIALS: Composites and Metallic Glasses

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Corrosion and Environmental Effects Committee, Physical Metallurgy Committee

Program Organizers: Russ H. Jones, Pacific Northwest Natl. Lab., Richland, WA 99352 USA; Richard Ricker, NIST, Materials Rm B254, Gaithersburg, MD 20899; Norman S. Stoloff, Rensselaer Polytechnic Inst., MS&E Dept., Troy, NY 12190-3590 USA

Wednesday AM Room: Paris C
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; N. S. Stoloff, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA

8:30 AM INVITED PAPER

MECHANISMS OF FRACTURE AND FATIGUE IN ADVANCED MATERIALS: R. O. Ritchie¹; ¹University of California, Materials Sciences Division, Lawrence Berkeley National Laboratory and Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA

Whereas damage-tolerant design and life-prediction methodologies have been widely practiced for metallic structures for years, particularly in the nuclear and aerospace industries, the development of advanced structural materials, such as metallic glasses, toughened ceramics, intermetallics and their respective composites, poses particular problems. These problems pertain to the fact that, in general, these advanced materials possess high strength yet invariably low ductility, meaning that they are highly sensitive to the presence of flaws. Moreover, it is now known that these materials are susceptible, as in metals, to premature failure by fatigue; this provides a prominent mechanism for the subcritical growth of cracks and further limits life. In this presentation, studies to discern the microstructural mechanisms associated with subcritical crack propagation in intermetallics, ceramics and metallic glasses are examined, and compared to behavior in traditional metals. Despite obvious differences in the processes of microstructural damage and crack advance in such ductile and brittle materials, the commonality of mechanisms both ahead and behind the crack tip are explored.

9:10 AM INVITED PAPER

OXIDATION EFFECTS ON THE TIME-DEPENDENT FAILURE OF FIBER-REINFORCED SiC AT ELEVATED TEMPERATURE: E. Lara-Curzio¹; Peter F. Tortorelli¹; Karen L. More¹; ¹Oak Ridge National Laboratory, Metals and Ceramics Division, P. O. Box 2008, Oak Ridge, TN 37831-6069 USA

Under constant tensile loading in air between 600 and 900°C, Nicalon-reinforced SiC composites can exhibit significant strains to failure. This behavior can be explained on the basis of a progressive fiber-failure model that incorporates removal of the fiber-matrix interphase by oxidation, formation of silica on the Nicalon and SiC, and subsequent fiber failure and stress redistribution. Using a straight-forward fiber-bundle analysis, measured oxidation rates, and reason-

able assumptions, the model can accurately predict the observed stress-rupture curves and measured times-to-failure over extended temperature and loading ranges. Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, U. S. Department of Energy under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation.

9:50 AM INVITED PAPER

OXIDATION EFFECTS ON MECHANICAL PROPERTIES OF BLACKGLASS| MATRIX NEXTEL|312 REINFORCED CFCC'S: P. J. Balin¹; Michael J. McNallan; ¹University of Illinois-Chicago, CME Dept., M/C 246, Rm 3083 ERF, 842 W. Taylor St., Chicago, IL 60607 USA

Blackglas™ is a silicon oxycarbide glass, produced by pyrolysis of a preceramic polymer. Composites based on Blackglas™ can be fabricated by resin transfer molding and are suitable for use at moderately elevated temperatures. Mechanical properties of these composites may degrade as a result of oxidation reactions in service, with the kinetics of degradation affected by the pyrolysis procedure and service conditions. Composite panels of Blackglas™ matrix- Nextel™ 312 fiber reinforced CFCC's have been fabricated. Coupons cut from the panels have been subjected to 3-point and 4-point bend flexure testing before and after oxidation exposures at 600°C in air, with and without humidification. The effects of exposure time, humidity, and test geometry on the measured mechanical properties of the CFCC's are discussed.

10:30 AM

HIGH-TEMPERATURE DAMAGE PROCESSES IN SiC/SiC COMPOSITES: R. H. Jones¹; C. A. Lewinsohn¹; C. H. Henager¹; ¹Pacific Northwest National Laboratory, MSIN P8-15, Richland, WA 99352 USA

There are a number of high-temperature processes that can produce damage in SiC/SiC composites. Time-dependent matrix cracking as a result of fiber relaxation mechanism (FRM), interphase removal mechanism (IRM) or oxidation embrittlement mechanism (OEM) are three such processes. FRM, IRM and OEM occur at different environmental conditions and exhibit different effects on the material. FRM occurs over a range of temperatures but at very low O₂ concentrations while both OEM and IRM require the presence of O₂. IRM occurs at T > T_g, the glass transition temperature for SiO₂, and pO₂ less than atmospheric pressure while OEM occurs at T < T_g and atmospheric pressure of O₂. Damage from matrix cracking includes changes in the compliance of the composite, hermetic performance and loss of protectiveness for the interior fibers and interphases. However, under some circumstances, the fracture strength of the composite is unaffected for matrix cracks up to a/W of 0.6. Comparison between the various crack growth processes and the type of composite damage will be summarized.

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EFFECTS OF TIME, STRESS, AND ENVIRONMENT EXPOSURE ON A GLASS POLYURETHANE AUTOMOTIVE COMPOSITE: Kelly L. Willson¹; John M. Henshaw¹; Dan Q. Houston²; ¹University of Tulsa, Mechanical Engineering, 600 S. College Ave., Tulsa, OK 74104 USA; ²Ford Motor Company, Manufacturing Systems Department, 20000 Rotunda, MD 3135 SRL, Dearborn, MI 48121 USA

A continuous-strand mat (CSM) glass reinforced polyurethane composite is exposed to different environments (distilled water and warm humid air) for times up to 2000 hours and at stresses up to 50% of ultimate strength. The effects of these conditions on the material are described and analyzed. Residual properties (ultimate strength and modulus of elasticity) are seen to degrade with increasing time and

stress level. Most of the effects of the environmental exposures on residual properties are shown to be reversible upon drying out of the material. Moisture absorption (weight gain) is more rapid at higher stresses, but the saturation level appears to be independent of stress level. The interactions among the material, stress, and environment are investigated and discussed.

11:10 AM

MECHANISMS FOR FRACTURE AND FATIGUE-CRACK PROPAGATION IN A BULK METALLIC GLASS: *C. J. Gilbert*¹; V. Schroeder¹; R. O. Ritchie¹; ¹University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA

Novel measurements of the fracture and fatigue properties of a newly developed bulk metallic glass alloy, $Zr_{41.2}Ti_{13.8}Cu_{12.5}Ni_{10}Be_{22.5}$ (at.%) have been made with specific emphasis on determining microstructural mechanisms. Fatigue pre-cracked compact-tension specimens indicated that the amorphous alloy had a plane-strain fracture toughness of ~ 55 MPa m. Under monotonic loading, fracture surfaces exhibited a typical vein morphology, and evidence for local melting was observed. Attempts were therefore made to rationalize the measured toughness in terms of a previously developed micromechanical model based on the Taylor instability, as well as on the observation of extensive crack branching and deflection. Upon crystallization, the alloy was severely embrittled with toughnesses dropping to ~ 1 MPa m. Commensurate with this toughness decrease was a marginal increase in hardness and a reduction in ductility (as measured via depth-sensing indentation experiments). Under cyclic loading, crack-propagation behavior in the amorphous structure was similar to that observed in traditional polycrystalline alloys, in that the crack-advance mechanism was associated with alternating blunting and resharping of the crack tip. This was evidenced by striations on fatigue fracture surfaces. Conversely, the (unnotched) stress-life properties were markedly different. Specifically, crack initiation occurred quite readily due to the lack of microstructural barriers which normally provide crack-arrest points; this resulted in a low fatigue limit of ~ 0.04 of the tensile strength.

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EXPERIMENTAL OBSERVATION OF DISLOCATION-TYPE DEFECTS IN AMORPHOUS ALLOYS: *Mikhail V. Finkel*¹; L. B. Zuev²; V. I. Danilov²; ¹DAATH-Scientific Center, Materials Science, 9926 Haldeman Ave. #36A, Philadelphia, PA 19115-1609 USA; ²Institute for Metal Physics, Tomsk Russia

Metallography methods have been used to experimentally discover dislocation-type defects in amorphous structure. Pile-ups of defects with the density of distribution up to $10^{12}m^{-2}$ have been revealed in various rapidly solidified amorphous alloys. Defects are oriented perpendicularly to the ribbon surface, have length of several micrometers, and their estimated cross-section diameter is 10^{-9} - 10^{-8} m. Reordering of defect pile-ups due to structural relaxation, plastic deformation, and fracture of material is studied. Formation of new and dissolving of existing defects is observed in zones of intensive plastic flow. Etching figure patterns similar to those observed for dislocations in crystals (such as straight chains of defects) are demonstrated. Fine structure and genealogy of defects are discussed from dislocation positions using existing and novel approaches.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Fracture

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Flow & Fracture Committee, Mechanical Metallurgy Committee

Program Organizers: Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Marc A. Meyers, IMM, University of California, San Diego, CA 92093 USA; Naresh Thadhani, Georgia Inst of Tech, Sch of Matis Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Wednesday AM Room: Sydney
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Naresh N. Thadhani, Georgia Institute of Technology, Materials Science and Engineering, Atlanta, GA 30332-0245 USA; George T. Gray, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

8:30 AM

DYNAMIC FRACTURE UNDER SPALL CONDITIONS: *Anna K. Zurek*¹; ¹LANL, MST-8, MS G755, Los Alamos, NM 87544 USA

The stress state of materials under spall conditions is drastically different than that for quasi-static loading. This difference arises from different stress triaxialities, global versus local strains, local porosity, and most of all from differences in strain rate and hardening due to the compressive wave preceding the spall inducing tensile wave. In this review paper we will describe the fracture process under spall conditions and compare it to fracture under quasi-static loading condition. We will concentrate on the shapes of the voids, their nucleation, growth and coalescence, as well as the total porosity arising from the voids dictated by the global and local stress triaxiality and strains. Finally, we will discuss recent advances in describing and quantifying damage accumulation specifically for incorporation into a micromechanical model of ductile fracture.

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DEFORMATION DYNAMICS OF AMORPHOUS SOLIDS: THE IMPLICATIONS FOR FRACTURE: *Michael L. Falk*¹; ¹University of California, Department of Physics, Santa Barbara, CA 93106 USA

Amorphous solids such as metallic glasses and non-crystalline ceramics exhibit both brittle and ductile fracture. Not coincidentally, these materials display complicated inelastic response to shear. Molecular dynamics investigations of the shear response of a simple two-dimensional two-component amorphous Lennard-Jones solid reveal analogous properties to those seen in actual materials. Examination of the rearrangements underlying these phenomena leads to the conclusion that the basic unit of inelastic shear can be understood as a microscopic two-state system. A theory of the dynamics of inelastic deformation under shear is developed from this assumption and compared to the simulation results. It is further conjectured that an understanding of this non-linear response sheds light on our recent simulations of brittle/ductile fracture behavior in computational models of amorphous solids as well as dynamic fracture experiments performed by others on polymeric systems in the laboratory.

9:10 AM

GOVERNING FACTORS OF THE SPALL FRACTURE: *Gennady I. Kanel*¹; ¹High Energy Density Research Center, IVTAN, Izhorskaya 13/19, Moscow, Russia 127412

Data on the resistance to spall fracture of a number of metals and alloys as a function of peak shock stress, load duration and orientation, material structure, and temperature are analyzed to determine the factors that govern metal fracture in uniaxial dynamic tensile fracture. It is shown that the dynamic strength is a structure-sensitive parameter. In commercial polycrystalline alloys fracture is nucleated at relatively large nonuniformities of the parent material. With an increase in the tension rate, the strength growth significantly and the excess stresses associated initiate fracture at increasingly smaller and more numerous damage nucleation sites. Initiation of the fracture on smallest defects which occurs in single crystals requires much greater stresses than that on grain boundaries, inclusions and other relatively coarse defects of polycrystalline alloys. When the spall thickness is less than the grain size, both in-granular and inter-granular strength can be recorded. The microscopic defects generated by shock compression do not appreciably affect the spall strength of commercial alloys. The dynamic strength of steels increases as a result of both quenching and the reversible high-pressure polymorphous transformation. The spall tests determine most fundamental bulk strength properties of materials. Besides short load duration, spalling conditions are characterized by small deformation at stressed states close to three-dimensional tension. Neither the surface of the body nor isolated coarse defects contribute to the main development of the spall fracture. A relationship between the yield stress, strain hardening, and the bulk strength indicates the material toughness. Due to that, the spall tests may be considered as a promising tool to characterize the materials.

9:30 AM

ROOM-TEMPERATURE DUCTILITY IN FINE-GRAINED ALUMINUM ALLOYS AT DYNAMIC STRAIN RATE: *Toshiji Mukai*¹; Koichi Ishikawa¹; Yoshihira Okanda¹; Kenji Higashi²; ¹Osaka Municipal Technical Research Institute, Mechanical Engineering Dept., 1-6-50 Morinomiya, Joto-ku, Osaka 532-8553 Japan; ²Osaka Prefecture University, Dept. of Metallurgy and Materials Science, 1-1 Gakuencho, Sakai, Osaka 593 Japan

Recently, microstructure of aluminum can be refined less than 1 micron by the advanced technique, such as mechanical alloying, crystallization from amorphous powder, consolidation of nano-crystalline powder, physical vapor deposition and/or severe plastic deformation. It has been extensively investigated mainly by compression and hardness tests that the strength of the fine-grained aluminum alloy increased with refining grain size. Very limited data on the tensile properties, however, has been reported for the fine-grained materials. To investigate the tensile properties at dynamic strain rates are very important for the application of these fine-grained materials to structural components. In this study, several fine-grained materials have been examined using a modified Hopkinson bar method for tensile tests. As a result, the fracture mechanism of the examined materials can be divided into two groups. The alloy strengthening by high volume fraction of second-phase particles exhibits almost brittle fracture, while the alloy strengthening by solute atoms or nano-dispersoids fractures with the ductile dimple. The ductile-fractured material exhibits the enhanced ductility with increasing strain rate, while the brittle-fractured one does the reduced ductility. The final goal of this study is to determine an optimum microstructure for high speed impact performance.

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SELF-SIMILARITY EFFECTS IN DYNAMIC FRACTURE OF MATERIALS: *Oleg Naimark*¹; ¹ICMM, Russian Academy of Science, Phys.Foundation of Strength, 1 Acad.Korolev Str., Perm 614013 Russia

Self-similar solid response on impact was established under spall failure of PMMA and ultraporcelain as stress amplitude independent response on fracture time ("dynamic branch" effect) with specific failure pattern (numerous mirror zones with different mean radius at spall surfaces). This result was predicted theoretically as resonance excitation of localised failure structures which subject failure kinetics under transition from damage to fracture. These structures represent areas with concordant blow-up microcrack growth kinetics and corre-

spond to eigen-function spectrum of self-similar solution. The existence of travelling structures allows the explanation of superdeep penetration effect for high-speed collision of particle flux with hard target. Eigenfunction spectrum is phase coordinates of strange attractor that determines the transition from steady-state to branching regime of crack propagation dynamics.

10:10 AM BREAK

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QUANTITATIVE DESCRIPTION OF DAMAGE EVOLUTION IN DUCTILE FRACTURE OF TANTALUM: *Jesus M. Rivas*¹; ¹Los Alamos National Laboratory, Materials Science and Technology Division, MST-8, MS G755, Los Alamos, NM 87545 USA

A systematic analysis of the evolution of several microstructural parameters during spallation of tantalum has been conducted in an effort to incorporate quantitative descriptors of damage accumulation into a micromechanical model of ductile fracture. In this paper, we describe a methodology to quantify important microstructural parameters of the ductile fracture process such as void size and void density. Four different samples of commercially pure and highly pure tantalum were incipiently spalled and recovered. Image analysis and optical profilometry techniques were used to study the recovered samples. The obtained results include statistical analyses of the void diameters, void volume fraction and its distribution with respect to the primary spall plane.

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QUANTITATIVE MICROSTRUCTURAL DAMAGE EVOLUTION IN COPPER UNDER SPALLATION CONDITIONS: *W. Richards Thissell*¹; *Anna K. Zurek*¹; *Jesus M. Rivas*¹; *Davis L. Tonks*²; *Robert S. Hixson*³; ¹Los Alamos National Laboratory, MST-8: Structure-Property Relationships, G755, Los Alamos, NM 87545 USA; ²Los Alamos National Laboratory, XNH: Nuclear and Hydrodynamic Applications, B216, Los Alamos, NM 87544 USA; ³Los Alamos National Laboratory, DX-1: Detonation Science and Technology, MS:P952, Los Alamos, NM 87545 USA

This paper quantitatively describes the damaged microstructures resulting from recovered incipient spallation experiments performed on 10100 OFHC. The starting material was annealed in vacuo at 600°C for one hour and has a mean grain size of 34 μm for all of the tests. We performed all of the experiments in a gas gun with identical flyer and target geometries, but at varying flyer plate velocities. Hence, the significant variable is the compressive and tensile stress amplitudes. The quantitative analysis combines two-dimensional image analysis with optical profilometry of metallographic sections cut normal to the incipient failure surface. The quantified microstructural parameters include void size (true and cross-sectional), position, aspect ratio, void clustering, void linking, and porosity versus position. We present statistical analyses of these parameters and describe the effect of stress pulse amplitude on the evolution of these parameters.

11:00 AM

INVESTIGATING THE ROLE OF VOID DAMAGE CLUSTERING IN DYNAMIC FRACTURE: *Michael J. Worswick*¹; *A. Keith Pilkey*¹; *Ahmed Kamal Kamel*¹; *Gang Liu*¹; *Pierre Pelletier*²; *Patrick J. Gallagher*³; *Denis Nandlall*⁴; ¹Carleton University, Mechanical and Aerospace Engineering, 1125 Colonel By Drive, Ottawa, Ontario K1S 5B6 Canada; ²SNC Industrial Technologies, 5 Montee des Arsenaux, LeGardeur, Quebec J5Z 2P4 Canada; ³Defence Research Establishment Suffield, Box 4000, Medicine Hat, Alberta T1A 8K6 Canada; ⁴Defence Research Establishment Valcartier, 2459 Pie XI Blvd. North, Courcelette, Quebec G0A 1R0 Canada

The role of void damage clusters during dynamic fracture is investigated on the basis of an experimental programme which combines high rate material testing with quantitative metallographic analyses of tested specimens. The nominal strain rate regime in this study is

2000/s, provided by a tensile split Hopkinson bar (TSHB) facility at Carleton University. Two very dissimilar materials are considered, namely free-machining brass and Armco iron, thereby facilitating an examination of clustering effects in the presence of many (brass) versus few (iron) void nucleation sites. For each material, several interrupted TSHB tests are conducted to obtain deformed microstructures at various strain states just below that of fracture. Spatial tessellation techniques are subsequently applied to large-scale high-resolution digital images of the potential fracture region in order to establish distinguishing characteristics of void clustering as damage evolves with applied strain. The significance of these stereological clustering measures in the prediction of dynamic fracture is supported by the results of a complementary numerical model which employs a characteristic damage length scale in its formulation.

11:20 AM

DYNAMIC DEFORMATION AND FRACTURE OF SHIP STEEL: *Gersom Pape*¹; ¹Delft University of Technology, Laboratory of Materials Science, Rotterdamseweg 137, Delft, - 2628 AL The Netherlands

The dynamic deformation of steel Fe E 355 for marine applications, along with the dynamic fracture process is the subject of this research work. For the prediction of the failure behaviour of dynamically loaded structures, several experiments are being done to examine the dynamic and static fracture characteristics for this kind of steel. For this purpose different geometries of tensile specimen will be considered to investigate the influence of the stress-triaxiality on fracture initiation. Finite element calculations will be used to determine the stress-triaxiality in the plastically deforming specimen. In these calculations an appropriate local fracture criterion predicts the onset of failure. To investigate the influence of the strain rate on the fracture initiation process, the experiments are being carried out at several strain rates, varying between approximately 1e-4 and 1e+2 per second. Use is made of a servo-hydraulic high rate single shot testing machine. Results of tensile tests at low and high strain rates will be presented along with some numerical results of calculations of the experiments.

11:40 AM

SPALL STRENGTH OF METALS AT HIGH TEMPERATURE AND HIGH PRESSURE: *Andrey Bogach*¹; ¹Institute of Chemical Physics, Combustion and Explosive, Institutskiy pr. 14, Chernogolovka, Moscow 142432 Russia

The spall strength of metals is known to have a threshold dependence on temperature [1] and peak pressure [2] and depends on the strain rate in unloading part of shock pulse as power function with small power index 1/m [1]. It is shown in present work that the power function take place for viscoplastic material, in which viscose stress depends on the strain rate as power function with power index 1/m, and is directly connected to dynamic resistance of material to dislocation motion. The viscoplastic dissipation of deformation energy results to localized heating in vicinity of pore. In assuming of localized melting for these viscoplastic material the expressions for threshold temperature and for dependence of spall strength on temperature were received. According to the solution the threshold temperature and the spall strength have the same dependence on the stress gradient in shock pulse. In assuming that shock-induced and initial heating is equivalent the model gives the spall strength dependence on peak pressure. The dislocations density in vicinity of pore was estimated from experimental results of the threshold temperature and the spall strength for Al,Mg,Mo. 1. Kanel G.I.,Razorenov S.V.,Bogach A.A.,Utkin A.V.,Fortov V.E., J.Appl.Phys.79,8310-8317(96). 2. Kanel G. I., Razorenov S.V., Utkin A.V., Grady D.E. ShockCompression of condensed metter, Seattle, USA, 1995

EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Stress & Microstructure; and Poster Talks

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Wednesday AM Room: International Ballroom
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Peter Voorhees, Northwestern University

8:30 AM INVITED PAPER

THERMODYNAMICS AND STRESS: *Francis C. Larchie*¹; ¹University Montpellier, Materials Science and Technology, GDPC - Case 26, Montpellier, Cedex 5 34095 France

Our work with J.W. Cahn on the thermodynamics of stressed solids is first put in an historical perspective through a brief review of the literature since J.W. Gibbs. It is shown that the basic thermodynamics is the same; the various theories differ mostly through the models used to describe the physics of the system. The assumptions of our model are then examined in detail, in order to test its merits and limitations in representing the behavior of real solid materials. Possible extensions are presented or suggested. We also consider the question of macroscopic behavior (at a scale large compared to the microstructure) and see how this change of scale can be translated into the thermodynamic results.

9:15 AM INVITED PAPER

STRESS EFFECTS IN MICROSTRUCTURE EVOLUTION: *Perry H. Leo*¹; ¹University of Minnesota, Aerospace Engineering, 107 Akerman Hall, 110 Union St. SE, Minneapolis, MN 55455 USA

We study the effects of elastic stress on the evolution of precipitate-matrix microstructures in binary alloys. The precipitate and matrix are both assumed to obey anisotropic linear elasticity, although they can have different elastic constants and possibly different symmetries. Stresses arise from misfit between the phases and from applied loads. Both sharp interface (boundary integral) and diffuse interface (Cahn-Hilliard) methods are used to track the evolution of the precipitates. Results from both methods show that elastic stress has a strong influence on precipitate shapes and interactions as well as on the overall growth and coarsening behavior of the alloy. Further, relatively small difference in the elastic constants of the phases can play an important role in this evolution, suggesting that modeling elastic inhomogeneities is crucial in simulating the microstructure of alloys such as nickel based superalloys.

10:00 AM BREAK

WEDNESDAY AM

10:20 AM INVITED PAPER

GRAIN BOUNDARY FACETING AND DIGM: *Duk Yong Yoon*¹; ¹Korea Advanced Institute of Science and Technology, Materials Science and Engineering, 373-1 Kusungdong, Yuseonggu, Taejeon 305-701 Republic of Korea

Grain Boundaries in a variety of crystals undergo faceting-defaceting transition at critical temperatures which strongly depend on the impurity content. In a relatively pure polycrystalline Ni, for example, all grain boundaries are faceted at temperatures below 0.8 T_m (where T_m is the melting point) in a carburizing atmosphere. According to the analysis of Herring, Frank, Cabrera, and Cahn, any grain boundary can be faceted if the grain boundary energy varies strongly with the inclination angle. A faceted grain boundary can exhibit zig-zag migration behavior during DIGM, because the direction of DIGM is determined by the relative coherency strain energies at the boundary segments of the pair grains. Under certain conditions grain boundaries undergoing DIGM may develop faceting. Critical experiments have been performed to show that the driving force for the migration of grain boundaries and intergranular liquid films during alloying (and dealloying) and discontinuous precipitation is the diffusional coherency strain energy. The theoretical analysis of Cahn, Fife, and Penrose is consistent with these observations.

11:05 AM

PHASE FIELD COMPUTATIONS USING AN ADAPTIVE GRID TECHNIQUE: *N. Provatas*¹; *N. Goldenfeld*¹; *Jon Dantzig*¹; ¹University of Illinois, Mechanical and Industrial Engineering, 1206 West Green Street, Urbana, IL 61801 USA

In recent years, the phase field technique has been developed to model evolution of solidification microstructures. In this method, the liquid-solid interface is modeled as a diffuse region whose thickness is characterized by an order parameter, known as the phase field. One of the difficulties encountered when applying the phase field method is the conflicting requirements of high resolution needed to successfully capture the physical phenomena at the interface, and the simultaneous need to fully resolve the diffusion field ahead of the advancing front. This has led to the use of very dense grids, with correspondingly long computation times. Further, the calculations have been limited to the regime of high growth rates, where the diffusion field is relatively small. In this work, we describe an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion field.

11:10 AM

COMPUTER SIMULATIONS ON THE PHASE TRANSFORMATION IN REAL ALLOY SYSTEMS BASED ON THE PHASE FIELD METHOD: *Toru Miyazaki*¹; *Toshiyuki Koyama*¹; ¹Nagoya Institute of Technology, Dept. of Materials Science and Engineering, Gokisocho, Showa-ku, Nagoya 466 Japan

The kinetic simulation based on the non-linear evolution equation become very powerful method in fundamental understanding the dynamics of phase transformation with the recent remarkable development of computer. In the present study, we calculate the dynamics of microstructure changes in real alloy systems, such as Fe-Al-Co, Fe-Si-V, Fe-Mo, Fe-Cr and Al-Zn based on the phase field method which is a new methodology to simulate the microstructure development by combining the Cahn-Hilliard diffusion equation and Cahn-Allen equation. The atomic interaction parameters, elasticity, and mobility are assumed to depend on local order parameters (composition, degree of order, etc) to calculate the phase transformation which is consistent with the equilibrium phase diagram of the actual alloys. Time dependent morphological changes of the microstructure (the formation of modulated structure by spinodal decomposition, strain induced morphological changes of precipitates, the interaction between the order-disorder phase transition and phase separation, discontinuous precipitation, and stable phase precipitation after the metastable phase decomposition, etc) will be demonstrated. The results simulated are quantitatively in good agreement with the experimental ones in the real alloy systems.

11:15 AM

INTERFACIAL STUDIES OF TERNARY ALLOYS: *Monica Olvera de la Cruz*¹; ¹Northwestern University, Dept. of Materials Science and Engineering, Evanston, IL 60208-3108 USA

We study interfaces of A-B-C ternary alloys decomposed into two and three phases. The effect of the gradient energy coefficients on the interface profiles of ternary alloys is examined. The interface profiles of ternary alloys are obtained numerically solving the steady-state solutions of the nonlinear spinodal decomposition equations. We also determine the composition profiles solving the two Euler-Lagrange equations resulting from minimizing the interfacial energy.

11:20 AM

THERMAL DRAG OF THE ANTIPHASE DOMAIN BOUNDARY MOTION: *Alexander Umantsev*¹; ¹Saint-Xavier University, Science Department, 3700 West 103rd Street, Chicago, IL 60655 USA

Antiphase domain boundary (APB) is a region in materials where ordering of atoms changes from one structural variant to another. Due to the surface tension, APB's constitute a network of structural defects which is in global disequilibrium with the crystal. Cahn-Allen's analysis of the structural coarsening of this network [Acta Metall, 27, 1085 (1979)] shed the light on the nature of the driving force in this problem: the free-energy change, if the material stays isothermal. We explore the influence of the internal energy transport on the dynamics of such boundary in the framework of the Onsager theory of linear response. The internal energy transport entails temperature humps in the transition regions and causes a drag effect as a consequence of the driving force reduction, implying that coarsening of the APB structure is not possible without heat conduction in the material. An evolution equation that takes into account the finite thermal conductivity is derived. Experimental setup to reveal the thermal drag is suggested.

11:25 AM

SIMULATING MICROSTRUCTURAL EVOLUTION IN MULTI-PHASE AND MULTI-DOMAIN MATERIALS USING PHASE FIELD METHOD: *Yunzhi Wang*¹; ¹The Ohio State University, Materials Science and Engineering, 2041 College Road, Columbus, OH 43210 USA

Many technologically important material systems are inherently heterogeneous. Structurally, they are multi-phase and multi-variant/domain aggregates whose morphology determines the important physical and mechanical properties. In this presentation, recent development of the continuum phase field method in simulating microstructural evolution in martensitic crystals and Ni-based superalloys will be discussed. The simulation models explicitly take into account the interactions between different orientation variants of the martensitic phase and the different antiphase domains of the L12 ordered phase in the elastic strain field generated by lattice misfit. Results of both 2D and 3D simulations illustrating the effect of these interactions on the kinetic and morphology of the multi-phase and multi-variant/domain microstructure development in these systems will be discussed. Also, the recent adoption of the phase field method to simulating the microstructural evolution and electrical conduction of ceramic thin film sensors will be presented. In this approach, detailed microstructural features developing during sintering, which are simulated using the phase field method, have been directly used in the mesoscopic resistor-lattice model for electrical conductivity calculations. Combination of these methods provides a direct link between the critical microstructural features developing during thermal processing and the electrical conducting behavior of the material. This work is supported by the National Science Foundation.

11:30 AM POSTER VIEWING

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Environmental and Surface Aspects of Fatigue

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; Daniel Eylon, Univ. of Dayton, Graduate Materials Engineering, Dayton, OH 45469-0240; J. P. Gallagher, University of Dayton, UDRI-Structural Integrity, Dayton, OH 45469-0120 USA; G. Lutjering, TU Hamburg-Harburg, Hamburg 21093 Germany

Wednesday AM Room: Athens/Berlin
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: P. Bowen; A. P. Woodfield

8:30 AM KEYNOTE

EFFECT OF ENVIRONMENT ON HIGH-TEMPERATURE FATIGUE OF IMI 834: O. Schauerte¹; A. Gysler¹; G. Lutjering¹; S. Mailly²; Y. Chabanne²; C. Sarrazin-Baudoux²; J. Mendez²; J. Petit²; ¹Technische Universität Hamburg-Harburg, Eissendorfer Str. 42, Hamburg, FRG 21073 Germany; ²L.M.P.M. - E.N.S.M.A., Site du Futuroscope - Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France

The fatigue properties of the Ti-alloy IMI 834 at high temperature (500°C) will be compared in air as an aggressive environment and in vacuum as an inert reference environment. This comparison includes the environmental effect on fatigue crack initiation in the LCF regime, as well as on the fatigue crack propagation behavior of self-initiated microcracks and in addition on the propagation behavior of long through cracks (macrocracks). Two different microstructural conditions will be considered, pure lamellar structures and bimodal microstructures, consisting of equiaxed primary α -phase in a transformed lamellar matrix. The characterization of the macrocrack propagation behavior includes the effects of mean stress variations and crack closure effects for both environmental conditions. The discussion will be focused on the environmental effects on the crack propagation mechanisms by detailed fracture surface studies and on variations in the through-thickness crack front profile appearances. The usually observed pronounced reduction in the resistance against high temperature fatigue crack nucleation as well as crack propagation in the aggressive air environment in comparison to vacuum will be discussed on the basis of hydrogen embrittlement caused by the water vapor in air environment.

9:00 AM INVITED PAPER

ENVIRONMENTAL INTERACTIONS IN HIGH TEMPERATURE FATIGUE CRACK GROWTH OF TITANIUM ALLOYS: J. Petit¹; C. Sarrazin-Baudoux¹; Y. Chabanne¹; G. Lutjering¹; G. Gysler¹; O. Schauerte¹; ¹L.M.P.M. - E.N.S.M.A., Site du Futuroscope - Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France

Titanium alloys are interesting to aerospace designers because of their low density, high specific strength and corrosion resistance. They are often used under cyclic conditions in aggressive environments such as moist air and at more and more elevated temperature levels. The present paper is addressing a study of the specific influence of atmospheric gaseous species (i.e. water vapor and oxygen) at 500°C on the fatigue cracking behavior of Ti6246 and IMI 834 Titanium alloys. A first comparison of the crack growth rates in air and in high vacuum confirms the existence of a detrimental effect of ambient environment which becomes more accentuated at high temperature. Complementary tests carried out at 500°C under selected environmental conditions as dry air, dry Argon and controlled low pressures of water vapour, demonstrate that the enhancement of the

fatigue crack growth rates in the near-threshold area observed in active environments in comparison with high vacuum is clearly related to the presence of water vapor even at very low partial pressure of about 1Pa. However comparison between growth data under atmospheric pressure of dry air and dry Argon containing few ppm of water vapor, suggests a complex interaction between oxygen and water vapour. Scanning electron microscope observations are investigated in order to identify the failure modes with respect to the microstructure of the specimens, and to precise the respective role of water vapor embrittlement and of oxidation. The contribution of crack closure is evaluated and the respective role of roughness induced closure and oxide wedging is discussed. A detailed analysis of the fatigue crack propagation resistance of the Ti6246 alloy is conducted at various loading frequencies ranging from 35Hz to 5x10⁻³ Hz. According to environment, loading conditions and test frequency, the respective contribution of potential time dependent damage cracking mechanisms assisted by water vapour, i.e. corrosion-fatigue and creep-fatigue, is discussed.

9:20 AM INVITED PAPER

EFFECT OF MECHANICAL SURFACE TREATMENTS ON FATIGUE PERFORMANCE OF TITANIUM ALLOYS: L. Wagner¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Mechanical surface treatments such as shot peening, roller-burnishing and deep-rolling are widely used to improve the fatigue performance of titanium alloys. Generally, these treatments induce high dislocation densities in near-surface regions. Due to the local plastic deformation, residual stresses are developed and the surface topography is changed. In this overview, the effect of the various process parameters, e.g., Almen intensity, burnishing pressure and rolling force on the change in surface properties and resulting fatigue performance will be presented for the various titanium alloy classes, i.e., α , ($\alpha+\beta$) and β alloys. Results will be compared with electrolytically polished references. The different response of the various titanium alloy classes to mechanical surface treatments will be discussed on the basis of the degree of the cyclic stability of process induced residual compressive stresses and their influence on the microcrack growth.

9:40 AM

ROLLER-BURNISHING TO IMPROVE FATIGUE LIFE IN BETA TITANIUM ALLOYS: A. Berg¹; A. Drechsler¹; L. Wagner¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Roller-burnishing is a common procedure for improving the fatigue performance of structural components which have a rotational symmetry, such as axles and shafts. Compared to shot peening, greater depths of induced plastic deformation combined with a much smoother surface finish can be achieved. In the present work, the effect of roller-burnishing on the change in surface layer properties and the resulting fatigue performance was studied on the metastable β titanium alloys Ti-10V-2Fe-3Al and Ti-3Al-8V-6Cr-4Mo-4Zr. On both alloys, suitable thermomechanical treatments were performed to cover conditions with low, intermediate and high strength levels. Hour-glass shaped specimens were roller-burnished in a conventional lathe using a hydraulic device by which a hard metal ball (6 mm diameter) is pressed on the surface of the specimen. For optimizing the fatigue response working pressures ranging from 25 to 400 bar were used. Induced dislocation densities and residual stresses were measured by X-ray diffraction techniques. Fatigue tests on the various roller-burnished conditions as well as on electrolytically polished references were performed in rotating beam loading ($R = -1$). Fatigue results will be discussed in terms of the effects of induced dislocation density and residual compressive stress on fatigue crack nucleation and microcrack growth.

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FATIGUE CRACK GROWTH OF TITANIUM ROTOR ALLOYS IN VACUUM AND AIR: R. Craig McClung¹; B. H. Lawless¹; M.

Gorelik¹; ¹Southwest Research Institute, Manager, Materials Integrity, P.O. Drawer 28510, San Antonio, TX 78228-0510 USA

A probabilistic damage tolerance design code for life management of aircraft turbine engine rotors is being developed under a major FAA grant (95-G-041), with an initial focus on fatigue cracking at hard alpha defects in titanium. Since HA defects are usually subsurface, any resulting cracks are embedded and hence isolated from the atmosphere (i.e., vacuum-like) for at least some of their life. FCG tests are conducted in vacuum at various temperatures for Ti-6-4 and Ti-6-2-4-2 rotor alloys, and results are compared with existing air data. The new design methodology is probabilistic in order to address uncertainties associated with the size, shape, and rate of occurrence of HA defects, as well as uncertainties in rotor inspection, stresses, and material properties. Replicate FCG data for titanium rotor alloys are analyzed statistically to characterize the FCG rate variability that could be significant for life calculations.

10:20 AM BREAK

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MODELING THE DAMAGING EFFECTS OF GASES ON THE FATIGUE CRACK GROWTH OF AN $\alpha+\beta$ TITANIUM ALLOY: *Stephen W. Smith¹; Robert S. Plascik¹; ¹NASA-Langley Research Center, MS 188E, NRC Resident Research Associate, Hampton, VA 23681-0001 USA*

The effects of air, oxygen, nitrogen, and water vapor on the fatigue crack growth behavior of an $\alpha+\beta$ titanium alloy (Ti 6-22-22, Ti - 6 Al - 2 Zr - 2 Cr - 2 Sn - 2 Mo) were evaluated to determine the mechanisms resulting in environmentally assisted fatigue. Tests were performed in laboratory air, ultrahigh vacuum, and high purity gaseous environments at various partial pressures for temperatures ranging from room temperature to 177°C. Accelerated fatigue crack growth rates (five to ten times greater) have been measured in laboratory air when compared to ultrahigh vacuum. Oxygen and water vapor are identified as the damaging species present in laboratory air. Fatigue crack growth tests were performed at constant ΔK , frequency, and stress ratio in high purity oxygen and water vapor environments at pressures ranging from ultrahigh vacuum ($\sim 9 \times 10^{-8}$ Pa) to atmospheric pressure at sea level ($\sim 1 \times 10^5$ Pa). From these tests, distinct transitions in the fatigue crack growth behavior have been observed. These transitions are related to three different mechanisms which affect fatigue crack growth at various pressure ranges. In low pressure oxygen and water vapor, an adsorption assisted damage process is suggested. This adsorption assisted process results in increasing fatigue crack growth rate with increasing pressure. A critical pressure is reached where a monolayer of adsorbed species is produced during each fatigue cycle. For pressures greater than the critical pressure, a crack-tip chemisorbed film and/or oxide is produced resulting in a decrease in fatigue crack growth rate with further increase in pressure. While the formation of this film results in decreased fatigue crack growth rates compared to the adsorption assisted regime, fatigue crack growth rates are accelerated compared to ultrahigh vacuum rates. A third damage mechanism is suggested for exposure to water vapor; here, increasing crack growth rate with increasing pressure is consistent with a hydrogen embrittlement mechanism.

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INTERFACE BETWEEN FATIGUE, CREEP, AND STRESS-CORROSION CRACKING ON Ti6246 TITANIUM ALLOY: *C. Sarrazin-Baudoux¹; Y. Chabanne¹; J. Petit¹; ¹L.M.P.M.-E.N.S.M.A., Site du Futuroscope-Chasseneuil-du-Poitou, B.P. 109, Futuroscope Cedex 86960 France*

Failure of structural materials due to cracking under coupled actions of mechanical stressing and environmental attack remains a safety and economical problem despite the effort that has been devoted to understand the cracking phenomena induced by stress-corrosion, corrosion-fatigue or creep-fatigue. Titanium alloys for aerospace applications are often used under a combination of cyclic and constant amplitude loading in aggressive environments such as moist

air, and at elevated temperature levels (up to 600°C). Then a well defined characterization of these alloys is required in order to ensure a good damage tolerance. The present paper is addressing a study of the influence of the mean stress on the fatigue crack growth behavior of a Ti6246 alloy, and is specially concentrated on the near-threshold condition at room temperature and 500°C. Threshold tests are performed at frequencies of 0.1 to 35 Hz and at a constant K_{max} level, which means increasing R ratio with decreasing ΔK ensuring the absence of closure during the threshold procedure. At room temperature and very low ΔK , it is shown that for a critical K_{max} level of about 70% to 80% of the fracture toughness, no threshold for crack propagation is reached and for ΔK range lower than 1.5 MPa m the growth rates appear to become time dependent instead of cycling dependent supporting a creep governing mechanism. At 500°C, tests performed in air and in humidified argon support a detrimental effect of water vapor consistent with a corrosion-fatigue mechanism in the mid-rate range and an additional process of stress-corrosion in the near threshold area. According to environment, loading conditions and test frequency, the respective contribution of the two involved competitive cracking mechanisms assisted by water vapor is discussed. Stress corrosion is shown to interact with corrosion-fatigue specially when the stress intensity factor range is lower than 2 MPa m and conjugated with K_{max} higher than 22 MPa m (rumpling tests). Such conditions of stress localization and environmental assistance can induce a failure by stress corrosion cracking for stress intensity level of about 40% of the fracture toughness. Scanning electron microscope observations and crack surface analysis are investigated in order to identify the failure modes and to precise the respective role of water vapor embrittlement and of oxidation with respect to the loading conditions.

11:20 AM

SLOW FATIGUE TESTING OF TITANIUM GRADE 29 IN AIR AND SEA WATER: *Lyder Atteraa¹; Gisle Hersvik¹; Haavard Solbakken¹; ¹University of Bergen, Applied Physics and Technology, Allegaten 55, Bergen N-5007 Norway*

Welded and unwelded specimens of ASTM Grade 29 titanium has been sinusoidally fatigue tested in air and seawater at 110°C at 0.2 Hz. The possible influence of seawater is completely masked by the fact that all the specimens of welded titanium, and most of the parent metal samples, had internal fracture initiations. A high maximum cyclic stress leads to a low fatigue life of the parent (unwelded) metal. Increasing the maximum stress from 75 to 85 % of the yield stress (at 110°C), the fatigue life is lowered by a factor of 3.6. Compared with the parent metal, the welded titanium has a significantly lower fatigue life. At 110°C, the fatigue life of weldmetal was reduced by ca. 93 % at the high stress level ($s_{max} = 0.85s_y$), and ca. 98 % at the low stress level ($s_{max} = 0.75s_y$). This drop in fatigue life is attributed to the presence of pores in the weld deposits. Fractographic studies indicate that the fracture initiation takes place in the material immediately surrounding a pore, with an abrupt crack formation, marking the beginning of the crack growth phase. The duration of the crack initiation phase shows large variations, whereas the growth phase duration varies little.

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FATIGUE OF TITANIUM GR. 29 UNDER CATHODIC "PROTECTION" IN HOT WATER: *Lyder Atteraa¹; Kjetil Foer¹; ¹University of Bergen, Applied Physics and Technology, Allegaten 55, Bergen N-5007 Norway*

Titanium Grade 29 has been tested in sinusoidal fatigue. The specimens were taken from a 340 mm pipe. To simulate a surface crack the specimens were notched. The results were obtained at 110°C. The testing was performed in air and in natural seawater. In water, specimens were subjected to three different corrosion potentials: Free corrosion potential, -1050 mV_{Ag/AgCl} and -1500mV_{Ag/AgCl}. The maximum nominal stress for all four series was 552 MPa (i.e. $0.75 * \sigma_y$ at 110°C) and the stress ratio was 0.1. The air testing frequency was 3 Hz, and the seawater test frequency 0.2 Hz. In each series 5 specimens were coupled in a column and tested together in

order to reduce the testing time. The post fracture examinations showed that all the fractures were initiated along the specimen surface, meaning that when submerged, seawater penetrated the crack from the start of the fatigue process. No significant reduction of the notched fatigue life due to seawater cathodic protection was found, but a tendency to a reduced fatigue life in the three seawater series was seen. The specimens experienced local plastic deformation in the notch during the fatigue testing. 10 of 21 specimens had more than one initiation site, at or close to the surface of the specimens. All fractures were transgranular and contained many cleavage-like facets near the ductile area. Fatigue testing in air showed that the fatigue life was reduced by 98% for the notched specimens compared to smooth specimens, when ignoring the elastic stress concentration factor. However, the static tensile strength was 60% higher in the notched specimens than in the unnotched specimens.

12:00 PM

EFFECTS OF TEMPERATURE AND FREQUENCY ON FATIGUE CRACK GROWTH RATE OF Ti-6Al-4V-0.1Ru IN AN OCEAN ENVIRONMENT: *Morten A Langoy*¹; *Stuart R. Stock*¹; ¹Georgia Institute of Technology, School of Materials Science and Engineering, 778 Atlantic Dr., Atlanta, GA 30332-0245 USA

The technological challenges associated with development of vessels for offshore oil production are considerable, and use of titanium for dynamically loaded production risers offers important economic advantages. However, limited experience exists with titanium risers. Recognizing that defects of some kind are inevitable in these types of components and that crack initiation will be only a minor portion of the lifetime. Hence, this study focuses on fatigue crack growth. In the offshore industry a fail safe design approach is commonly employed, and in this respect the synergic effects of temperature and frequency in ocean water is of interest. In titanium a unusual frequency cross-over effect has been reported (at low stress intensities higher frequencies promote more rapid fatigue crack growth while at high stress intensity, the converse is true). This paper reports the influence of temperature on the effect of frequency on fatigue crack growth in Ti-6Al-4V-0.1Ru.

systems. Heterogeneous nucleation occurs at the interface between the primary phase substrate and the liquid phase. During extensive thermal cycling, distinct changes in the nucleation behavior have been observed. These changes reflect transitions in the density of specific nucleation sites and can be correlated to a faceting tendency of the nucleating substrate during cycling, as observed by Scanning Electron Microscopy (SEM). Since it is expected that the development of smooth liquid-solid interfaces at the micron scale is reflected at the atomic scale in a similar structural configuration, the removal of large substrate features during consecutive cycles is anticipated to result in the removal of the more potent nucleation sites, exposing previously concealed sites. With controlled thermal cycles on drop-let samples as well as isothermal holding treatments, the nucleation temperatures can be correlated to particular sites on the primary substrate. In order to determine the catalytic potency of the investigated nucleation sites and to resolve closely spaced exotherms, a modification of the isothermal nucleation rate expression has been utilized to simulate the continuous cooling approach commonly used in heterogeneous nucleation analysis. The support of the NSF (DMR-9712523) is gratefully acknowledged.

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SOLUTE EFFECTS IN BAINITE IN Fe-C-X STEELS: *R. E. Hackenberg*¹; *D. G. Granada*¹; *G. J. Shiflet*¹; ¹University of Virginia, Department of Materials Science and Engineering, Thornton Hall, Charlottesville, VA 22903

Several ternary Fe-C-X steels were investigated with the goal of better understanding the role that the alloying element X plays in determining the kinetics and morphology of the bainite reaction. Particular attention has been paid to the "bay" region on the T-T-T diagram, in which the start curve passes through a minimum. The existence of a bay in Fe-C-W alloys is documented for the first time, and morphological similarities with Fe-C-Mo alloys are observed. This result is consistent with the theory that a solute drag-like effect (SDLE) accounts for the kinetic and morphological transitions seen in the bay region. The existence of SDLE in selected Fe-C-Mo alloys is tested with analytical TEM to determine the distribution of solute in and around the growth front. Particular attention is paid to the time-temperature regions (above the bay) where growth stasis is known to occur. Early results from this study will be presented, and attention will be given to the role played by carbide precipitation. This research was supported by the National Science Foundation.

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IDENTIFICATION OF THE SOLUTE DIFFUSION PATHS AT THE REACTION FRONT IN LAMELLAR PHASE TRANSFORMATIONS: *C. R. Hutchinson*¹; *G. J. Shiflet*¹; ¹University of Virginia, Department of Materials Science and Engineering, Thornton Hall, Charlottesville, VA 22903

Modeling of growth kinetics in lamellar phase transformations usually assumes that a high diffusivity path is employed for substitutional solute redistribution at the reaction front. Previous research in ternary Fe-C-X pearlite using analytical microscopy has not been of sufficient spatial resolution to confirm the diffusion path. In addition to ferrous pearlite transformations, the cellular transformation in alloys, such as Cu-Ti, has also been investigated. Although a precipitation reaction, the synchronous growth of the precipitate phase and depleted matrix results in a similar reaction front to that observed in pearlite. Mathematically modeling this phase transformation also incorporates boundary diffusion of the solute element and closely follows the approach for pearlite. To obtain chemical information, High Resolution Analytical Electron Microscopy has been used to critically evaluate alloy content in the various interfaces associated with these two lamellar phase transformations as a function of temperature. Concentration profiles in the vicinity of the reaction front and the interlamellar interphase boundaries will be reported. These chemical profiles will be correlated with the interfacial structure. The authors gratefully acknowledge support from the National Science Foundation.

GENERAL ABSTRACTS: Phase Transformations

Sponsored by: TMS

Program Organizers: TMS, Warrendale, PA 15086 USA; David Bourell, University of Texas, Austin, TX 78712-1063 USA; Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Howard Sizak, Inco Alloys International, Mechanical Testing, Huntington, WV 25705

Wednesday AM Room: Zurich/Tokyo
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Zi Kui Liu, Questek Innovations, Evanston, IL 60201

8:30 AM

HETEROGENEOUS NUCLEATION BEHAVIOR AS A FUNCTION OF SUBSTRATE MORPHOLOGY: *Peter G. Höckel*¹; *John H. Perepezko*¹; ¹University of Wisconsin - Madison, Materials Science and Engineering, 1500 Engineering Drive, Madison, WI 53706 USA

The droplet emulsion technique (DET) and Differential Scanning Calorimetry (DSC) have been used to investigate the undercooling and nucleation behavior of powder samples from several binary alloy

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A KINETICS MODEL FOR CONTINUOUS COOLING TRANSFORMATION AND ITS APPLICATION TO PLAIN CARBON STEELS: *Choo Se Don*¹; Choo Wung Yong¹; ¹Pohang Iron & Steel Co., Technical Research Labs, Pohang P.O.Box 36.1, Koedong-dong, Nam-ku, Pohang-shi, Kyungbuk 790-785 South Korea

A new model was suggested to describe the kinetics of the austenite-to-ferrite phase transformation during continuous cooling. It was based on the simplified Fe-C phase diagram and some thermodynamic theories were considered. The final equation derived was as follows; $\ln(-\ln(1-X))=K\alpha t + \text{constant}$ where, X is the transformed volume fraction, K is a kinetic parameter, t is the time and α is the cooling rate. It is somewhat different from the Avrami type equation which is for isothermal transformation behaviors. Using a dilatometer the phase transformation has been investigated in a kind of plain steels with compositions C:0.05~0.2, Mn:0.5~2.0, Ti:0.01 under the various cooling rate 0.2°C/sec up to 30°C/sec. The Experimental data were statically treated and the kinetic parameter K in the above equation was revealed to depend only on the chemical compositions. The predicted time-transformation curves using the above equation and the measured ones were compared and reasonably good agreement was obtained. The equation has been integrated into a full microstructural model which has been applied to prediction of mechanical properties. It was found that the predicted values of tensile strength of plates processed under various manufacturing were in reasonable agreement with the measured ones.

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PHASE EQUILIBRIA AND REACTIONS IN THE Ni-Bi SYSTEM: *Chengheng R. Kao*¹; M. S. Lee¹; C. M. Liu¹; ¹National Central University, Dept. of Chemical Engineering, Chungli, Taiwan ROC

The phase equilibria and the reactions between Ni and liquid Bi were studied. It was found that the only intermetallic compound formed in the reactions was NiBi₃. The other intermetallic compound NiBi, which is thermodynamically stable at these temperatures, did not form. Reaction at 300 C produced a thick reaction zone, which is a two-phase mixture of NiBi₃ needles dispersed in Bi matrix. The thickness of the reaction zone increased rapidly with reaction time, reaching 400 microns after 360 minutes. Reactions at 360 and 420 C produced very thin reaction zones, and the major interaction was the dissolution of Ni into liquid Bi. Reaction at 480 C produced extremely thin reaction zone, and the dissolution of Ni into liquid Bi was very fast and was the major interaction. It is proposed that the formation of the reaction zone is controlled by two factors: the solubility limit and the diffusivity of Ni in liquid Bi. Small diffusivity and small solubility limit, i.e. lower temperature, tend to favor the formation of thin reaction zone. In addition to the NiBi₃ formed within the reaction zone, NiBi₃ also formed outside the reaction zone as long needles with hexagonal cross-section. It is believed that these NiBi₃ needles formed during the solidification of liquid Bi.

10:35 AM BREAK

10:50 AM

A TEM STUDY OF THE EFFECT OF COHERENCY STRAIN ON THE COARSENING RATE OF THE GAMMA' PRECIPITATES IN NICKEL BASE SUPERALLOYS: *Michael J. Pollard*¹; Steve L. Weber¹; *Haydn H. Chen*¹; ¹University of Illinois at Urbana-Champaign, Materials Science and Engineering, 1304 W. Green, Urbana, IL 60565 USA

The objective of this study was to determine the coarsening rate of γ' precipitates in several alloys of the Ni-Al-Si system in the composition region near complete coherency in order to observe the effect of coherent misfit strain on coarsening rates. Samples with total nickel concentration fixed at 88 at%, but with varying Al/Si ratio, were aged at 550°C and 600°C for 2, 4, 6, 8, 10, and 12 hours. The samples were then viewed in a TEM in the dark field of a superlattice spot to see the precipitate structure. The micrographs were scanned and analyzed to obtain the average size of the precipitates versus time for each composition and temperature, and there-

fore the coarsening rate. The coarsening rates for these compositions were compared to the results of the study of the same alloys by Muralidharan and Chen (SANS), who demonstrated that small misfit strains in either the positive or negative direction caused increases in coarsening rate. The TEM study was able to reproduce the coarsening rate increases on either side of zero misfit.

11:15 AM

SOLIDIFICATION PROCESSING OF HIGH-TEMPERATURE Mo-B-Si ALLOYS: *Ridwan Sakidja*¹; H. Sieber¹; G. Wilde¹; H. Perepezko¹; ¹University of Wisconsin-Madison, Dept. of Materials Science and Engineering, 1509 University Ave., Madison, WI 53705 USA

Some of the microstructural options available in high-temperature Mo-B-Si alloys have been identified in an examination of different solidification processing treatments. In-situ intermetallic matrix composites consisting of Mo solid solution phase dispersed in a Mo₅SiB₂ (T₂) intermetallic phase were produced using arc-casting (AC) and rapid solidification processing (RSP). In conventionally cast Mo-B-Si samples, significant solidification segregation involving the formation of additional high melting phases such as MoB and Mo₅Si₃ was found. These phases showed a very slow dissolution rate even at temperatures as high as 1600C. From the arc-cast sample, the stable (Mo + T₂) two-phase alloys could be attained only after a prolonged subsequent annealing treatment at high temperature. In contrast, the same Mo-B-Si samples produced by means of RSP such as splat-quenching (SQ) and drop-tube processing (DTP) yielded a homogeneous two-phase microstructure. The in-situ formation of the two-phase microstructure directly from the melt allows for new options in the microstructural design of Mo-B-Si alloys. The advantages of using RSP for solidification processing of high temperature alloys will be discussed. In addition, a Mo precipitation reaction that develops in a T₂ phase upon subsequent annealing provides an additional means of microstructural control. The support of AFOSR (F49620-96-1-0286) is gratefully acknowledged.

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EXAMINATION OF SPINODAL DECOMPOSITION USING ANALYTICAL TRANSMISSION ELECTRON MICROSCOPY: *K. J. Doherty*¹; D. J. Li²; G. J. Shiflet¹; S. J. Poon²; ¹University of Virginia, Department of Materials Science, Thornton Hall, Charlottesville, VA 22903 ; ²University of Virginia, Department of Physics, McCormick Road, Charlottesville, VA 22901

The spinodal reaction is commonly observed as a precursor to devitrification of bulk amorphous metal alloys. An objective of this study is to re-examine spinodal decomposition within the miscibility gap in the Nb-Zr system using high-resolution analytical transmission electron microscopy with electron energy loss spectroscopy and small probe energy dispersive spectroscopy. These advanced techniques are employed to physically measure composition profiles along the spinodally transformed <100> directions as a function of time and temperature with the objective of forming three dimensional chemical maps. Following a discussion of the binary alloy results, this procedure is expanded to study the phase separation in a 5-component metastable bcc (β) alloy Ti₆₅Cr₁₃Cu₁₆Mn₄Fe₂ where the spinodal reaction also occurs. The authors acknowledge the Army Research Office for their support.

12:05 AM

A NEW ANALYSIS FOR THE DETERMINATION OF TERNARY INTERDIFFUSION COEFFICIENTS FROM A SINGLE DIFFUSION COUPLE: *Mysore A Dayananda*¹; *Yong-Ho Sohn*¹; ¹Purdue University, School of Materials Engineering, 1289 MSEE Building, West Lafayette, IN 47907 USA

Concentration profiles that develop in a ternary diffusion couple during an isothermal anneal can be analyzed directly for average ternary interdiffusion coefficients. A new analysis is presented for the determination of average values for the main and cross interdiffusion coefficients over selected regions in the diffusion zone from an

integration of interdiffusion fluxes, which are calculated directly from experimental concentration profiles. The analysis is applied to selected isothermal diffusion couples investigated with alpha(fcc) Cu-Ni-Zn alloys at 775°C, beta (bcc) Fe-Ni-Al alloys at 1000°C and gamma (fcc) Ni-Cr-Al alloys at 1100°C. Average ternary interdiffusion coefficients are calculated over composition ranges on either side of the Matano plane and examined for the diffusional interactions among the diffusing components. The ternary interdiffusion coefficients determined from the new analysis are observed to be consistent with those determined by the Boltzmann-Matano analysis at selected compositions in the diffusion zone. The average ternary interdiffusion coefficients are also employed in the analytical solutions based on an error function for the generation of concentration profiles for the selected diffusion couples. The generated profiles are a good representation of the experimental profiles exhibiting up-hill diffusion or zero-flux plane development for the individual components.

HOT DEFORMATION OF ALUMINUM ALLOYS: Process Simulation and Developmet - I

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

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Session Chairs: Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada; E. Nes, Norwegian University of Science and Technology, Department of Metallurgy, Trondheim, N-7034 Norway

8:30 AM INVITED PAPER

MICROSTRUCTURE CONTROL ON AN ALUMINIUM HOT REVERSING FINISHING MILL: *Jurgen Hirsch*¹; Rainer Grenz²; ¹VAW Aluminium AG, R&D, Bonn 53014 Germany; ²Mannesmann Demag AG Metallurgy, Ratingen Germany

With most Aluminium alloys the hot rolling process has a major effect on finished material quality. Special attention must be paid to the process configuration selected and the factors affecting the development of the microstructure and hence metallurgical properties of the rolled strip. The strength and forming properties of the strip depend to a high degree on the complex control of rolling parameters such as temperature, strain, strain rate and interstand time - especially critical in the hot reversing finishing process. By utilizing precise simulation models the effect of recrystallization and cube texture formation during the hot rolling process can be predicted and the corresponding rolling parameters optimized. In combination with powerful process control and automation systems the high product quality standards required, for example, for can body sheet can be achieved. The new hot mill complex comprising a hot reversing roughing mill coupled with a hot reversing finishing mill currently being implemented with Hulett Aluminium in South Africa illustrates the trend towards low capital cost, high-tech "minimill" configurations. The two-stand process configuration has been selected to improve mill productivity, plant flexibility and product quality. The Hulett hot mill concept is used as a case study and reviewed within this paper.

9:00 AM INVITED PAPER

MODELING THE EFFECTS OF FRICTION AND GEOMETRY ON DEFORMATION PATH DURING HOT ROLLING OF ALUMINUM: *D. A. Korzekwa*¹; A. J. Beaudoin²; ¹Los Alamos National Laboratory, Los Alamos, NM 87545 USA; ²University of Illinois Urbana-Champaign, Dept. Mechanical Engineering, Urbana-Champaign, IL 61801 USA

Processing conditions in the hot rolling of aluminum alloys lead to non-uniform deformation within the roll bite. The ratio of slab thickness to arc of contact in the roll gap can vary over a wide range because of the large initial slab thickness and the very large reductions that are possible for thinner slabs. The degree of non-uniform deformation also depends on friction conditions and the rate dependence of the material. It is necessary to know the regime of deformation rate and temperature space that is developed in the roll bite to make proper application of evolving material models. In this work, a parametric study of hot rolling is conducted. The effects of friction model, friction coefficient, roll gap geometry and temperature on the deformation rate field are demonstrated. The degree and nature of redundant work (shearing) is contrasted for different streamline locations within the bite. Recommendations for the application of material models in analysis of rolling are made with consideration of the simulation predictions.

9:30 AM INVITED PAPER

FORMATION OF SURFACE COARSE GRAINS IN ALUMINUM ALLOYS DURING EXTRUSION: *P. T. Wang*¹; R. D. Dogerty²; ¹Alcoa Technical Center, Aluminum Co. of America, Alcoa Center, PA 15069 USA; ²Drexel University, Department of Material Engineering, Philadelphia, PA 19104 USA

Surface coarse grain formation in aluminum alloys during extrusion has been investigated using a sub-scale indirect extrusion approach where various thermomechanical histories under extremely large strains and elevated temperatures could be imposed on extrudates. During the course of coarse grain formation in extrusion, three types of recrystallization behavior could be observed: in-situ recrystallization during extrusion, primary and secondary (coarsening) recrystallization during post cooling and solution heat treatment. A recrystallization front criterion is proposed and is used as a constitutive model to describe the thickness of in-situ surface recrystallized layers formed during extrusion. In conjunction with finite element analyses, a recrystallization front from the surface to the near surface areas and the formation of coarse grains behind the front for direct extrusion can be described. For tracking a given material point or an extruded grain along a material flow line, the proposed recrystallization criterion is sensitive to thermal and deformation histories, which realistically reflects experimental observations. It is suggested that surface temperature and strain rate, and surface strain profiles are key parameters for the control of surface quality. Additional research effort in modeling primary and secondary recrystallization is mentioned. The methodology presented here can be utilized for studying the effect of dispersoid on recrystallization for extrusion products.

10:00 AM INVITED PAPER

MODELLING RECRYSTALLISATION IN MULTI-PASS HOT ROLLING AND EXTRUSION OF COMMERCIAL ALUMINIUM ALLOYS: *Hans Erik Vatne*¹; ¹Aluminium, R&D Materials Technology, Sunndalsora N-6600 Norway

A model for recrystallisation after hot deformation of aluminium alloys has recently been developed [1,2]. The model is based on thorough experimental investigations of recrystallisation behaviour and deformed microstructure and texture of hot plane strain compressed AA1050 and AA3104 alloys, where special attention has been paid to the industrially relevant cube orientation [3,4]. The model differs from other recrystallisation model in the sense that several of the basic equations are physically based rather than purely empirical. In the present paper the model has been compared to multipass hot deformation of AA3104 alloys and extruded AA6060 and AA6082

types of alloys. Model validation for multi-pass hot rolling is not easy, as industrial tests are commercially sensitive and specimen collection is difficult from a practical point of view. Here, the multi-pass aspect has been tested in several ways. Firstly, by 2-pass plane strain compression laboratory tests, where the inter-annealing time between the 2 deformation passes was varied to give different amounts of recrystallisation between the 2 deformation passes. Secondly, by comparing to a fairly controllable laboratory hot rolling mill. Thirdly, the multi-pass aspect was tested by industrial hot rolling trials by a reversible break-down mill in connection with a 2-stand tandem mill. The model has also been applied to extrusion of 6xxx series alloys (by a vertical laboratory press), where recrystallisation kinetics, texture and grain size was modelled. The alloy AA6082 is particularly challenging, as precipitation of Mn-bearing dispersoids before and during annealing strongly interacts with the recrystallisation process.

10:30 AM INVITED PAPER

QUANTIFICATION AND SIMULATION OF TEXTURE GRADIENTS IN ROLLED ALUMINUM PLATE: *Paul Dawson*¹; Matthew P. Miller¹; Todd J. Turner¹; ¹Cornell University, Sibley School of Mechanical and Aerospace Engineering, 196 Rhodes Hall, Ithaca, NY 14853 USA

Rolled, thick gauge plate, such as that used for the fabrication of airframe components, exhibits through-thickness gradients in the crystallographic texture which arise from the heterogeneous nature of processing deformations. Near-surface material is subject to more intensive shearing than material that is closer to the plate centerplane, shearing that reverses its sense as material passes the neutral point. The prediction of the crystallographic texture, including the strength of its gradients, is a goal of process simulation, and having an effective means of representing the texture is critical to being able to quantify its gradients with accuracy. The crystallographic texture for a sufficiently large volume of material (sufficient in the sense that the volume contains on the order of thousands of crystals) can be represented in a number of ways. A discrete collection of orientations is one possibility, while an orientation distribution function is another. Orientation distribution functions specify the probability density (a scalar) as a function of position in orientation space. The representation can be constructed in terms of various basis functions, such as harmonics in which approximating functions each span the entire space or finite elements in which the basis functions are piecewise. Further, the choice of spaces are many, including conventional Euler angle representations as well as angle-axis representations. Here we utilize a combination of an angle-axis representation (Rodrigues vectors) and finite element interpolation, noting that the parameters of this representation are nodal values which coincide with the orientation distribution value at the nodal point position and which can be interpolated easily between neighboring points. In this paper we discuss the robustness of this methodology for quantifying texture gradients in relation to measurements made on rolled aluminum plate using SEM electron backscatter patterns. The texture obtained by interpolating between two known distributions is compared to the measured texture. Further, we discuss the distributions computed using a finite element, polycrystal plasticity simulation for aluminum rolling in terms of the ability to capture the texture gradient, especially in terms of important texture components.

11:00 AM INVITED PAPER

MODELLING TEXTURE EVOLUTION DURING BREAK-DOWN ROLLING OF AA3104: *M. R. Van der Winden*¹; H. E. Vatne²; C. M. Sellars³; ¹Koninklijke Hoogovens NV, P.O. Box 10.000, 1970 CA IJmuiden The Netherlands; ²Hydro Aluminium a.s., P.O. Box 219, Sunndalsora N-6601 Norway; ³The University of Sheffield, Engineering Materials, Mappin Street, Sheffield S1 3JD United Kingdom

Recently a new experimental technique has been developed to simulate industrial break-down rolling. The Sheffield Mill for Aluminium Rolling at Temperature provides a possibility to study the microstructural development of a specimen at each stage between as-cast ingot to transfer gauge. In the present work, this technique has been used to determine the texture evolution during break-down roll-

ing. In parallel to the experimental technique, a model was developed that can be applied to break-down rolling. One of the parameters calculated by the model is texture. So far, this aspect of the model has not been validated. It is the objective of the present work to do this. Also, the model has been refined. The main alteration enables the changes in the second phase particle distribution to be taken into account.

11:30 AM INVITED PAPER

EXPERIMENTAL TECHNIQUES AND APPLICATIONS OF SUB SCALE PHYSICAL SIMULATIONS: *Vivek M. Sample*¹; ¹Alcoa Tech Center, 100 Tech Dr., Alcoa Center, PA 15069 USA

Physical simulations of thermo mechanical histories can be successfully conducted to gain a fundamental understanding of microstructure evolution as well as to obtain an empirical relationships of process-microstructure and microstructure-properties. Such simulations often provide several distinct advantages over full scale plant experiments such as: economic and expeditious means of obtaining desired data; independent and precise control of process parameters; better data of the actual history applied; ability to understand the microstructure evolution through a sequence of operations; and, ability to go beyond the process envelope of production equipment (e.g. temperatures, strain rate and stress). Techniques and limitations of conducting sub scale thermo mechanical simulations will be discussed.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Particle Reinforced Composites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: Bhaskar S. Majumdar, UES, Inc., Dayton, OH 45432-1894 USA; S. Krishnamurthy, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Wednesday AM Room: Paris A
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: M. L. Gambone, Air Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH 45433 USA

8:30 AM

FRACTURE BEHAVIOR OF SiC PARTICULATE-REINFORCED ALUMINUM-ALLOY COMPOSITES: *A. B. Pandey*¹; B. S. Majumdar¹; D. B. Miracle²; ¹UES, Inc., 4401 Dayton-Xenia Road, Dayton, OH 45432 USA; ²Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

Discontinuously reinforced aluminum (DRA) composites have poorer ductility and fracture toughness than the base matrix alloy. The effects of microstructural parameters, such as particle size and volume fraction, are not well established as yet, and are the subject of this controlled study. Two matrix alloys were selected: 2009 and 7091 alloys. Additionally, two different particle sizes, 4.9 μm and 10.4 μm , and volume fractions, 15 and 25 vol.%, of SiC, were considered. These composites were processed using a powder metallurgy technique, and tested in the nominally underaged and peak-aged conditions. The composite containing finer SiC particles showed slightly higher yield strength and ductility than that with the coarse particles. The volume fraction of SiC had considerable influence on the ductility of the composite. The fracture toughness exhibited some depen-

dence on the volume fraction, but was negligibly influenced by the particle size. Fractography and damage observations will be presented to explain some of the results.

8:55 AM INVITED PAPER

STUDIES OF DAMAGE IN A MODEL ZrO_2 PARTICLE REINFORCED Al-6061 MATRIX COMPOSITE USING DUAL ENERGY X-RAY TOMOGRAPHY: Mr. Justice¹; B. Derby¹; P. Anderson²; G. Davis²; J. Elliott²; ¹University of Oxford, Department of Materials, Parks Road, Oxford, Oxi 3PH UK; ²Queen Mary and Westfield College, Department of Biophysics, School of Dentistry, Mile End Road, London, England E1 4NS UK

It is well known that particle fracture or particle/matrix decohesion are important damage mechanisms which occur in particulate reinforced metal matrix composites soon after the onset of tensile plastic flow. There has been considerable discussion in the literature concerning the role of reinforcement particle clustering in controlling the onset of damage nucleation in these materials. In this study we use a dual energy X-ray tomography technique which allows the separation of reinforcement and void spatial distribution from the same reconstructed slice. Unfortunately, to achieve this either the matrix or reinforcement must contain a component with a strong X-ray absorption edge. Here we study a model ZrO_2 particulate reinforced Al-6061 alloy. In this system our results indicate a correlation between void distribution and regions of higher than normal reinforcement density.

9:20 AM

FAILURE OF MMCS UNDER CYLIC LOADING - MEASUREMENT AND INTERPRETATION OF STRESSES USING NEUTRON DIFFRACTION: M. R. Daymond¹; M. E. Fitzpatrick²; ¹IRIS, Rutherford Appleton Labs, Chilton, Oxon UK; ²Open University, Dept. of Materials, Milton Keynes UK

Neutron diffraction allows non-destructive monitoring of the stress state within the phases of a composite. Multiple phases can be simultaneously examined in situ while macroscopic temperature or stress loads are applied. The technique has been used to characterise the deformation and load sharing characteristics of Metal Matrix Composites undergoing isothermal creep, thermal cycling creep and cyclic fatigue loading. This talk will focus on the latter form of failure in MMCs. The residual stress state of a composite material is known to affect the initiation and growth of fatigue cracks, and thus fatigue resistance. For instance the compressive stress produced in the reinforcement phase of an MMC due to thermal misfit upon cooling from fabrication retards the early stages of fatigue crack growth. We have explored the effect of fatigue cycling on the residual stress state of an Al/20%SiC particulate reinforced MMC, using pulsed neutron diffraction. A novel stroboscopic data collection system has been used to allow data collection in situ during fatigue cycles at various stress levels. Comparisons are made with finite element model predictions.

9:45 AM INVITED PAPER

EFFECT OF REINFORCEMENT CLUSTERING ON THE STRENGTH AND DUCTILITY OF PARTICLE-REINFORCED COMPOSITES: J. Llorca¹; M. Elices¹; C. Gonzalez¹; ¹Polytechnic University of Madrid, Department of Materials Science, E.T.S. de Ingenieros de Caminos, Madrid 28040 Spain

A micromechanical model is developed to simulate the mechanical response of particle-reinforced metal-matrix composites including the effect of reinforcement clustering. The microstructure of the composite is represented as a three-dimensional array of hexagonal prisms with one reinforcement at the center of each prism. The shape, volume fraction and state (either intact or broken) of the reinforcement is independent for each cell, so it is possible to study the interaction among regions with large and small reinforcement volume fractions. The tensile response of the composite is determined from the behavior of the intact and damaged cells, the fraction of damaged cells being calculated on the assumption that the

reinforcement strength follows the Weibull statistics. The model is used to determine the effect of the heterogeneous distribution of the reinforcements with different shape within the composite on the tensile strength and ductility.

10:10 AM

EFFECTS OF STRESS TRIAXIALITY ON THE DAMAGE EVOLUTION OF DISCONTINUOUSLY REINFORCED COMPOSITES: Peravudh Lowhaphandu¹; John J. Lewandowski¹; ¹Case Western Reserve University, Dept. Materials Science and Engineering, 10900 Euclid Ave., Cleveland, OH 44106 USA

The effects of changes in stress triaxiality on the damage evolution were determined for AZ91 Mg matrix composites in addition to 7093Al matrix composites containing several types of reinforcement. The stress triaxiality variation was obtained via utilizing notched axisymmetrical tensile specimen and superimposed hydrostatic pressure. SEM fractography and optical metallography were used to quantify the damage levels. The damage levels (i.e. amount of particle cracking) observed were correlated with the level of imposed stress triaxiality and particle geometry.

10:35 AM BREAK

10:50 AM INVITED PAPER

A MICROSTRUCTURE BASED COMPUTATIONAL MODEL FOR DAMAGE EVOLUTION: S. Moorthy¹; S. Ghosh¹; B. S. Majumdar²; ¹The Ohio State University, Dept. of Aerospace Engineering, Applied Mechanics and Aviation, Columbus, OH 43210 USA; ²UES Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

A Voronoi Cell finite element model (VCFEM) is developed for damage initiation and evolution in composite microstructures. An elastic constitutive relation is used for the inclusion, for which particle cracking is modeled by the Mohr's theory of maximum principal stress or the Rankine criterion. To represent ductile failure, the matrix material is represented by a pressure dependent elastic-plastic constitutive relation for porous materials. In particular a non-local form of the Tvergaard-Gurson model with void nucleation, growth and coalescence is used for modeling the ductile response of the matrix. Evolution of damage in the microstructure is monitored through growth in porosity of the microstructure. In the experimental component of this work, metal matrix composites with different particle volume fractions and particle sizes subjected to different strain levels by uniaxial tension, were studied. Microstructural images formed input for the VCFEM. Damage evolution from VCFEM are compared with the experimental observations. In particular, damage in the microstructure near a structural flaw is compared with experimental observations by modeling a distribution of particles ahead of the flaw in a compact tension test. Damage in the form of brittle particle cracking and ductile matrix softening are compared with experimental observations obtained by image analysis.

11:15 AM INVITED PAPER

INFILTRATED BORON CARBIDE AND ALUMINA PARTICLE REINFORCED ALUMINUM COMPOSITES: M. Kouzeli¹; T. Grant¹; L. Weber¹; B. Viguier¹; C. San Marchi¹; A. Mortensen¹; ¹Swiss Federal Institute of Technology in Lausanne, Department of Materials, Laboratory for Mechanical Metallurgy, Lausanne, EPFL CH-1015 Switzerland

The fracture and deformation characteristics of particle reinforced metal-matrix composites are often complicated by the large number of microstructural variables typically present in this class of material, such as particle distribution, porosity and secondary phases. The present study is thus partly motivated by the comparatively greater microstructural simplicity of pressure-infiltrated composites, which are pore-free, contain a uniform distribution of particles, and can feature microstructurally simple matrix and reinforcement structures. Focus is placed on composites which are based on aluminium reinforced with approximately 50% of boron carbide or aluminum oxide particles, to

highlight the importance of particle characteristics. The incidence of particle size and other microstructural variables on the mechanical behavior of these materials is investigated with a dual focus on tensile behavior and fracture toughness

11:40 AM INVITED PAPER

THE CONSTRAINT EFFECT ANALYSIS OF SiC PARTICLE REINFORCED ALUMINUM MATRIX COMPOSITES: *M. Kikuchi*¹; *M. Geni*¹; ¹University of Tokyo, Dept. of Mechanical Engineering, Noda, Chiba 278 Japan

The dimple fracture test is conducted by changing the stress triaxiality using notched specimen of SiC particle reinforced aluminum alloy. The fracture surface is observed by SEM, and it is found that the stress triaxiality has large effect on the dimple size and the critical void volume fraction. Using the three dimensional FEM, based on the large deformation theory and Gurson's constitutive equation, the interaction effect of SiC particles during dimple fracture is evaluated. The distribution of the stress triaxiality is also obtained and the effect on the dimple fracture process is discussed.

12:05 PM INVITED PAPER

OPTIMIZING THE FRACTURE RESISTANCE OF PARTICULATE COMPOSITES BY MICROSTRUCTURAL MODIFICATION: *David L. Davidson*¹; ¹Southwest Research Institute, 6220 Culebra Road, San Antonio, TX 78238 USA

Somerday and Gangloff found that the fracture of notched specimens of particulate containing composites is insensitive to notch shape. This result indicates that the effect of macroscopic notch geometry related constraint is not applicable to particulate composites. Measurement of constraint on a microstructural scale (Davidson and McClung) confirms that particles cause microscale constraint. Particle induced constraint, together with other fracture related parameters, has been combined into a model for computing fracture toughness based on particle volume fraction, size, fracture toughness, and matrix strength and fracture strain. The model is calibrated with results from some Nb-based composites. Using the model allows estimates to be made of the effects on fracture toughness of changing particle and matrix characteristics. Thus, the microstructural characteristics that optimize the fracture resistance are predicted, which can provide guidance for further development of tough particulate composites.

MODELLING OF TEXTURE AND ANISOTROPIC PROPERTIES IN STEEL AND ALUMINUM INDUSTRIES FOR OPTIMUM MATERIALS PERFORMANCE: Session 1

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee

Program Organizers: Hasso Weiland, Alcoa, Alcoa, PA 15069 USA; J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Wednesday AM Room: Madrid
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Hasso Weiland, Alcoa Technical Center, Alcoa Center, PA 15069 USA

8:30 AM INVITED PAPER

SURFACE ROUGHENING AND MICROSTRUCTURE IN SHEET METALS: *A. D. Rollett*¹; *P. S. Lee*¹; *G. Jarvis*²; *H. R. Piehler*²; *B. L. Adams*²; *S. Saigal*³; *C. Shet*³; ¹Dept. of Mater. Sci. & Eng., Carnegie

Mellon University, Pittsburgh, PA 15213-3890; ²Dept. of Civil and Environmental Eng., Carnegie Mellon University; ³Alcoa Technical Center, 100 Technical Drive, Alcoa Center, PA 15069-0001

Considerable effort has been expended over the years to develop processing methods and sheet forming techniques that preserve a high quality surface finish. The crystallographic nature of plastic deformation in sheet metals means, however, that a free surface will tend to deform non-uniformly. Our studies of surface roughening have shown that, in addition to roping and L-ders bands, distributed linear localization can occur. This form of localization which occurs in a range of straining modes may be linked to shear banding in the sheet materials. Evidence will be presented from Orientation Imaging Microscopy (OIM) on 6111-T4 aluminum sheet that this form of surface roughening is associated with strain inhomogeneities through the thickness of the sheet. The microtexture in the sheet was also observed to be inhomogeneous and the relationship between microtexture and shear banding will be explored. Techniques used in this work include OIM, interference microscopy, digital image analysis, polycrystal plasticity simulations and finite element simulations with constitutive descriptions of materials that incorporate crystal plasticity.

9:00 AM

TEXTURE EVOLUTION IN ALUMINUM ALLOY SHEETS DURING DEEP DRAWING : *Shi-Hoon Choi*¹; *Jae-Hyung Cho*¹; *Kwansoo Chung*²; *Frederic Barlat*³; *Kyu Hwan Oh*¹; ¹Seoul National University, School of Materials Science and Engineering, Research Institute of Advanced Materials, San 56-1 Shinrim-dong, Kwanak-ku, Seoul, Korea; ²Seoul National University, Department of Fiber and Polymer Science, San 56-1 Shinrim-dong, Kwanak-ku, Seoul, 151-742 Korea; ³Aluminum Company of America, Alcoa Technical Center, 100 Technical Drive, Alcoa Center, Pittsburgh, PA USA

The texture evolution by deep drawing of AA1050 sheet metals was investigated and the stable orientation was predicted using rate sensitive model. The measured textures show the different behavior with the initial crystal orientations and strain state. Among the crystal orientations of RD sample, the crystal orientations around the D component rotated toward the Cu component and the crystal orientations along the α fibre rotated toward the $\{1\ 1\ 0\}\langle 0\ 0\ 1\rangle$ and $\{1\ 1\ 0\}\langle 1\ 1\ 1\rangle$ components during deep drawing. In the case of the TD sample, the crystal orientations around the D component rotated toward the Rotated Cube and the crystal orientations along α fibre rotated toward the $\{1\ 1\ 0\}\langle 1\ 1\ 3\rangle$ component. In order to calculate the strain path during deep drawing, deep drawing for aluminum alloy sheet was simulated using anisotropic elasto-plastic finite element analysis. In this study, the orientation density in the wall part which undergoes tensile deformation was interpreted by considering sequential deformation states such as plane strain and tensile strain state.

9:30 AM INVITED PAPER

ASSESSMENT OF VARIOUS YIELD CRITERIA FOR PREDICTION OF FORMING LIMIT STRAINS IN AN ALUMINUM SHEET: *J. Savoie*¹; ¹Alcan, Kingston Canada

In order to simulate adequately the formability of sheet materials, it is important to use a yield criterion which accurately captures the anisotropy of the material due to crystallographic texture. These yield functions must be in a form that can be used easily in finite element analysis. This work investigates the use of two commonly used yield criteria by Hill [1990] and Barlat [1989], as well as a 4th order inverse potential function [1996] for forming limit predictions. User-material subroutines, based on these functions, have been written and implemented into a commercial finite element code - Abaqus/Implicit. Finite element simulations of hemispherical punch stretching process are carried out for an automotive aluminium sheet. Different values of coefficient of friction between the punch and sheet surfaces are employed in the models to simulate strain paths in the range between plane strain and equi-biaxial tension. The model results are compared with a range of experimental parameters such as punch load versus displacement, strain profiles along the longitudinal and transverse orientations of the sheet (with respect to the rolling direction), and limit strains, to assess the suitability of various yield

criteria. It is observed that not only necking but also fracture mechanisms are controlling the forming limits close to equi-biaxial tension. Thus, a fracture criterion was incorporated into the models to improve the predictions of limit strains.

10:00 AM

THE ROLE OF INHOMOGENEITY IN PLASTIC FLOW ON PLASTIC ANISOTROPY IN AA5XXX ALLOYS: *G. Burger*¹; ¹Alcan International Limited, Kingston Research and Development Center, Kingston, Ontario K7L 5L9 Canada

The yield strength, R value and work hardening behaviour of AA5754 and AA5182 sheet material in the O temper were evaluated along orientations at 0, 45 and 90 degrees to the rolling direction. AA5182 sheet in a hard-rolled temper was also considered. Plastic straining at room temperature occurs inhomogeneously, exhibiting serrated flow. To assess the role of serrated flow, testing was also carried out at -70 C, at which plastic flow occurs homogeneously. In O-temper sheet, the yield point at -70 C was almost identical to that at room temperature, although the Lüders elongation was smaller. Lüdering phenomenon was not observed in hard-rolled material. Low temperature did promote an increase in work hardening rate and uniform elongation, however, the increases in d/d_0 in different orientations were equivalent. The elimination of serrated flow had no significant influence on measured R values in any orientation. The development of crystallographic texture during tensile straining at these two temperatures was also identical. Thus it was concluded that the inhomogeneous plastic straining occurring during serrated flow did not influence the macroscopic plastic behaviour of the sheet. The anisotropy in R value and in flow stress in these materials was not well predicted based on a full constraints Taylor-Bishop-Hill calculation utilizing the measured crystallographic textures of the sheet in O temper.

10:30 AM

DO SOLUTE SHEAR BANDS PROMOTE THE BRASS DEFORMATION TEXTURE IN HEAVILY COLD ROLLED 2024 AND 7475?: *Sabir Doherty*¹; Carl T. Necker²; Roger D. Dougherty¹; Surya Kalidindi¹; Ehab El-Danaf¹; ¹Drexel University, Dept. of Materials Engineering, Philadelphia, PA 19104 USA; ²Los Alamos National Laboratory, Los Alamos, NM USA

The texture transition from a copper-type rolling texture to a brass type one associated with low stacking fault energy driven deformation twinning is believed by some investigators to arise not directly with the onset of twinning but with the twinning and high strain hardening induced shear banding. In order to test this idea the deformation textures in heavily rolled aluminum alloys containing high solute contents (solution treated quenched 2024 and 7475 known to form many shear bands) are being studied to determine if shear banding in these high stacking fault energy alloys, which do not twin, give a conventional copper type Texture or one more similar to the Brass texture.

11:00 AM

PLASTIC ANISOTROPY IN PRECIPITATION-STRENGTHENED ALUMINUM ALLOYS DEFORMED IN UNIAZIAL AND PLANE STRAIN COMPRESSION: *Barlat and J. Liu*¹; ¹Alcoa Technical Center, 100 Technical Drive, Alcoa Center, PA 15069-0001 USA

The goal of this work was to quantitatively assess the plastic anisotropy of strongly textured polycrystals with and without a distribution of hard (non-shearable) precipitates. The aluminum-copper system was selected as a model material. The strong texture in the experimental design provided a high level of sensitivity for evaluating plastic anisotropy. The choice of polycrystals rather than single crystals was to favor multiple slip, which is the behavior more representative of commercial materials. The anisotropic behavior was characterized by the shape change of the specimen cross-sections and the shape of the stress strain curves after uniaxial and plane strain compression tests. A model based on kinematic hardening concepts

was developed to quantitatively describe the plastic behavior of materials containing non-shearable precipitates such as q' in Al-Cu alloys. This model was validated by the experimental results.

11:30 AM

THE EFFECT OF SPECIAL PROCESSING TECHNIQUE ON THE ANISOTROPY OF Al-Li ALLOY: *L. Brandao*¹; P. N. Kalu¹; H. Garmestani¹; O. Es-Said²; Eui Lee³; ¹FAMU-FSU College of Engineering, Department of Mechanical Engineering and Center for Materials Research and Technology, (MARTECH), Tallahassee, FL 32310-6046 USA; ²Loyola Marymount University, Department of Mechanical Engineering, Los Angeles, CA 90045 USA; ³Naval Air Warfare Center, A/C Division, Code 4.3.4.2, Warminster, PA 18974 USA

The use of aluminum-lithium alloys in aerospace applications has been limited due to their undesirable characteristics, e.g. anisotropy. A distinct thermomechanical processing, which combines rolling and stretching (at five different angles) was used to process Al-2095 alloy with the view to eliminate anisotropy. The material was tested in tension and characterized. This paper presents a preliminary attempt to correlate the mechanical anisotropy and texture development with some texture theories. Analyses based on upper bound model using restricted and pencil glide approaches suggested that the contribution of crystallographic texture to anisotropy was only part of the total mechanical anisotropy.

12:00 PM

UNIAXIAL AND BIAxIAL ANISOTROPY IN ALUMINUM ALLOYS: *C. T. Necker*¹; C. N. Tomel¹; D. A. Korzedowa¹; ¹Los Alamos National Laboratory, Los Alamos, NM USA

The mechanical anisotropy of sheet material is often measured using the uniaxial tensile test and is represented as Lankford coefficients (R values), the ratio of width strain to thickness strain. There is a correlation between R values and formability in steel however the same can not be said for aluminum. R values less than unity are typical for aluminum. However when the formability of sheet material is tested under biaxial tensile (bulge) conditions the measured through-thickness strength is often found to be greater than the in-plane strength, counter-intuitive to the R value measurement. These apparently contradictory strain proportionalities are investigated computationally using a visco-plastic self-consistent approach to model both uniaxial and biaxial conditions. The approach uses the initial texture combined with imposed stress state conditions to model the evolution of the yield surface and calculate R values. The calculated R values generally relate reasonably well with the measured values. The modeling effort is extended into the plastic regime by combining the texture and imposed stress state conditions with experimentally determined material hardening information. Modeling of both uniaxial and biaxial conditions out to the ultimate strength of the sheet shows that the differences between experimental conditions are captured by this model.

12:30 PM INVITED PAPER

TEXTURE AND EARING AL CAN-SHEET: *J. Hirsch*¹; ¹VAW Aluminium AG, R&D, Bonn Germany

The earing profile obtained in deep drawing of cups must be kept low in order to maintain optimum output and performance during can manufacturing operations. Since sheets of the Aluminium (AA3004) alloy are used in a highly cold rolled state form strong 45° ears this effect must be balanced by the 0°/90° earing characteristic of the initial hot band cube texture. During cold rolling the earing profile changes from 0°/90° to 45° earing with characteristic intermediate profiles (with 6-ears). This transition is analyzed systematically for various initial hot band cube textures and for different rolling reductions. A new method for the evaluation of earing profiles in Al-sheet is presented and applied which gives a quantitative description of the profiles and their transition and the correlation between textures and earing profiles. The variation of initial textures and the minimum rolling strain to achieve sufficient strength can be predicted and the

rolling schedule can be optimized in order to obtain the best combination of low earing and required strength properties.

PAUL A. BECK MEMORIAL SYMPOSIUM: Paul A. Beck Memorial Symposium - III Magnetism

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Program Organizers: Karl A. Gschneidner, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Michael V. Nevitt, Clemson University, Dept of Physics & Astronomy, Clemson, SC 29634 USA; Robert D. Shull, NIST, Bldg. 223 Rm. B152, Gaithersburg, MD 20899

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Session Chair: Charles A. Wert, University of Illinois, Materials Science & Engin., Urbana, Illinois 61801 USA

8:30 AM INVITED PAPER

IRON IN GOLD, PAUL A. BECK: *Anthony Arrott*¹; ¹Virginia State University, Physics Department, P.O. Box 9325, Petersburg, VA 23806 USA

An alloy of iron in gold is inhomogeneous at various length scales depending on the metallurgical treatment. The magnetic moment and anisotropy of each iron atom depend on the arrangement of neighboring iron atoms. The exchange interactions vary with the distances between each pair of iron atoms and the distribution of neighboring iron atoms. These interactions are both ferromagnetic and antiferromagnetic in sign. The exchange interactions are tensors with off diagonal elements that contribute to non-collinearity of magnetic moments. Spin-spin correlation effects are entwined with atom-atom correlation effects. Armed with a phenomenological understanding of metallurgy, thermodynamics and magnetism, Paul A. Beck sought to understand the results of specific heat and magnetic moment measurements and their dependence on the metallurgy. He was able to show that a simple primary assumption of all previous and most continuing theoretical discussion was in doubt. The so called 'spin glass' state seen at low temperatures for concentrations less and greater than the critical concentration for the onset of ferromagnetism is often called the 'reentrant spin glass' phase at the higher concentrations. This is because it was assumed that the higher temperature phase is usual ferromagnetism. Beck's analysis showed that, on a diagram of temperature and concentration, the higher temperature phase was continuous with both the reentrant spin glass phase at low temperatures and the usual ferromagnetic phase at higher concentrations. He called this 'mictomagnetism'. In this state there are clusters of magnetic moments acting together as giant magnetic molecules, but there is an absence of long range order among these clusters. In Au(Fe) the occurrence of mictomagnetism disturbs the clarity of simple models in which the complexities of mictomagnetism are overlooked. The problem sat there and smiled during the years in which Beck worked on it, now that he is gone, the problem still sits there but sadly neglected. Today there are materials that are clearly mictomagnetic as envisioned by Beck, but what about Au(Fe)?

9:05 AM INVITED PAPER

LOCAL ATOMIC ARRANGEMENT AND MAGNETIC PROPERTIES IN Au-Fe SPIN GLASS SYSTEM: *Haydn H. Chen*¹; ¹University of Illinois, Department of Materials Science & Engineering, 1304 West Green Street, Urbana, IL 61801 USA

The Au-rich Au-Fe face-centered cubic alloy system has long been a topic of considerable study due to the spin-glass magnetic behavior displayed for compositions >0.5 at. pct Fe at temperatures below ~ 100 K. The interpretation of the magnetic behavior is not without controversy. For instance, magnetic susceptibility and neutron scattering measurements have given evidence of a "double" magnetic transition corresponding to an interim ferromagnetic state existing between the low-temperature spin-glass state and the high-temperature paramagnetic state. However, Raker and Beck showed through low applied fields and special specimen geometry that no ferromagnetic state exists in water-quenched Au-17.5 at. pct Fe. This paper will review those most recent magnetic results as well as present a detailed local atomic arrangement study based upon diffuse x-ray scattering measurements utilized synchrotron radiation and computer simulation of the short-range ordered structure. Results show for the first time that two different short-range order environments exist. The Fe-enriched environment, although quite disordered, showed a Fe-segregation preference toward {100} and {110} matrix planes. These become more predominant with aging. The Fe-depleted environment is best described using the SCWP (Standing Concentration Wave Packet) model, and the results derived from this work give a real-space visualization of the SCWP. (* Work supported by the US National Science Foundation.)

9:40 AM INVITED PAPER

MAGNETIC PHASE TRANSITIONS PROBED AT VERY LOW MAGNETIC FIELDS: *Helmut Claus*¹; ¹University of Illinois at Chicago, Dept. of Physics, 845 W. Taylor St., Chicago, IL 60607-7059 USA

With the availability of high sensitive SQUID magnetometers it has become possible to probe magnetic phase transitions down to very low magnetic fields. This has led to the discovery of new interesting effects. One is the paramagnetic Meissner effect in superconductors another the diamagnetic response of ferromagnets. As examples we will present data for two paramagnetic superconductors, metallic Nb and the high temperature superconductor YBCO, as well as for the diamagnetic ferromagnet Pd-0.5%Fe. We will demonstrate that in these cases the unexpected sign of the magnetic response is caused by the interaction of a surface layers of slightly enhanced transition temperatures, with the bulk of the samples. Whereas the diamagnetic response of the ferromagnet is well understood, there is still no explanation for the paramagnetic signal for the two superconductors. Part of this research was supported by the US Department of Energy, Basic Energy Science, Materials Science, under Contract No. W-31-109-ENG-38

10:15 AM BREAK

10:35 AM INVITED PAPER

MAGNETICALLY GRANULAR, BUT STRUCTURALLY GLASSY Nd-Fe-Al BASED EXCHANGE COUPLED HARD MAGNETS: *K. V. Rao*¹; *R. J. Ortega*¹; *A. Inoue*²; ¹Royal Inst. of Technology, Condensed Matter Physics, Stockholm SE-100 44 Sweden; ²Inst. of Metals Research, Tohoku University, Sendai, 980 Japan

An unusual maximum of $H_c = 3.5$ Tesla around 30K in the temperature dependence of coercivity is observed in a new class of bulky glassy Fe-Nd-Al based hard magnets. These glassy materials in the form of 3-7 mm rods, fabricated perhaps by the slowest known cooling rates, show isotropic hard magnetic behaviour with room temperature coercivities as high as one Tesla in 30 Tesla fields, but still cannot be magnetically saturated. The origin of the hard magnetic properties appears to be due to exchange hardening provided by the coupling between Fe clusters and an antiferromagnetic matrix. Our studies suggest a new class of Nd-Fe-Al based bulk glassy materials which are magnetically granular although structurally glassy.

11:10 AM

MAGNETIC EXCHANGE BIAS IN A NiO/FeNi ANTIFERRO-MAGNETIC/FERROMAGNETIC BILAYER: Robert D. Shull¹; A. J. Shapero¹; V. Nikitenko²; V. Gornakov²; A. Khapikov²; L. M. Dedukh²; A. Chaiken³; ¹National Institute of Standards and Technology, Metallurgy Division, Bldg. 223, Rm. B152, Gaithersburg, MD 20899 USA; ²Institute of Solid State Physics, RAS, 14232, Chernogolovka Russia; ³Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

The magnetic domain structure in an antiferromagnet (AF) - ferromagnet (FM) bilayer crystal possessing unidirectional magnetic anisotropy was imaged using a special magneto-optic imaging technique (MOIF) developed jointly by NIST and the Institute of Solid State Physics in Chernogolovka, Russia. In this MOIF technique, a thin film of a material with a large magneto-optic Faraday effect is placed on top of the sample of interest. Leakage fields from the sample (primarily near domain walls) locally polarize the imaging film and reveal the domain structure of the sample. By applying a magnetic field to the sample, domain dynamics are then observable. For a NiO/permalloy bilayer crystal with unidirectional magnetic anisotropy, magnetization reversal upon application of a reversed magnetic field was found to occur by domain nucleation and growth. Different domain nucleation sites were observed in such an AF/FM sandwich for the remagnetization processes in opposite directions while cycling the field, as in the measurement of a magnetic hysteresis loop. These findings indicate that the nucleation of domains in such a material is intimately connected with the magnetic bias imposed on the material, as well as on the sample's microstructure. This finding explains the high coercivity and reduced exchange bias always observed. This has important ramifications for magnetic recording devices.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VII: Exotic Processing Techniques for Materials

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Wednesday AM
October 14, 1998

Room: Chicago
Location: O'Hare Hilton Hotel

Session Chairs: Dr. Patrick Taylor, University of Idaho, Plasma Processing Laboratory, Moscow, ID 83844-3024 USA; Dr. L. V. Ramanathan, Cidade Universitaria, Energy and Nuclear Research Institute, Travessa R 400, CEP 05508-900

8:30 AM INVITED PAPER

PLASMA SPRAYING OF HYDROXYAPATITE COMPOSITE COATINGS: K. A. Khor¹; C. H. Quek¹; V. J. P. Lim¹; P. Cheang¹; ¹Nanyang Technological University, Singapore, School of Mechanical & Production Engineering, Singapore 63978

Biomedical implants using metals, polymers, ceramics and composites are increasingly being designed to restore bio-function and to facilitate healing of the ailing body part. It is often the material design aspects of such implants that will determine the stability of the biomedical implant in-vivo. This paper investigates the design of hydroxyapatite (HA) based composite layers using dc plasma spraying. Composites based on the combination of HA with Ti-6Al-4V and HA with YSZ are investigated. HA being a bioactive material

combined with non-toxicity and bioinert. Ti-6Al-4V provides an implant that is biocompatible, light and stable. YSZ is known for imbuing improved fracture toughness to many ceramic matrices. In the HA/Ti-6Al-4V composites, the powder feedstock using calcined HA and Ti-6Al-4V was prepared by the ceramic slurry mixing method in the ratio of 80 wt%: 20 wt % and 50 wt%: 50wt% respectively. Yttria stabilized zirconia (YSZ) was ball milled together with HA in the compositions 10% HA, 30% HA and 50% HA, and then plasma sprayed together with the HA onto a Ti-6Al-4V substrate under various spraying parameters. The composite powder was deposited onto the titanium substrate by means of a robot-controlled 100kW plasma gun (Praxair Surface Technologies, USA) equipped with an advanced computerized closed-loop powder feed system. Mechanical properties of the individual layers were tested using the tensile adhesion test. The surface morphology and microstructure of the composite layers were examined by a scanning electron microscope. The phase composition and crystallinity were determined by the X-ray diffractometer. The investigation shows that the as-sprayed coating of the individual layers possess a higher porosity level in the HA-rich regions. Tensile adhesion test revealed a higher bond strength for the composite coatings compared to pure HA coatings.

8:55 AM INVITED PAPER

PRODUCTION OF HOLLOW FIBERS FROM AN IMIDE COPOLYMER: Gaajanan S. Bhat¹; ¹University of Tennessee, 230 Jessie Harris Building, Knoxville, TN 37996-1900 USA

Hollow fibers spun from synthetic polymers have been investigated for a long time, especially for producing high-bulk, low-density fabrics. Such fibers produced from a high temperature resistant polymer have some advantages in intended applications. Ultem, a polyetherimide resin marketed by General Electric company is a copolymer with ether molecules between imide groups. The fully reacted polyimide with the imide group being part of the linear polymer chain makes this polymer thermoplastic and easily dissolvable. Being an amorphous thermoplastic polyimide, the Ultem resin combines the high performance associated with exotic specialty polymers and the good processability of typical engineering plastics. In addition to high strength and modulus, and heat resistance, the polymer has high dielectric strength, broad chemical resistance, transparency and good processability. A special set-up was built at the Phillips Laboratory of Edwards AFB, CA to spin hollow fibers. The spinneret designed has a hollow tube supported in the center of the orifice and an inert gas is injected through the needle to maintain the tubular shape until the solidification of the fiber. Using that set-up, fibers were spun from two grades of Ultem under several different conditions. Effect of these processing conditions on the structure and properties of the formed hollow fibers will be discussed.

9:20 AM INVITED PAPER

RESPONSE SURFACE STUDY ON PRODUCTION OF EXPLOSIVELY-WELDED ALUMINUM-TITANIUM LAMINATES: Efe S. Ege¹; Osman T. Inal¹; C. A. Zimmerman¹; ¹New Mexico Tech, Materials and Metallurgical Engineering, Socorro, NM 87801 USA

Aluminum-titanium multi-layered composites (laminates) were produced through explosive welding, for light weight, structural applications. The purpose of lamination was to have a material exhibiting superior mechanical properties due to the, explosive-welding introduced, plastic deformation throughout each layer and to the lamination-introduced strengthening. A total of eighteen laminates, nine of which were used as points defined by central composite design, were produced. Yield strength, ultimate tensile strength, elongation, and toughness data were collected from tensile tests. A second-order model was fitted and a three dimensional response surface was built to see the relationship between the mechanical properties of the laminates, namely yield strength and toughness and two design variables, abundance of interfaces (or total interfacial area) and volume percentage of the more ductile component. The fitted second-order model clearly shows that the mechanical properties of the laminates strongly depend on the relative amounts of the components but not on the abundance of interfaces within the selected operability region.

Further characterization of the laminates were done by microhardness testing, optical microscopy (OM) and scanning electron microscopy (SEM). Microhardness values revealed extensive hardening, over 25%, especially in titanium layers. Optical microscopy revealed plastic flow in the layers. SEM studies showed that laminates failed in a ductile manner. The presentation will detail our efforts.

10:00 AM INVITED PAPER
TAPE CASTING OF COMPOSITE CERAMIC SUBSTRATES USING HOLLOW MICRO-SPHERICAL POWDERS: Alfred I.Y. Tok¹; *Freddy Y. C. Boey*¹; K. A. Khor¹; ¹Nanyang Technological University, School of Mechanical & Production, Nanyang Avenue, Singapore 639798

The current device of miniaturization and higher device counts in IC (integrated circuit) packages has significantly increased the use of both multilayer ceramic packages (MLCP) and multilayer capacitors (MLC). Both employ thin ceramic tapes with very high dielectric properties which are cast using solid powders. Crucial to the production of these ceramic substrates is the use of a tape caster with precise control of tape thickness, uniformity and surface finishes. A novel approach is presently taken in producing a composite ceramic substrate using hollow micro-spherical powders via the tape casting method. The advantage being significantly lower density and better dielectric behaviour. This involved the proper control of both the formulation as well as the processing parameters, both of which were critical to the quality and reproducibility of the substrates.

10:25 AM INVITED PAPER
THE SOLVENT LAMINATION FOR POWDER INJECTION MOLDING FEEDSTOCKS: *Ting-Chu Ko*¹; James D. Cawley¹; Arthur H. Heuer¹; ¹Case Western Reserve University, Department of Materials Science and Engineering, 10900 Euclid Avenue, Cleveland, OH 44106-7204 USA

Rohm & Haas stainless steel 316L powder injection feedstocks have been evaluated as a raw material for solvent lamination. This was performed by compression molding the feedstocks into sheet that was cut into the desired dimension for lamination aided by solvent, and then debinded and sintered. Various solvent-based techniques were evaluated for sheet lamination, and the optimum set of lamination parameter results was determined. Employing to this optimized lamination formulation, complex objects were generated. In some, a fugitive material has been used to aid in registration. The effect of solvents on mechanical properties will be also reported. Modeling of the solvent concentration profile in the tape was performed in order to confirm and explain the measured property change after the solvent treatment. The polymers in feedstocks were identified and the kinetics of phase separation for those polymers was determined using capillary rheometer and Dynamic Mechanical Analysis (DMA).

10:50 AM INVITED PAPER
THE DEVELOPMENT OF AMORPHOUS Fe-Cr-P ELECTRODEPOSITS: C. T. Kunioshi¹; O. V. Correal¹; N. B. de Lima¹; *L. V. Ramanathan*¹; ¹Cidade Universitaria, Energy and Nuclear Research Institute, Travessa R., CEP CDP 05508-900

Some metallic amorphous alloys have shown superior corrosion resistance and could thus be used for protecting other less resistant alloys in aggressive environments. A number of techniques have been used to obtain amorphous alloy coatings. One of the techniques, aqueous electrodeposition is simple and economical. This paper presents the effect of electroplating parameters on the structure, morphology, composition and corrosion resistance of Fe-Cr-P deposits. The effect of various complexing agents and other bath additives as well as current density, duration of deposition and the use of ion selective membranes on deposit characteristics have been investigated. Deposits with average grain size as low as 15Å were obtained. Corrosion measurements on uncoated and amorphous alloy coated mild steel specimens in acidic media revealed significant increases in corrosion resistance of the coated specimens. These data along with

the effect of varying the plating parameters during electrodeposition to obtain gradient amorphous deposits will be presented and discussed.

11:15 AM INVITED PAPER
PRODUCTION OF BARIUM FERRITE FILMS USING AN INDUCTIVELY COUPLED PLASMA REACTOR: Edgar E. Vidal¹; *Patrick R. Taylor*¹; ¹University of Idaho, Plasma Processing Laboratory, Department of Metallurgical and Mining Engineering, Moscow, ID 83844-3024 USA

The production of barium ferrite thin films has been evaluated experimentally using Plasma Assisted Chemical Vapor Deposition in an Inductively Coupled Thermal Plasma (ICP) Reactor. Thermal plasma appears to demonstrate technological and economical advantages over existing methods. The films were characterized using Scanning and Transmission Electron Microscopy (SEM and TEM) and X-Ray Diffraction (XRD). Magnetic properties of the films were also determined: saturation magnetization, coercivity, and squareness ratio. A mathematical model that describes the deposition process at the substrate is presented and compared with experimental results. Solutions prepared with the salts of the desired elements are atomized in the plasma region, ensuring complete vaporization of the precursors. Experimental variables studied include plasma torch power, relative substrate-plasma position, solution stoichiometry, and substrate material. It was observed that under certain conditions the resultant films were amorphous, thus requiring annealing.

11:40 AM
THE EFFECT OF PRIOR HEAT TREATMENT AND PROCESSING VARIABLES ON THE HARDENED LAYER OF NITRIDED EN40B STEEL: *S. Mridha*¹; D. H. Jack¹; ¹Nanyang Technological University, School of Applied Science, Nanyang Avenue, Singapore 63978

The growth kinetics of nitrided layer is investigated by nitriding EN40B steel at 470°, 520°, and 570°C for 6h to 96h in gaseous environments containing different amounts of ammonia. The effects of austenitizing and tempering temperatures, and surface preparation prior to nitriding are also discussed. The nitrided layer thickness is found to increase parabolically with increasing processing time. The growth of this hardened layer shows a linear relationship with nitriding potential at all temperatures up to a certain point where white layer forms. At 470°C the growth rate is constant where presumably γ-Fe₄N is present while at 520° this constancy appears in the presence of ε-Fe₃N. At 570° C the growth rate continues to increase; but not linearly, even when γ-Fe₄N is present and constancy appears well ahead of the presence of ε-Fe₃N phase. The diffusivities of nitrogen in alpha-iron at different nitriding temperatures, calculated for this steel using experimentally observed r[X] values and the growth rates in the internal nitriding model, are about 50% lower than those for pure iron. The presence of grain boundary cementite and/or the counterflow of carbon from the carburized layer in the nitrided steel is proposed to oppose the diffusion of nitrogen, and the internal nitriding model; therefore, cannot be used for any quantitative prediction in steels.

12:00 PM
WETTING AND INTERFACIAL REACTIONS BETWEEN SNPB SOLDER AND BULK Ni SUBSTRATE: *Kwan Hoe Han*¹; Soong Joon Jeon¹; ¹Yeungnam University, Department of Metallurgical and Materials Engineering, 214-1 Dae-Dong, Kyongsan, Kyongbuk 712-749 Korea

Ni has been being widely used as a Barrier-coating layer, in microelectronics industries, for making chip mount and lead frame Interconnects through soldering. In spite of the technological importance, however, the soldering reactions between the molten solder and Ni, were not fully explored. In an effort to obtain better understanding of the wetting and interfacial reactions between eutectic SnPb solder and Ni an experimental work has been carried out in this study. We used pure Ni plate and eutectic, SnPb solder beads as a substrate and solder, respectively. The experiments were carried out for various reflow

times in mildly activated resin (type RMA) flux at temperatures between 200 and 250°C. The morphology and microstructural observation and the phase analysis were performed using optical and scanning electron microscope equipped with EDX. The spread radius and the wetting angle Cross-sectional microstructural examination showed that the intermetallic compounds forming at the interface possess a rod-shaped morphology, being different from the same phase formed in the solid state. The morphology and the growth kinetics of the compounds will be presented as well as the wetting/spreading characteristics.

12:20 PM

TITANIUM CARBIDE WHISKERS SYNTHESIZED USING METHANE AS A CARBON SOURCE: T. Hashishin¹; N. Tsukamoto¹; Y. Kaneko¹; ¹Ritsumeikan University, Department of Chemistry, Faculty of Science and Engineering, Shiga Prefecture, Kusatsu-shi, 1-1-1, Noji-Higashi

Titanium carbide (TiC) whiskers were deposited on the outer surface of a graphite tube and on a graphite boat in 10vol% methane gas stream following a heat treatment at 1000-1400°C. This temperature range was broader and lower than the 1250-1400°C range in which TiC whiskers were obtained when 5vol% methane gas was used in a thermal-decomposition-reduction-carbonization reaction. The lattice constant of TiC whiskers deposited on the outer surface of a graphite tube after the heat treatment at 1400°C was 0.432084 ± 0.007 nm, indicating that this TiC had a stoichiometric composition. The cylindrical whiskers formed at 1050-1200°C were considered to have been grown via a vapor-solid (VS) growth mechanism. Because the side faces of the rectangular prismatic whiskers formed at 1300-1400°C were dented, whisker growth in the radial direction may be caused by the layer stacking growth mechanism (a VS mechanism) due to two-dimensional nucleation on the {110} plane.

12:40 PM

POLYSILOXANE MODIFIED EPOXY - OLIGOIMIDE COMPOSITES: A. Ashok Kumar¹; M. Alagar¹; S. Anand Kumar¹; K. P. O. Madesh¹; ¹Anna University, Department of Chemical Engineering, Chennai 600 025 India

A novel hybrid variety of polydimethylsiloxane modified epoxy - oligoimide matrix resin systems have been developed from diglycidyl ethers of bis phenol-A and hydroxyl terminated polydimethylsiloxane with 3-aminopropyltriethoxysilane as a crosslinking agent. The objective of development of modified epoxy resin system is to improve the impact, thermal, dielectric and aging characteristics of conventional epoxy systems and to use them for the fabrication of high performance advanced composites for engineering and aerospace applications. The different types of curatives viz. aromatic bis(maleimide)-diaminodiphenylmethane oligomers having multifunctional amine groups are synthesized through the Michael addition reaction. The amine terminated oligomers are used for the curing of polydimethylsiloxane modified epoxy resin and the results are compared with those of conventional aromatic amine cured epoxy systems. The mechanical, electrical, thermal and aging characteristics of cured siliconized epoxy systems have been carried out as per ASTM standards. The optimum siloxane introduction into epoxy resins to achieve reasonable impact characteristics have been ascertained from experimental data. The siloxane introduction influences and improves the impact, dielectric, thermal and aging characteristics of the cured matrix systems and reinforced composites according to the percentage composition of siloxane content and the nature of curative involved.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Properties

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Wednesday AM Room: London
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Arvid Pasto, Oak Ridge National Laboratory, Director, Oak Ridge, TN 37831-6062 USA

8:30 AM KEYNOTE

WHAT MAKES SILICON NITRIDE WORK: Sheldon M. Wiederhorn¹; ¹National Institute of Standards and Technology, MSEL Bldg 223 Room B309, Gaithersburg, MD 20899 USA

Silicon nitride has now reached the stage where it is an economic material. There are some areas where silicon nitride is accepted commercially (bearings, seals etc.), others that look promising (automobile valves) and still others that look like a promise on the horizon (turbine blades and nozzles). In this talk, I address the subject from the point of view of these practical applications and discuss those property that make the material work. Silicon nitride is a family of materials, and the optimum microstructure is application sensitive. The toughness-strength relation is the critical parameter in valves for example, where high toughness and high strength result in high reliability and long life. In wear applications material parameters are selected to avoid the formation of Hertzian cracks under sliding conditions. Now the properties that control distributed damage in silicon nitride are important. Short crack toughness is now the important design parameter. At high temperature, creep dominates the mechanical behavior. The damage that eventually leads to failure is generated by deformation and oxidation. Now the refractoriness of the intergranular phase that bonds the silicon nitride grains together is the dominate property that leads to low creep rates and long life. In the course of the lecture, we consider economic factors, since these are the deciding factors as to whether silicon nitride is to be used in a given application.

9:10 AM INVITED PAPER

CERAMIC LAMINATES WITH THE FIBROUS MONOLITH ARCHITECTURE: B. King¹; John W. Halloran¹; R. Trice¹; D. Kovar²; G. Hilmas³; ¹University of Michigan, Materials Science and Engineering Department, 2205 H.H. Dow Bldg., Ann Arbor, MI 48109-2125 USA; ²University of Texas, Austin, TX USA; ³Advanced Ceramics Research Inc., Tucson, AZ USA

Fibrous monoliths are two phase monolithic ceramics fabricated from powders using coextrusion methods to create a woody fibrous texture of strong silicon nitride cells and weak boron nitride cell boundaries. With this texture, the fibrous monoliths fracture gracefully, splintering like wood. Uniaxially aligned Si₃N₄/BN fibrous monoliths have peak flexural strengths around 600 MPa and retain about half of their load bearing ability after fracture initiates, with apparent fracture energies around 6000 J/m². Multiaxial materials have about half this strength and fracture energy, but have quasi-isotropic mechanical properties. This paper will discuss the mechanics of these materials, and the influence of the strong cell/weak cell boundary architecture

on properties, elevated temperature properties, and behavior of turbine engine components.

9:40 AM INVITED PAPER

OXIDE LAMINATED COMPOSITES WITH GRACEFUL FAILURE: *Waltraud M. Kriven*¹; ¹Univ. of Illinois at Urbana-Champaign, Materials Science and Engineering, Urbana, IL 61801 USA

A new "transformation weakening" mechanism for inducing debonding in an oxide interphase is introduced. It is based on inducing a displacive phase transformation accompanied by a significant unit cell volume contraction and/or shape change, in a coating around a reinforcement or between laminates of a ceramic matrix. In a thermally-induced transformation, all interphases are pre-transformed before the approach of a crack, with some consequent loss of overall strength of the material. In the ideal, shear-stress induced case, an oncoming crack induces a transformation in its immediate environment as necessary, with strength only minimally reduced throughout the bulk. Maximum toughening may then be achieved, since the propagating crack needs to do work to overcome the nucleation barrier and cause transformation, as well as the onset of other synergistic toughening mechanisms. A model laminate system has been designed and fabricated with a cristobalite (SiO₂) interphase between mullite (3Al₂O₃*2SiO₂) / cordierite (2MgO*2Al₂O₃*5SiO₂) mixed-matrix laminates. The mixed matrix optimized the sinterability of the laminate system, as well as the thermal expansion mismatch between the matrix and interphase layers. In pure SiO₂ the beta (cubic) to alpha (tetragonal) transformation occurs at 265°C on cooling, with a volume change of -3.2 %. For better control of the critical particle size for transformation, the SiO₂ was Al and Ca-doped. Depending on the interphase grain size then, the cristobalite interphase underwent either predominantly thermal or shear-induced transformation, exhibiting characteristics of graceful failure.

10:10 AM BREAK

10:20 AM INVITED PAPER

INTERFACE CONTROL FOR DURABLE CERAMIC COMPOSITES: *Ronald J. Kerans*¹; ¹Air Force Research Laboratory, Materials Directorate, AFRL/MLLN, Wright-Patterson AFB, OH 45433-7817 USA

Interest in ceramic composites is a consequence of their potential for dramatically improved damage tolerance as compared to monolithic ceramics while delivering similar high temperature properties. The remarkable toughening effect resulting from proper combination of three brittle phases in the form of fiber, coating and matrix is a consequence of a particular sequence in the development of damage. It is understood that there are both beneficial and detrimental ways for damage to develop; hence, successful design of composites requires the management of the progression of such damage. The situation is greatly complicated by the fact that the most productive uses of ceramic composites are dependent upon the development of fiber coatings that not only promote the desired interfacial failure, but satisfy the additional requirement of being oxidation resistant. Identification of suitable materials and their evaluation, development and incorporation into composites is key to the development of broadly useful composites. These and other issues related to the development of ceramic composites will be discussed with the intention of identifying topics of interest for future research.

10:50 AM INVITED PAPER

MECHANICAL PERFORMANCE OF POROUS-MATRIX ALL-OXIDE CERAMIC COMPOSITES: *F. W. Zok*¹; *J. A. Heathcote*¹; *X.-Y. Gong*¹; *J. Yang*¹; *U. Ramamurty*²; *C. G. Levi*¹; *F. A. Leckie*³; ¹University of California, Materials Department, Santa Barbara, California 93106 USA; ²Nanyang Technological University, School of Mechanical and Production Engineering Singapore; ³University of California, Mechanical and Environmental Engineering, Santa Barbara, CA 93106 USA

The conventional strategy for imparting high toughness and damage tolerance in fiber-reinforced ceramic composites involves the use of weak interphases between fibers and matrix, providing a path for crack deflection. An alternate approach for promoting such crack deflection is to utilize a highly-porous matrix that is well-bonded to the fibers. Because of its low elastic modulus and low fracture toughness, the matrix acts as the crack-deflecting medium, analogous to the weak interphase in conventional (dense-matrix) composites. The present paper will highlight recent progress in the development and performance of porous-matrix all-oxide ceramic composites. The emphasis will be on the mechanisms and mechanics associated with the in-plane tensile properties, both in the absence and in the presence of holes or notches. To provide a proper perspective, comparisons will be drawn with the behavior of conventional SiC-based composites with weak fiber-matrix interphases.

11:20 AM INVITED PAPER

ADVANCED CHARACTERIZATION TECHNIQUES FOR ADVANCED MATERIALS: *Arvid Pasto*¹; ¹Oak Ridge National Laboratory, Director, High Temperature Materials Laboratory, Oak Ridge, TN 37831-6062 USA

The HTML (High Temperature Materials Laboratory) is a U. S. Department of Energy User Facility, offering opportunities for in-depth characterization of advanced materials, specializing in high-temperature-capable structural ceramics. Available are electron microscopy for microstructural and microchemical analysis, equipment for measurement of the thermophysical and mechanical properties of ceramics to elevated temperatures, x-ray and neutron diffraction for structure and residual stress analysis, and high speed grinding machines with capability for measurement of component shape, tolerances, surface finish, and friction and wear properties. This presentation will focus on structural materials characterization, illustrated with examples of work performed on heat engine materials such as silicon nitride, industrial refractories, metal-and ceramic-matrix composites, and structural alloys. * Research sponsored by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corp. for the U.S. Department of Energy under contract number DE-AC05-96OR22464.

11:50 AM

ELECTRON MICROSCOPE INVESTIGATION OF SILICON CARBIDE FIBER REINFORCED SILICON CARBIDE COMPOSITES: *Mani Gopal*¹; *Gareth Thomas*¹; ¹Department of Materials Science and Mineral Engineering, University of California, Berkeley CA 94720 and, Center for Advanced Materials, Lawrence Berkeley National Laboratory, Berkeley, CA 94720 USA

One class of silicon carbide (SiC) composites is that reinforced with SiC fibers. The fibers are coated with boron nitride (BN) to improve the mechanical properties, by contributing to crack deflection and fiber pull-out. Transmission electron microscopy has been used to characterize the coated fibers and the fiber-reinforced composites. The BN coating on the fiber has been found to be textured, with its basal plane parallel to the surface of the fiber. This texture permits easy deflection of the crack as BN readily cleaves along its basal plane. Analysis of the composite indicates that during processing, interfacial reaction occurs between the fiber and the Si-C matrix. Further, EELS imaging coupled with conventional imaging shows the presence of elemental silicon saturated with carbon. This implies that the reaction between molten silicon and carbon, used to form the SiC matrix, is incomplete. The mechanisms by which this occurs, and steps to improve the processing will be presented. The authors thank T. Kameda of Toshiba for the specimens, NCEM for their cooperation and DOE for the funding.

DAMAGE PROCESSES IN ADVANCED MATERIALS: Intermetallics and Hydrogen Effects

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee, Corrosion and Environmental Effects Committee, Physical Metallurgy Committee

Program Organizers: Russ H. Jones, Pacific Northwest Natl. Lab., Richland, WA 99352 USA; Richard Ricker, NIST, Materials Rm B254, Gaithersburg, MD 20899; Norman S. Stoloff, Rensselaer Polytechnic Inst., MS&E Dept., Troy, NY 12190-3590 USA

Wednesday PM Room: Paris C
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: R. H. Jones, Pacific Northwest National Laboratory, Richland, WA 99352 USA; N. S. Stoloff, Rensselaer Polytechnic Institute, Troy, NY 12180-3590 USA

2:00 PM INVITED PAPER

FATIGUE DAMAGE IN INTERMETALLICS: *N. S. Stoloff*¹; ¹Rensselaer Polytechnic Institute, Materials Science and Engineering Department, Troy, NY 12180-3590 USA

Studies of fatigue damage in intermetallics have lagged behind investigations of tensile ductility and creep behavior. However, there is now a sizeable body of stress-controlled fatigue crack growth data in aluminides and silicides (especially Fe₃Al, TiAl and MoSi₂) as well as strain-controlled low cycle fatigue data for Ni₃Al, NiAl and TiAl. This paper will summarize recent observations of fatigue damage in these intermetallics and some of their composites, and will attempt to correlate microstructure and observed fatigue resistance. In addition, the influence of external environments on fatigue crack growth behavior will be described.

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IMPACT DAMAGE AND HIGH TEMPERATURE FATIGUE BEHAVIOR OF Γ -TiAl: *T. S. Harding*¹; J. W. Jones¹; T. M. Pollack²; M. P. Rubal²; P. S. Steif²; ¹University of Michigan, Ann Arbor, MI 48019 USA; ²Carnegie-Mellon University, Pittsburgh, PA 15213 USA

The elevated temperature fatigue response of gamma titanium aluminide following impact has been investigated. Axial fatigue specimens machined from cast Ti-47.9Al-2.0Cr-1.9Nb and Ti-47.3Al-2.2Nb-0.5Mn-0.4W-0.4Mo-0.23Si alloys, were impacted at low speeds with a 60Y; wedge shaped indenter under controlled conditions in order to simulate manufacturing related damage. Additional specimens from the same alloys were impacted at high speeds with a 1.6 mm steel ball bearing near the specimen edge to simulate leading edge FOD/DOD impacts on turbine blades. Damage was characterized and related to different impact parameters in both scenarios. A step-loading fatigue test procedure is used to determine the fatigue failure stress of the impacted specimens at 650Y. A threshold based fracture mechanics approximation of the failure stress is presented for both impacting conditions. Fractography has been conducted to determine the extent of the initial impact damage within the specimen, and to identify the mechanisms responsible for the propagation of the crack from the impact site. These results are used to verify the threshold based approximation.

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MICROSTRUCTURAL EVOLUTION, CREEP AND TENSILE BEHAVIOR OF A Ti13Al-39Nb BETA+ORTHORHOMBIC ALLOY: *C. J. Boehlert*¹; B. S. Majundar¹; V. Seetharaman¹; D. B. Miracle²; ¹UES Inc., 3301 Dayton-Xenia Road, Dayton, OH 45420-1894 USA; ²Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

The microstructural evolution, creep and tensile deformation behavior of a Ti-13Al-39Nb(at.%) alloy were studied. Monolithic sheet materials were produced through conventional thermomechanical processing techniques comprising nonisothermal forging and pack rolling. TEM studies showed that, depending on the heat-treatment schedule, this alloy contains two constituent phases including: (disordered body-centered cubic) and O (ordered orthorhombic based on Ti₂AlNb). Heat treatments above 800Y, followed by water quenching, resulted in fully- β microstructures. Below 800Y, fine O-phase needles precipitated within the β grains. The fully- β microstructure exhibited a room temperature elongation of more than 27% and a yield strength of 550 MPa. The O+ β microstructures, containing approximately 30 volume percent O, exhibited elongations of more than 12% and yield strengths up to 810 MPa. Metallographic observations revealed that slip was compatible between the two phases. Constant load tensile creep experiments were conducted in the temperature range 650-705Y and stresses were varied between 35-172 MPa. Measured creep exponents and activation energies suggested that the creep mechanisms were dependent on stress. For applied stresses less than 125 MPa, the creep exponents were between 1.6-2.0. For higher stresses, the stress exponents were between 3.5-7.2. The calculated activation energies for the low-stress regime were approximately half those calculated for the high-stress regime. These data suggest that Coble creep operates at low-stress levels and dislocation climb is active at high stresses. The β grain size did not significantly influence the creep rates within the high-stress regime. Within the low-stress regime, grain size did influence the creep behavior and a low dislocation density was observed. Microstructural effects on the tensile properties and creep behavior are discussed in the light of existing models. Overall, the elevated and room-temperature properties of this O+ β alloy are quite attractive.

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MECHANISMS OF FRACTURE AND FATIGUE-CRACK PROPAGATION IN A SHAPE-MEMORY ALLOY TiNi: *A. L. McKelvey*¹; R. O. Ritchie¹; ¹University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720-1760 USA

The effect of stress-induced phase transformations upon fracture and fatigue-crack growth resistance in shape-memory and super-elastic TiNi (Nitinol) are investigated. Particular emphasis is placed on the damage processes during monotonic and cyclic crack growth as a function of temperature, microstructure, and environment. Resistance curve tests indicate that super-elasticity results in crack-tip shielding under monotonic loading. The degradation of crack-tip shielding during cyclic loading and its effect on crack-growth rates are examined. Tests are conducted at temperatures to correspond with non-transforming martensite, transforming martensite (i.e., shape-memory or thermoelastic), transforming austenite (i.e., super-elastic), and non-transforming austenite. As super-elastic Nitinol is a candidate material for cardiovascular stents, additional tests are being conducted to examine the role of crack-tip corrosion at 37Y in deionized water and Hank's solution (a simulated body fluid). The mutual competition of intrinsic microstructural damage mechanisms ahead of the crack tip, which promote crack advance, and extrinsic crack-tip shielding (e.g., transformational toughening and closure) behind the tip, which act to impede it, is investigated.

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HYDROGEN ENVIRONMENT-ASSISTED CRACKING IN BETA-TITANIUM: *B. P. Somerday*¹; R. P. Gangloff²; ¹Sandia National Laboratories, Livermore, CA 94550 USA; ²University of Virginia, Department of Materials Science, Charlottesville, VA USA

The objective of this research is to determine the critical metallurgical factor(s) and associated embrittlement mechanism that govern hydrogen environment-assisted cracking (HEAC) in beta-titanium alloys. The metallurgical variable(s) that govern intergranular (IG) HEAC of Ti-8V-6Cr-4Mo-4Zr-3Al (Beta-C) in aqueous NaCl are examined by fracture mechanics experiments on both immune and susceptible microstructures; including solution heat treated, cold worked, isothermally aged, and double-aged conditions; coupled with high-resolution imaging and analytical electron microscopy. Segregated Si at beta grain boundaries is the likely requisite for IG HEAC in Beta-C. The fracture mechanics measurements and fractographic results for susceptible isothermally aged and double-aged Beta-C microstructures are consistent with effects of heat treatment time and temperature on solute segregation-induced hydrogen embrittlement, and both small-probe TEM EDS and Auger measurements of grain boundary composition reveal elevated Si concentrations. Immunity to HEAC may be achieved in high-strength beta+alpha microstructures by a two-step heat treatment, where initial high-temperature aging for short times depletes Si from the single-phase beta matrix by forming Ti-Zr silicide particles, then subsequent low-temperature aging precipitates alpha. The hydrogen embrittlement mechanism that operates at beta grain boundaries in HEAC-susceptible Beta-C is probed using a field-emission gun SEM, which allows resolution of fine IG fracture surface features. Intergranular cracking of Beta-C in aqueous NaCl is likely from hydrogen-assisted decohesion of grain boundaries, based on the presence of contiguous featureless areas on all fracture surface facets that suggest cracking along a continuous beta/beta interface path. The complex beta+alpha microstructure causes a variety of distributed fine-scale features on decohered grain boundary surfaces. Neither fine dimples nor slip offsets are observed on the surface of most IG facets, indicating that hydrogen-enhanced plasticity does not operate in Beta-C.

DYNAMIC BEHAVIOR OF MATERIALS: Dynamic Behavior III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Flow & Fracture Committee, Mechanical Metallurgy Committee

Program Organizers: Rusty Gray, Los Alamos National Lab, Dynamic Properties, Los Alamos, NM 87545-0001; Marc A. Meyers, IMM, University of California, San Diego, CA 92093 USA; Naresh Thadhani, Georgia Inst of Tech, Sch of Matls Sci & Engrg, Atlanta, GA 30332-0245; Ken S. Vecchio, University of California, Dept of Ames, San Diego, CA 92093 USA

Wednesday PM Room: Sydney
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: John J. Gilman, University of CA, Materials Science and Engineering, Los Angeles, CA 90095 USA; Marc A. Meyers, University of CA, Dept. of AMES, La Jolla, CA 92093-0411 USA

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LOW-VELOCITY-TO-HYPERVELOCITY PENETRATION TRANSITION FOR IMPACT CRATERS IN METAL TARGETS:

Lawrence E. Murr¹; S. A. Quinones¹; *E. T. Ferreyra*¹; A. Ayala²; O. L. Valerio¹; ¹The University of Texas at El Paso, Dept. of Metallurgical

and Materials Engineering, Engineering Science Complex M-201, El Paso, TX 79968-0520 USA

Projectile/target behavior for 1100 Al Cu, soda-lime glass/Cu, soda-lime glass/1100 Al, ferritic stainless steel/Cu, and ferritic stainless steel/1100 Al for a range of spherical (3.18 mm diameter) projectiles at impact velocities ranging from 0.8 km/s to ~6 km/s has been examined by light metallography, SEM, and TEM. At a reference velocity of 1 km/s, the crater depth/crater diameter ratio (p/D_c) is observed to be linearly related to $(\rho_p/\rho_t)^{1/2}$ and $(E_p/E_t)(\rho_p/\rho_t)^{1/2}$; and to vary from about 0.2 to 2.95. The hypervelocity threshold value for p/D_c ($u_p > 5$ km/s) is also shown to be linearly related to these functionalities; ranging from $p/D_c = 0.4$ for the 1100 Al/Cu system and 0.85 for the ferritic stainless steel/1100 Al system.

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INTERFACIAL FRICTIONAL RESISTANCE OF SLIDING INTERFACES AT HIGH SLIP SPEEDS AND HIGH TEMPERATURES:

*Vikas Prakash*¹; Mohammad A. Irfan¹; ¹Case Western Reserve University, Department of Mechanical and Aerospace Engineering, 10900 Euclid Avenue, Cleveland, OH 44106-7222. USA

In the present study, a plate impact pressure-shear loading device is used to investigate the frictional characteristics of sliding interfaces at high slip speeds, high pressures, high temperatures, and time varying normal and shear tractions. The experimental configuration employed represents a significant improvement over conventional dynamic friction experiments by allowing the control of the interfacial tractions through the use of combined pressure-shear waves instead of manipulating actuator motion. The use of planar pressure shear waves enables critical frictional parameters such as the interfacial slip resistance, slip speeds, interfacial temperatures, and the applied normal pressures to be interpreted by the framework of one-dimensional plane wave analysis. Results will be presented for pressure-shear friction experiments conducted on a Ti-6Al-4V/steel tribo pair.

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DYNAMIC BEHAVIOR OF Zr-2.5Nb PRESSURE TUBE MATERIAL: *Darren D. Radford*¹; Mike J. Worswick²; ¹Atomic Energy of Canada, Reactor Core Integrity, WL, Pinawa, Manitoba R0E-1L0 Canada; ²Carleton University, Mechanical and Aerospace Engineering, Ottawa, Ontario K16-5B6 Canada

The current design of Canada Deuterium Uranium (CANDU) nuclear reactors utilize cold-worked Zr-2.5Nb pressure tubes to contain the uranium fuel bundles and the heavy water (D2O) coolant [1]. The tubes are required to operate at temperatures in the range of 250 to 310°C, an internal pressure up to 13 MPa, and a fast ($E > 1$ MeV) neutron flux of 2.5×10^{17} n/m²/s. In certain hypothetical accident scenarios it is postulated that a pressure tube could burst prior to the detection of a through-wall longitudinal crack and the shut-down of the reactor. Assuming also, as a worst-case scenario, that the surrounding calandria tube fails, the longitudinal crack could continue to propagate down the length of the tube. In this situation the pressure tube would deflect vertically due to the release of the pressurized coolant, thereby dynamically loading the ends of the pressure tube in tension. Depending on the final crack geometry and thermal-hydraulic conditions, the ruptured tube could impact surrounding fuel channels causing further in-core damage. Preliminary analyses indicated that plastic deformation at the ends of a rupturing pressure tube would occur at strain rates in the range of 500 to 3000 s⁻¹. In order to accurately simulate pressure tube deformation and the consequences of pressure tube rupture, it is necessary to have an accurate description of the stress-strain behavior of Zr-2.5Nb pressure tube material for the relevant conditions. Although there is a considerable amount of data related to the deformation of Zr [2-6], and Zr-Nb alloys [7-8], it was either obtained at conditions not applicable to pressure tube rupture scenarios, or determined from material with considerably different crystallographic structure and texture. In this investigation, results from high strain rate tensile tests conducted on non-irradiated [9] and irradiated Zr-2.5Nb pressure tube material using a direct-tension version of the split Hopkinson bar apparatus [10, 11] are reported. Specimens were manufactured from both the longitudinal and

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transverse tube orientations and tested at temperatures in the range of 20 to 300°C and strain rates in the range of 500 to 3000 s⁻¹. The irradiated specimens were manufactured from pressure tubes that had been in service for up to 18 years and had received a total neutron fluence of up to 7.4×10^{25} n/m². Results from tests performed on non-irradiated material at 20°C demonstrate that the pressure tube material remains anisotropic at high strain rates [9]. As observed in low strain rate tests [12], the ultimate tensile strength (UTS) in the transverse direction is higher than in the longitudinal direction. The stress-strain behavior for Zr-2.5Nb pressure tube material is quite different depending on the specimens orientation, due to the relative abundance of slip systems in the longitudinal direction and lack of primary slip systems in the transverse direction [13]. The effect of increasing the strain rate was to increase the strength in both directions. The values of flow stress at high strain rates increased approximately 40% above quasi-static values for both specimen orientations. Also, as seen in a number of materials [14] a bilinear dependence of flow stress on logarithm of strain rate was observed, with the strain rate sensitivity being higher at high strain rates. At elevated temperatures, the pressure tube material remained anisotropic and the strength increased above quasi-static values as the strain rate was increased for specimens tested in both directions. The increase in strength was approximately in the same proportions as observed in the 20 °C tests and a bilinear dependence of flow stress on logarithm of strain rate was also exhibited. UTS values obtained at high strain rates and 300 °C were approximately 20% lower than those obtained at high strain rates and 20 °C, for both specimen orientations. Previous results from tensile tests performed on irradiated Zr-2.5Nb pressure tube material at quasi-static strain rates [8] demonstrated that irradiation increases the strength and decreases the ductility in both the longitudinal and transverse directions. These changes occurred at low levels of fluence (1×10^{25} n/m²) and did not increase further as fluence levels increased to approximately 7×10^{25} n/m². The overall stress-strain responses, as a function of tube orientation, were consistent with that of the non-irradiated material, and UTS values remained higher in the transverse direction. The effect of irradiation on the tensile properties at high strain rates was to increase the strength and decrease the ductility for all test temperatures. The reduction in ductility at high strain rates, however, was less than that observed at quasi-static strain rates. As observed in the quasi-static tests, the increase in strength due to irradiation took place at low fluences and did not significantly change for fluence levels up to approximately 7×10^{25} n/m². A bilinear dependence of flow stress on logarithm of strain rate was also observed in the irradiated material for strain rates ranging from 1×10^{-4} to 3×10^3 s⁻¹.Re

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DYNAMIC HARDNESS AND RATE SENSITIVITY IN METALS: *Ghatu Subhashi*¹; Brian J. Koepfel¹; ¹Michigan Technological University, Mechanical Engineering-Engineering Mechanics Department, 1400 Townsend Drive, Houghton, MI 49931 USA

Indentation hardness is one of the simplest and the most commonly used measures for quickly characterizing material response under static loads. Hardness may mean resistance to cutting to a machinist, resistance to wear to a tribologist, or a measure of flow stress to a design engineer. In this simple technique, a predetermined force is applied to an indenter for 5-30 seconds causing it to penetrate a specimen. By measuring the load and the indentation size, a hardness value is determined. However, the rate of deformation during indenter penetration is of the order of 10⁻⁴ s⁻¹. In most practical applications, such as high speed machining or impact, material deformation occurs at strain rates in excess of 10³-10⁵ s⁻¹. At such high strain rates, it is well established that the plastic behavior of materials is considerably different from their static counterpart. For example, materials exhibit an increase in their yield stress, flow stress, fracture stress, and fracture toughness at high strain rates. Hence, the use of static hardness as an indicator of material response under dynamic loads may not be appropriate. Accordingly, a simple dynamic indentation hardness tester is developed for characterizing materials at strain rates similar to those encountered in dynamic situations. The experimental technique uses elastic stress wave propagation phenom-

ena in a slender rod. The technique is designed to deliver a single indentation load of 100-200 microseconds duration. Similar to static measurements, the dynamic hardness is determined from the measured load and indentation size. Hardness measurements on a range of metals have revealed that the dynamic hardness is consistently greater than the static hardness. The percent increase in hardness under dynamic conditions is found to be similar in magnitude to the percent increase in yield stress under uniaxial compression. Therefore, it is suggested that the current technique can be used to quickly assess the rate sensitive nature of engineering materials. To further characterize the plastic strains within the indentation volume, static microhardness measurements were performed within this region. The contours of microhardness values thus generated give a measure of accumulated plastic strains beneath the indenter. It was observed that this plastic zone is typically smaller under dynamic conditions compared to static loading for rate sensitive materials. To assess the influence of the elastic modulus, yield stress, and work hardening coefficient on the induced plastic volume, finite element simulations were performed using the explicit finite element code LS-DYNA3D. The parametric study revealed that the yield stress has the most significant influence on the size and shape of the plastic zone. The above microstructural and numerical results can be used as guidelines for proper selection and design of engineering materials in applications involving high strain rate loading. Moreover, the contours of microhardness variation can be used to verify the suitability of analytical models developed for characterizing the deformation behavior of engineering materials under complex three dimensional loading conditions.

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DAMAGE EVOLUTION OF DYNAMIC DEFORMATION OF SILICON CARBIDE: *C. James Shih*¹; Marc A. Meyers²; Vitali F. Nesterenko²; ¹Ceradyne Inc, 3169 Redhill Avenue, Costa Mesa, CA 92626 USA; ²UCSD, Materials Science, Mail Code 0411, 9500 Gilman Drive, La Jolla, CA 92093-0411 USA

Damage evolution was investigated in silicon carbide by subjecting it to dynamic deformation in (a) a compression Hopkinson bar (compressive stresses of 5 GPa), and (b) high-velocity impact under confinement (compressive stresses of 19 to 32 GPa) by a cylindrical tungsten alloy projectile. Considerable evidence of plastic deformation, as dislocations and stacking faults, was observed in the fractured specimens. A polytype transformation was observed through a significant increase in the 6H-SiC phase at compressive stresses higher than 4.5 GPa (in the vicinity of the dynamic compressive failure strength). Profuse dislocation activity was observed in the frontal layer in the specimen recovered from the projectile impact. The formation of this frontal layer is proposed to be related to the high lateral confinement, imposed by the surrounding material. The microstructural defects and evolution were found to be dependent on the concentration of boron and aluminum, which were added as sintering aids. Finite element calculations showed that tensile stresses are generated due to elastic compatibility strains. Two mechanisms are proposed for the initiation of fracture: dilatant cracks and Zener-Stroh cracks. Dilatant cracks are induced by mismatch in the effective elastic moduli between two adjacent grains, leading to internal tensile stresses and creating transgranular fracture. Zener-Stroh cracks are nucleated by the piled up dislocations along grain boundaries, and result in intergranular fracture. The high dislocation density observed in the impacted specimen is consistent with existing models of microplasticity. This research is supported by the U.S. Army Research Office through AASERT (DAAH04-94-G-0314) and MURI (DAAH04-96-1-0376) programs, and the National Science Foundation through the Institute for Mechanics and Materials at the University of California, San Diego.

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INFLUENCE OF COLD ROLLING ON PLASTIC RESPONSE OF PM AND CVD RHENIUM: *Ghatu Subhashi*¹; Brian J. Koepfel¹;

¹Michigan Technological University, Mechanical Engineering-Engineering Mechanics Department, 1400 Townsend Drive, Houghton, MI 49931 USA

Plastic response of rhenium (Re) produced by powder metallurgy (PM) and chemical vapor deposition (CVD) was investigated under uniaxial compression at a range of strain rates. The PM Re was also cold rolled by 50% and 80% reduction (denoted as PM50 and PM80, respectively) in thickness. Both cold rolled and CVD Re have been found to have a strong basal texture. Rhenium exhibited a two stage hardening in its plastic response. It revealed a high strain hardening rate and a strong strain rate sensitivity of flow stress. Increased cold work in the PM Re resulted in an increase in the initial yield stress with a concurrent reduction in the hardening rate and failure strain. CVD Re exhibited a distinctly higher hardening rate and ultimate flow stress than the above cold rolled PM Re. After accumulating a certain amount of plastic strain, the deformation in the specimens localized leading to formation of shear bands at an angle to the loading axis. Fracture immediately followed the shear bands. Twinning was found to be a dominant deformation mode in both PM and CVD Re. Extensive twinning was observed in larger grains of CVD Re. Twins in fine grained PM specimens were less common and occurred primarily in the regions close to the shearband. Based on the experimental results and microscopic investigations, the micromechanisms of deformation responsible for the observed behavior of Re will be discussed.

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QUASI-STATIC AND DYNAMIC CRUSHING OF AN ALUMINUM CLOSED CELL FOAM: *Ian W. Hall*¹; *Mustafa Guden*²; ¹Department of Mechanical Engineering, University of Delaware, Spencer Laboratory, Newark, DE 19716 USA; ²Izmir Institute of Technology, Gaziosmanpasa Bulvarı No:16, Cankaya, Izmir Turkey

An aluminum 6061 closed cell foam has been compression tested at quasi-static (1.5x10⁻³s⁻¹) and high strain rates (400 and 1700 s⁻¹) in order to assess strain rate effect on the flow stress /strain behavior of the foam. Experimental results have shown that increasing strain rate from quasi-static to dynamic increased the flow stress and hence energy absorption capacity of the foam. Application of scaling relations to the flow stress vs. relative density of the foam suggested that increased flow stress at high strain rates compared to quasi-static rates was due to the strain rate sensitive flow stress behavior of the foam alloy. Microscopic observations clearly showed that compression of the foam resulted in crushing of the cell walls by buckling and tearing in localized regions and deformation was progressed from localized through the undeformed regions of the foam. The final deformation stage, densification, was the result of cell touching which significantly increased the flow stress of the foam at relatively large strains.

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AN ANALYSIS OF THE CRSS FOR TWINNING IN LOW-SYMMETRY METALS: *George C. Kaschner*¹; *Thomas A. Mason*²; *George T. Gray*¹; ¹Los Alamos National Laboratory, MST-8, M/S G755, Los Alamos, NM 87545 USA; ²Los Alamos National Laboratory, MST-6, M/S G755, Los Alamos, NM 87545 USA

Previous work has demonstrated that the deformation of low-symmetry metals is a balance of accommodation by dislocation slip and deformation twinning. Zirconium was selected as the model material to explore the correlation of local crystallographic orientation and the CRSS for twinning. Samples were tested in cryogenic quasi-static conditions and characterized using the recently developed automatic electron backscatter diffraction techniques of OIM. This analysis will attempt to correlate the volume of twinned matrix and the accommodation of strain in textured h.c.p. metals.

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INFLUENCE OF ALLOYING ELEMENTS ON THE STRAIN RATE AND TEMPERATURE DEPENDENCE OF THE FLOW STRESS OF STEELS: *Volker Schulze*¹; *Otmar Voehringer*¹; ¹Universitaet

Karlsruhe (TH), Institut fuer Werkstoffkunde I, Kaiserstr. 12, Karlsruhe, FRG 76128 Germany

The relation between flow stress σ , strain rate $\dot{\epsilon}$ and temperature T of metallic materials is controlled for $\dot{\epsilon} < 10^4 \text{s}^{-1}$ and $T < 0.3 T_m$ (T_m = melting temperature in K) by the thermally activated slip of dislocations over short-range obstacles. The quantitative description of the effect of strain rate and T on the flow stress is given by the constitutive equation $\sigma = \sigma_G + \sigma_o^* (1 - (T/T_o)^n)^m$ with G_o = free activation enthalpy, k = Boltzmann constant, $\dot{\epsilon}_o$ = strain rate constant, σ_G = athermal flow stress, σ^* = thermal flow stress at $T = 0$ K and m, n = exponents which characterize the force-distance shape of the short range obstacle. The properties of the constitutive equation were estimated for several plain carbon steels in normalized conditions as well as quenched and tempered alloyed steels based on their temperature dependence and strain rate sensitivity of the flow stress at different $\dot{\epsilon}$ -values and low temperatures. Increasing contents of soluted alloying elements lead to decreasing values of σ_o^* and ΔG_o . This effect is called pseudo-alloying-softening and depends strongly on type and content of the alloying element. It will be discussed from a microstructural point of view.

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STRUCTURAL TRANSITIONS IN ENSEMBLES OF MESODEFECTS AS MECHANISM OF SHEAR LOCALISATION UNDER IMPACT LOADING: *Oleg Borisovich Naimark*¹; ¹ICMM, Russian Acad.Sci., Phys.Foundation of Strength, 1 Acad.Korolev Str., Perm 614013 Russia

Surprising universality for viscosity value of shocked materials obtained first by Sakharov's group is explained. This property is caused by orientation transition in microshear ensemble under subjection of relaxation material ability to self-similar defect ensemble behaviour which corresponds to resonance excitation of solitary waves of orientation defect instability. The latter leads to strain localisation waves propagating with velocities determined by orientation transition kinetics. New nature of shear strain instability is shown. Phenomenology modification and minimum testing extension is proposed for dynamic problem studying. Conditions of resonance excitation of self-similar regimes are studied theoretically and experimentally for projectile-target interaction.

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EVOLVING PARADIGMS IN MICROSTRUCTURE EVOLUTION: A SYMPOSIUM DEDICATED TO DR. JOHN W. CAHN: Thermodynamics and Concluding Talk by J. W. Cahn

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Chemistry & Physics of Materials Committee, Phase Transformations Committee, Thermodynamics & Phase Equilibria Committee
Program Organizers: William C. Johnson, University of Virginia, Materials Science & Engineering Dept.; William J. Boettinger, National Institute of Standards & Technology, Room A153 Bldg 223; Carol Handwerker, National Institute of Standards & Technology, Room A153 Bldg 223, Gaithersburg, MD 20899 USA; Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Dept.; John Morral, University of Connecticut, Dept. of Metallurgy, Storrs, CT 6260

Wednesday PM Room: International Ballroom
 October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Sam Allen, MIT

2:00 PM INVITED PAPER

CLASSIFICATION OF PHASE TRANSFORMATIONS: *John A. L. Ågren*¹; ¹Royal Institute of Technology, Materials Science and Engineering, S-100 44, Stockholm 100 44 Sweden

Phase transformations may be classified in many different ways. The division introduced by Gibbs into homogenous and heterogeneous transformation as well as Ehrenfest division into first, second and higher order transformations are well known to all students in physics, chemistry and materials science. Other ways of classifying the transformations are based on the most important atomistic or molecular mechanisms, e.g. diffusional and displacive transformations. In the talk various classification methods are discussed and criticized from a practical as well as a more fundamental point of view. For example, it is shown that it is generally not meaningful to consider first and second order transformations as equivalent with heterogeneous and homogeneous transformations respectively.

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FROM NON-EQUILIBRIUM TO EQUILIBRIUM STUDIED BY CLUSTER VARIATION AND PATH PROBABILITY METHOD: *Tetsuo Mohri*¹; ¹Hokkaido University, Division of Materials Science and Engineering, Graduate School of Engineering, Sapporo 060-8628 Japan

Cluster Variation Method (CVM) has been recognized as one of the powerful theoretical tools to predict and to analyze phase equilibria in an alloy system. The combination with electronic structure calculations enable us to investigate the phase equilibria even from the first-principles. Path Probability Method (PPM) is the natural extension of the CVM to time domain, and it has been confirmed that the derived quantities in the long time limit exactly reproduce the equilibrium ones independently obtained by the CVM. Hence, the combination of the CVM and PPM is best suited to study time evolution and devolution behavior of microscopic quantities starting from non-equilibrium state towards the equilibrium state. Among the various calculations attempted, phase transition kinetics and relaxation kinetics are discussed.

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THE JOYS AND NEVER-ENDING CHALLENGES OF PARADIGM BUILDING; WILL OURS EVER BE FLAWLESS?: *John W. Cahn*¹; ¹NIST, Materials Science and Engineering Laboratory, Materials Building, Room A153, Gaithersburg, MD 20899 Xanadu

Fundamental ideas have played a central role in the long history of our field and led to many useful inventions and discoveries, even when they were later proven wrong. The scientific revolution has changed many of these ideas, and has even changed their nature. The pace of these changes is accelerating as our field keeps expanding. Paradigms provide a framework for the development of ideas, but ones created for fluids are severely challenged when applied to solids; ones advanced for metals may not survive without modification when applied to polymers or ceramics. New experimental and theoretical tools permit the asking of new questions and examination of previously inaccessible details. Some of the recent changes will be examined for the challenges they still pose.

FATIGUE BEHAVIOR OF TITANIUM ALLOYS: Application, Life Prediction and Design

Sponsored by: Structural Materials Division, Titanium Committee
Program Organizers: Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; Daniel Eylon, Univ. of Dayton, Graduate Materials Engineering, Dayton, OH 45469-0240; J. P. Gallagher, University of Dayton, UDRI-Structural Integrity, Dayton, OH 45469-0120 USA; G. Lutjering, TU Hamburg-Harburg, Hamburg 21093 Germany

Wednesday PM Room: Athens/Berlin
 October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: J. Larsen; D. Eylon

2:00 PM KEYNOTE

APPLICATION AND LIFE PREDICTION OF TITANIUM ALLOYS IN AIRCRAFT GAS TURBINE ENGINES: *B. A. Cowles*¹; ¹Pratt & Whitney, P.O. Box 109600, MS 707-22, West Palm Beach, FL 33410-9600 USA

Since initial introduction in the 1950's, application of titanium alloys has steadily increased in aircraft gas turbine engines. The low density and high specific strength of titanium alloys have contributed significantly toward attainment of today's high thrust, light weight, extremely efficient engines. Today, titanium alloys comprise more than one-third of total engine weight, much of it in structurally critical parts such as fan and compressor rotors and airfoils, and engine mainframe structures. Materials processing, and structural design, durability and life prediction practices have continuously evolved to facilitate such applications. This paper presents an overview of current titanium applications in gas turbine engines, the associated structural and durability considerations, and areas that appear significant for future applications.

2:30 PM INVITED PAPER

APPLICATION OF Ti-ALLOYS AS COMPRESSOR DISKS AND BLADES: *D. Helm*¹; ¹MTU, Munchen, FRG 80976 Germany

Ti-alloys are widely used as materials for compressor discs, blades, vanes and housings in modern aero-engines due to their excellent strength-to-weight ratio. Their high corrosion resistance and good weldability are additional factors for their application as main com-

pressor components. A variety of different Ti-alloys have been developed in recent years and introduced into jet engines in order to fulfill the large spectrum of required mechanical properties. The main topic of the present paper is to describe the correlation between essential microstructural features of a number of Ti-alloys for compressor disc and blade applications and such mechanical properties as strength, creep and fatigue resistance, and crack propagation behavior. It will be shown that for different engine requirements the choice of the suitable alloy, as well as tailoring of microstructural features with the aim to achieve an optimized balance of relevant mechanical properties, is necessary. The influence of thermomechanical treatment on morphology and dimensions of phases, and the resulting correlation to mechanical properties, will be discussed in detail for selected Ti-alloys, i.e. Ti-6Al-4V, Ti-6Al-2Sn-4Zr-2Mo and IMI 834 (Ti-5.8Al-4Sn-3.5Zr-0.5Mo-0.7Nb-0.35Si). Additionally, effects of surface conditions caused by surface treatment (shot peening) or by service exposure (surface oxidation) will be presented.

2:50 PM INVITED PAPER

TITANIUM IN FATIGUE CRITICAL MILITARY AIRCRAFT STRUCTURES: *Franna Gillespie*¹; ¹The Boeing Company, Information, Space and Defense Systems; Military Airplanes, P.O. Box 3707, Seattle, WA 98124 USA

Titanium comprises a major portion of military aircraft structure, in a large part because of its temperature capability, corrosion resistance, and high specific strength. Today's military aircraft have increased performance requirements to the point that static strength in titanium is much less important than fatigue performance, fatigue crack growth and fracture toughness. A combination of analysis techniques and manufacturing processes are used by the designer to ensure that future military aircraft meet the customer's requirements. This paper describes the impact of fatigue analyses and designated manufacturing processes on titanium structure.

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FATIGUE CRACK INITIATION AND FATIGUE LIFE PREDICTION OF Ti-6Al-4V ELI: *M. Niinomi*¹; *T. Akahori*¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

The specimen of Ti-6Al-4V ELI with Widmanstätten α structure fractures before adequate fatigue hardening because a lot of microstructure-cracks are easily formed while the specimen with equiaxed α structure fractures after adequate fatigue hardening. The mechanical properties, that is, tensile strength and hardness tend to increase clearly according to the fatigue steps in particular in the low cycle fatigue (LCF) region, while impact toughness and elongation show the reverse trend. The hardness far from the specimen surface is greater than that near the specimen surface at the early stage of LCF. Both hardnesses are however nearly equal each other at the latter stage of fatigue. It can be understood therefore that the dislocation multiplies near the specimen surface at the early stage of fatigue while at the latter stage of fatigue, the dislocation multiplies also near the center of the specimen. The dislocation multiplication will finally saturate in the whole specimen.

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DESIGN AND QUALIFICATION OF TITANIUM FOR DEEPWATER HIGH PRESSURE DRILLING RISERS: *J. Murali*¹; *M. M. Salama*²; *M. W. Joosten*²; ¹Norske Conoco AS, Tangen 7, P.O. Box 488, 4070 Randaberg, Stavanger 4001 Norway; ²Conoco Inc., 100 South Pine, P.O. Box 1267, Ponca City, OK 74603 USA

Titanium alloy Ti-6Al-4V ELI was selected for fabricating Heidrun's high pressure drilling riser because of its high specific strength, corrosion resistance, and favorable elastic properties. The qualification of this titanium alloy required assessing its resistance to hydrogen embrittlement and stress corrosion cracking due to seawater

with/without cathodic protection, evaluating its wear resistance against a rotating steel drill string, and studying the influence of service induced defects on fatigue and crack growth behavior when subjected to the operating environment. The paper presents an overview of the design requirement and the materials performance properties which were proposed, evaluated, specified and validated for the Ti-6Al-4V ELI used in this application.

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MICROSTRUCTURE & TEXTURE MODEL FOR THE MEAN STRESS SENSITIVITY OF HCF STRENGTH IN A+B TITANIUM ALLOYS: *Frederic Cohen*¹; *S. Ivanova*²; *R. D. Sisson*²; *R. R. Biederman*²; ¹Pratt & Whitney, Mail Stop 114-40, 400 Main Street, East Hartford, CT 06108 USA; ²Worcester Polytechnic Institute, 100 Institute Road, Worcester, MA 01609-2280 USA

The effect of microstructure and crystallographic texture on the mean stress sensitivity of the fatigue strength has been investigated for equiaxed and bi-modal microstructures in Ti-6Al-4V. Most microstructures exhibited a pronounced mean stress sensitivity. A quantitative model has been developed which accurately predicts the fatigue strength and its mean stress dependence as a function of the microstructure and texture characteristics of the $\alpha + \beta$ titanium alloys.

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FATIGUE CHARACTERISTICS OF BIOMEDICAL TITANIUM ALLOYS: *Mitsuo Niinomi*¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

Fatigue characteristics and microstructure relation of various biomedical titanium alloys like Ti-5Al-2.5Fe, Ti-6Al-7Nb and Ti-6Al-4V ELI were investigated in air and simulated body environment, that is, Ringer's solution. Fatigue strength of Ti-5Al-2.5Fe with various microstructures is not degraded in ordinary Ringer's solution while that of stainless steel, 316L, degraded. However, fatigue strength of Ti-5Al-2.5Fe is degraded in Ringer's solution with N_2 gas flow, that is, low oxygen content. The long fatigue crack propagation rate, da/dN , of Ti-5Al-2.5Fe is accelerated in ordinary Ringer's solution when da/dN is related with nominal cyclic intensity factor range, ΔK , while da/dN in ordinary Ringer's solution is nearly the same as that in the air when da/dN is related with effective cyclic stress intensity factor range, ΔK_{eff} . The short fatigue crack propagation characteristics of Ti-6Al-7Nb with various microstructures will be also reported, comparing with that of Ti-6Al-4V ELI.

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EFFECT OF SURFACE FINISHING ON FATIGUE PERFORMANCE OF THE SURGICAL IMPLANT ALLOY Ti-6Al-7Nb: *J. Kiese*¹; *U. Holzwarth*¹; *L. Wagner*¹; ¹Technical University of Brandenburg at Cottbus, Department of Materials Technology, Postfach 101344, Cottbus 03013 Germany

Latest technologies in designing and testing of new implants require higher strength in addition to improved fatigue performance in comparison to the properties of the as-delivered condition. The decision to use the reliable alloy Ti-6Al-7Nb was made as a result of its high biocompatibility and its high potential of property improvement. Thermomechanical treatments were used to develop duplex microstructures in Ti-6Al-7Nb, containing 20 and 60% primary- α . By varying the cooling rate from the duplex anneal, two strength levels of the lamellar matrix were achieved. In addition, mechanical surface treatments such as shot peening and roller-burnishing were performed to improve the fatigue resistance. Hour-glass shaped specimens were shot peened and roller-burnished by a wide range of Almen intensity and working pressure, respectively, to evaluate optimum conditions for fatigue performance. A part of specimens was additionally grit blasted with Al₂O₃ to increase the surface roughness and prevent iron contamination of the titanium surface that may be caused by embedded steel particles from the shot peening process. Fatigue behavior was studied in rotating beam loading at frequencies of 60 Hz in air and corrosive Ringer's solution and compared to the electrolyti-

cally polished reference. A combination of suitable microstructure and optimum surface treatment led to an increase in fatigue strength of Ti-6Al-7Nb of up to 60%. The results will be explained in terms of the influence of surface roughness, induced dislocation density and residual stresses on crack nucleation and microcrack growth.

HOT DEFORMATION OF ALUMINUM ALLOYS: Process Simulation and Development - II

Sponsored by: Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Thomas R. Bieler, Michigan State University, Dept. of Matls Sci & Mechanics, East Lansing, MI 48824-1226; Lawrence Lalli, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Stuart MacEwen, Alcan International, Kingston, Ontario K7L 5L9 Canada

Wednesday PM Room: Orchard
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chairs: Terrence Langdon, University of Southern California, Departments of Material Science & Mechanical Engineering, Los Angeles, CA 90089-1453 USA; M. E. Kassner, Oregon State University, Dept. of Mechanical Engineering, Corvallis, OR 97331 USA

2:00 PM INVITED PAPER

SIMULATION OF COMMERCIAL HOT ROLLING BY LABORATORY PLANE STRAIN COMPRESSION AND ITS APPLICATION TO ALUMINUM INDUSTRY CHALLENGES: *G. J. Marshall*¹; ¹Alcan International, Banbury Laboratory, Southam Road, Banbury OX167SP UK

The majority of semi-fabricated, flat rolled products are manufactured by the aluminium industry using large and expensive hot and cold rolling facilities to meet the high standards required for subsequent downstream processing and in-service performance. For many the hot rolling stage is crucial in determining the correct microstructure, including crystallographic texture, prior to cold rolling and, in some cases, final annealing. On-line monitoring for the control of metallurgical parameters is in its infancy within the industry and strict practices for process control are enforced to achieve the correct metallurgy. These practices are traditionally established from experience and plant trials. In recent years the determination of process-microstructure-property relationships has relied on extensive use of laboratory plane strain compression (PSC) testing and the introduction of this information into physically based models. In 1991 Alcan's Banbury Laboratory designed and commissioned a PSC rig to simulate the deformation history of commercial hot multi-stand rolling (tandem mill rolling), typical of the major Aluminium rolling companies. This paper will review the capability of the Alcan PSC tester with reference to current industrial challenges in the production of packaging, architectural and transportation products. Examples will be provided to demonstrate particular issues, progress towards solutions and future objectives.

2:30 PM INVITED PAPER

RESTORATION MECHANISMS AND HOT ROLLING OF AL ALLOYS: *H. J. McQueen*¹; ¹Concordia University, Mechanical Engineering, Montreal H3G 1M8 Canada

The industrial rolling of Al alloys is generally conducted in multi-stage schedules of 10-15 passes, partly on reversing and partly on continuous mills, with temperature declining from 500 to between

300 and 250°C (which might be called warm rolling). Such schedules have been physically simulated on a torsion machine with measurement of flow stresses and interpass softening microstructures were examined at many stages. During the passes of 10-40% reduction, recrystallized material undergoes monotonic hardening possibly to steady state, with development of a dynamically recovered substructure in elongated grains. During most intervals, only static recovery takes place so that in the following passes, the flow curves exhibit higher initial stresses but less strain hardening than described above. Static recrystallization may take place to some degree in long intervals after passes with higher temperature and strain so that the behavior in the following pass is some combination of those described above according to the role of mixtures. The constitutive equations determined for recrystallized aluminum and particle alloys are not applicable to calculation of the mean stresses in the passes of such schedules because of the recovered substructure carried over. However, those determined for Al-Mg alloys appear to be applicable because the solute drag effect has a more significant role in defining initial flow stress than the substructure.

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COMPARISON OF EXTRUSION MODELING OF AA7075 WITH OTHER ALLOYS AND COMPOSITES: *E. V. Konopleva*¹; M. Sauerborn¹; H. J. McQueen¹; ¹Concordia University, Mechanical Engineering, Montreal H3G 1M8 Canada

The extrusion of AA7075 alloy was modeled by DEFORMTM for comparison with AA6061 and AA2618 alloys and particulate composites of those three alloys that had been prepared by liquid metal mixing. The modeling was based on constitutive data established over ranges 250-540°C and 0.1 to 4.0 s⁻¹ by torsion testing. In these tests hot ductility of 7075 declined above 400°C become much lower than 2618 which has low incipient melting (503°C) and 6061 which has very high ductility. The extrusion was modeled for a range of billet temperatures 350-500°C, ram speeds 2.6 and 5 mm/s and extrusion ratio of 31. Distribution of velocity, strain rate, temperature and mean stress were determined to clarify the process details. The maximum loads and temperatures are compared to understand the relative extrudability.

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MICROSTRUCTURAL DEVELOPMENT FOR SUPERPLASTICITY IN Al-Mg-Mn ALLOYS: *J. S. Vetrano*¹; M. T. Smith¹; C. H. Henager¹; S. M. Bruemmer¹; ¹Pacific Northwest National Laboratory, P.O. Box 999, Richland, WA 99352 USA

Thermomechanical processing and alloying additions have been utilized in Al-Mg-Mn alloys to create microstructures suitable for superplastic deformation. Superplasticity typically requires a grain size of <10 µm that is stable against growth at temperatures close to the melting point of the material. We have examined the role of Mn, Sc and Zr additions on recrystallization and grain growth during thermomechanical processing and superplastic deformation of cast Al-Mg alloys. Particles containing Mn which formed during solidification were observed to be efficient nuclei for recrystallization but their effectiveness was reduced when Zr was present. This was a result of a shape change in these particles which did not allow them to break up and disperse during hot and cold rolling. Smaller Al₆Mn dispersoids, Al₃Zr and Al₃Sc particles acted to pin grain boundary migration. Their effectiveness was maximized by tailored heat treatments to create an optimum dispersion and particle size. Grain sizes less than 5µm could be achieved by conventional cold-rolling and recrystallization treatments, resulting in superplastic elongations of more than 700%. *Work supported by the Materials Division, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC06-76RLO 1830

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FLOW AND FRACTURE BEHAVIOR DURING WARM FORMING OF ALUMINUM ALLOYS: *A. K. Ghosh*¹; Z. Guo¹; ¹The University of Michigan, Ann Arbor, MI 48109-2136 USA

Constitutive Behavior — temperature and strain rate effects, microstructural and textural changes, cavitation and plastic growth of voids.

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REFINEMENT OF CONE TESTING METHODOLOGY FOR CAVITATION STUDIES OF SUPERPLASTIC ALUMINUM ALLOYS: *V. I. Levit*¹; R. Crooks¹; R. N. Shenoy¹; Yu N. Gornostyrev²; ¹Analytical Services and Materials, Inc., 107 Research Drive, Hampton, VA 23666 USA; ²Institute of Metal Physics RAS, 18 Kovalevskaya St., Yekaterinburg 620219 Russia

Cavitation occurs during superplastic forming (SPF) of aluminum alloys primarily as a result of decohesion at grain or interphase boundaries. Cone testing allowed the evaluation of temperature, pressure differential and back pressure effects on: maximum strain at fracture, strain distribution inside the cone, total porosity level and porosity size and morphology. A few geometrical parameters were used to both characterize and simulate strain distribution in the cone test. The cavitation behavior in a 2xxx series and selected other superplastic aluminum alloys was characterized in the cone test samples using quantitative metallography and density measurements. Images showing pore morphologies and distributions were collected by scanning electron microscopy (SEM) of metallographically sectioned cones. A commercial image analysis package was then used to determine the dimensions, volume fraction and various distribution statistics of the cavities. The total porosity measured by SEM was in good agreement with the density measurements. Characteristic morphologies were attributable to forming parameters, especially temperature and strain rate. A useful correlation was found for representations of the distribution of pore volume fraction based on total volume within given pore-size ranges. Using this approach it is possible to distinguish between "ordinary" cavitation, which accompanies superplastic deformation processes, and the stage of void coalescence which precedes fracture.* Work performed at NASA Langley Research Center under contract NAS1-96014.

MATERIALS CHARACTERIZATION VIA THE INTERNET

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Committee

Program Organizers: L. Allard, Oak Ridge National Laboratory; K. Alexander, Oak Ridge National Laboratory; U. Kahmen, Lawrence Berkeley National Laboratory

Wednesday PM Room: Dublin
October 15, 1998 Location: O'Hare Hilton Hotel

Session Chair: L. Allard, Oak Ridge National Laboratory, Oak Ridge, TN

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MATERIALS CHARACTERIZATION VIA THE INTERNET:

The combination of high speed communication via the Internet and the increasing capabilities of desktop computers has made it possible to extend the control of expensive scientific instruments such as electron microscopes from the laboratory to distant locations. It is now possible for scientists at widely disparate locations to participate in a "TelePresence Microscopy" session, using inexpensive teleconferencing tools and their own personal computers. This includes the ability to assume full control of the microscope operation, if desired, so that a local operator need not even be present. Collaborators doing materials research can view results and share ideas in real time, improving the potential to obtain reliable interpretation and understanding of the

data. The implications of this new way for scientists to work together, unencumbered by time and space, to do better science will be explored via live demonstrations of TelePresence Microscopy with several national laboratory microscopy facilities.

MECHANISMS AND MECHANICS OF COMPOSITE FRACTURE: Particle Reinforced Composites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Composite Materials Committee

Program Organizers: Bhaskar S. Majumdar, UES, Inc., Dayton, OH 45432-1894 USA; S. Krishnamurthy, UES, Inc., Dayton, OH 45432-1894 USA; Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA

Wednesday PM Room: Paris A
October 14, 1998 Location: O'Hare Hilton Hotel

Session Chair: Benji Maruyama, Air Force Research Laboratories, Materials and Manufacturing Directorate, Wright Patterson Air Force Base, OH 45433 USA

2:00 PM INVITED PAPER

FACTORS AFFECTING THE FRACTURE TOUGHNESS OF COMPOSITES: *John J. Lewandowski*¹; ¹Case Western Reserve University, Dept. Materials Science and Engineering, 10900 Euclid Ave., Cleveland, OH 44106 USA

The fracture toughness of discontinuously reinforced aluminum (DRA) is affected by a number of factors controlled by the reinforcement volume fraction, size, and distribution, while equally important microstructural effects on the evolution of damage and fracture toughness have been recorded. The presentation will provide a review of ongoing work in the field which has been conducted on such systems where systematic changes to the variables outlined above have been conducted. The talk will conclude with a brief review of opportunities for toughness enhancement in such systems and the variables which have been identified that contribute to the magnitude of toughening possible.

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EFFECTS OF INTERFACIAL PFZ LAYERS AND PRECIPITATES ON STRENGTH OF AGE-HARDENABLE ALUMINUM MATRIX MMCS: *Toshiro Kobayashi*¹; Hiroyuki Toda¹; ¹Toyohashi University of Technology, Department of Production Systems Engineering, Toyohashi, Aichi 441-8580 Japan

The existence of the thin layers of precipitate free zone have been observed around reinforcements in several MMCs having age-hardenable aluminum alloys as matrices, together with the formation of coarse interfacial precipitates and local solute atom segregation. The effects of such inhomogeneity of the matrix microstructure are analyzed by means of the elastic-plastic finite element analysis. The plastic flow of a matrix is concentrated within the PFZ layers due to its ductile nature, thereby reducing the overall strength of MMCs. On the other hand, the interfacial precipitates effectively retard the concentrated plastic flow within the PFZ layers. The interfacial precipitates never exhibit such preferable behavior when the reinforcements are surrounded by the homogeneous matrix. Both the axial stresses within the reinforcements and the cavity formation rate at the interfaces increase with increasing the thickness of the PFZ layers and the size of the interfacial precipitates. The cavity formation rate is re-

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markedly affected by the morphology and density of the interfacial precipitates.

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FAILURE MECHANISMS AND MODELING OF FRACTURE OF

DRA: *B. S. Majumdar*¹; *D. B. Miracle*²; ¹AFRL, Materials Directorate, Wright Patterson Air Force Base, OH 45433 USA; ²UES, Inc., Dayton, OH 45432 USA

In this study, the mechanisms of failure of discontinuously reinforced aluminum (DRA) alloy composites were investigated using a 15 volume percent 7093-Al-alloy/SiCp composite. Different heat treatments were employed to change the flow stress of the matrix, to allow probing of the damage mechanisms and the elongation and fracture toughness of the DRA. Except for the highly overaged condition, the sequence of damage consisted of particle fracture, followed by damage and failure of the matrix ligaments. Elastic-plastic analysis and probability statistics were used to extract the Weibull strength characteristics of the particles. The matrix ligament failure was then modeled using the damage model of Cockcroft and Latham, to derive the failure strain of the composites. The results show reasonable agreement with experimental data, and suggest that at high volume fraction it is critical to retain high particle strength. The fracture toughness of the materials were also analyzed, and issues related to testing and analyses, as well as the inverse dependence of toughness on strength will be discussed.

3:15 PM INVITED PAPER

THE CREEP STRENGTHENING OF SiC PARTICULATES IN

POWDER METALLURGY SiC-Al COMPOSITES: *Farghalli A. Mohamed*¹; ¹University of California, Department of Chemical and Biochemical Engineering and Materials Science, Irvine, CA 92697-2575 USA

A systematic comparison between the creep behavior of discontinuous SiC-Al composites and that of their unreinforced matrix alloys under similar experimental conditions is of practical and scientific significance. From a practical point of view, such a comparison is essential to the process of assessing the potential of these composites for use as structural materials for high temperature applications. From a scientific point of view, information obtained from such a comparison is critical to the identification of various processes that may limit the creep strengthening imparted by SiC particulates or whiskers. In this paper, a comparison between the steady-state creep rates of SiC-2124 Al composites and those of the unreinforced matrix alloy, 2124 Al, over eight orders of magnitude of strain rate reveals that, for constant temperature, strengthening arising from SiC particulates is eliminated at high strain rates. An examination of the microstructure of deformed samples suggests that the loss of strengthening at high strain rates is due to the occurrence of debonding between SiC particulates and 2124 Al (interfacial debonding). This suggestion is supported by the observation that no significant interfacial debonding is detected in a SiC-6061 Al composite which exhibits better creep resistance than 6061 Al over the entire strain rate range measured (no loss of the creep strengthening at high strain rates).

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IMPROVEMENT OF FRACTURE BEHAVIOUR OF SiCP REIN-

FORCED 6XXX AL-MATRIX COMPOSITES BY TAILORING

MATRIX MICROSTRUCTURE: *J. K. Chakravarty*¹; *R. Nagarajan*¹;

*I. Dutta*¹; *T. R. McNelley*¹; ¹Naval Postgraduate School, Center for Materials Science and Engineering, Department of Mechanical Engineering, Monterey, CA 93943 USA

The influence of matrix microstructure on the fracture behaviour of 6092 Al-17.5 vol. % SiC particulate composites has been studied. Both crack initiation and propagation phenomena have been investigated to predict overall fracture behaviour of this composite. It has been successfully demonstrated that with a combination of thermo-mechanical processing and heat treatment, it is possible to modify crack nucleation behaviour and to obtain a desirable strength-fracture toughness combinations. Novel heat treatment methods have

been employed to obtain a ductile layer at the SiC-matrix interface by the introduction of precipitate free zones (PFZs), with the objective of inhibiting propagation of cracks from fractured SiC particles to the surrounding matrix. The impact of this ductile layer on the fracture behaviour of the composites will be discussed. In order to assess the damage tolerance of various microstructural states, evolution of young's modulus, poisson's ration and dilatational strain as a function of strain has been studied. An attempt will be made to rationalize the observed effect of microstructure on fracture behaviour and damage tolerance. * Supported by the US Army Research Office and the Wright Materials Laboratory of the US Air Force

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FATIGUE BEHAVIOR OF PARTICLE REINFORCED METAL

MATRIX COMPOSITES: *Nikhilesh Chawla*¹; *J. Wayne Jones*¹; *John E. Allison*²; ¹University of Michigan, Department of Materials Science and Engineering, 2300 Hayward Dr., Ann Arbor, MI 48109-2136 USA; ²Ford Motor Co., Ford Research Laboratory, Dearborn, MI 48124 USA

SiC particle reinforced Al matrix composites, for example, are being targeted for use as automotive components, where high cycle fatigue resistance (at least 10⁷ cycles) and low cost are the primary considerations. The cyclic fatigue behavior of SiC particle reinforced Al 2080 matrix composites was investigated. The fatigue response of these materials can be described in terms of combinations of classical composite strengthening (direct strengthening) and changes in matrix microstructure and deformation characteristics (indirect effects) which arise from the presence of the reinforcement. By keeping the matrix microstructure constant, a systematic investigation of the effect of reinforcement size and volume fraction on fatigue life was obtained. Matrix microstructure was also varied to examine the effect of precipitate size and distribution on the fatigue behavior of the composites with a given amount of reinforcement. The extent of damage during fatigue in the composite and the unreinforced alloy was characterized by the evolution of stress-strain hysteresis loop shape, plastic strain amplitude, and modulus reduction. Finally, fatigue crack initiation and propagation processes were evaluated and related to matrix microstructure and the presence of reinforcement.

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THE QUASI-STATIC AND CYCLIC FATIGUE FRACTURE BE-

HAVIOR OF A DISCONTINUOUSLY-REINFORCED TITANIUM

ALLOY METAL MATRIX COMPOSITE: *T. S. Srivatsan*¹; *W. O.*

*Soboyejo*²; *R. J. Lederich*³; ¹The University of Akron, Department of

Mechanical Engineering, Akron, OH 44325 USA; ²The Ohio State

University, Department of Materials Science and Engineering, Co-

lumbus, OH 43210 USA; ³Boeing Aerospace Company, St. Louis, OH

USA

A study was undertaken with the primary objective of rationalizing the underlying mechanisms and intrinsic micromechanisms governing the quasi-static fracture, cyclic deformation and cyclic strain-controlled and cyclic stress controlled fatigue fracture of a discontinuously reinforced titanium alloy metal-matrix composite. The discontinuous titanium boride whisker-reinforced Ti-6Al-4V alloy was cyclically deformed under fully-reversed, tension-compression loading over a range of strain amplitudes giving cyclic fatigue lives of less than 104 cycles. The whisker-reinforced titanium alloy matrix was also cyclically deformed under total stress amplitude-control, over a range of stress amplitudes giving fatigue lives in the high-cycle range (> 104 cycles), at both ambient and elevated temperatures. The fundamental kinetics governing the quasi-static and final fatigue fracture processes will be discussed and rationalized in light of the mutually interactive influences of composite microstructural effects, matrix deformation characteristics, cyclic strain and stress amplitudes, composite response stress, and test temperature.

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MECHANICS AND MECHANISMS OF FRACTURE IN TOUGHENED NiAl COMPOSITES: M. Li¹; R. Wang²; D. Zheng²; W. O. Soboyejo¹; ¹The Ohio State University, Department of Materials of Science and Engineering, 2041 College Road, Columbus, OH 43210 USA; ²The Ohio State University, Department of Aerospace Engineering, Applied Mechanics and Aviation, 155 West Avenue, Applied Mechanics Section, Columbus, OH 43210 USA

The mechanics and micromechanisms of fracture in toughened NiAl composites are elucidated in this paper. Following an initial discussion on transformation toughening of NiAl, the effects of layer thickness on the fracture behavior of NiAl composites will be examined within a combined mechanics and materials framework. The synergistic interactions between shielding contributions from crack bridging and transformation toughening are also elucidated. The implications of the results are discussed for the design of toughened NiAl composites.

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STRAIN ACCUMULATION DURING SLIDING WEAR OF SiC WHISKER REINFORCED ALUMINUM COMPOSITES: A. P. Sannino¹; H. J. Rack²; ¹Sengate Technology, Bloomington, MN 55435 USA; ²Clemson University, Clemson, SC 29634-0907 USA

Extensive tribological investigations of discontinuously reinforced metal matrix composites have generally shown that these materials provide significantly improved wear resistance vis a vis their unreinforced matrices. However when delamination is the controlling wear mechanism composite wear performance is similar to and may actually be inferior to the corresponding unreinforced alloy. This presentation will discuss the development and implementation of a coupled finite element - computerized image analysis model for predicting the precursor subsurface strains developed prior to delamination during surface deformation. Comparison between these computational results and experimental examination of global damage accumulation in a SiC whisker reinforced 2124 Al/17-4 PH tribo-couple will also be presented.

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CREEP CRACK GROWTH BEHAVIOR OF BRIDGED CRACKS IN COMPOSITES: S. B. Biner¹; ¹Iowa State University, Ames Laboratory, Ames, IA 50011 USA

In this study, the creep crack growth behavior of bridged cracks that occurs in the composites is investigated numerically. In the analysis, crack growths resulting from the growth of pre-nucleated creep cavities with diffusional and dislocation assisted mechanisms are fully accounted for. The bridging elements are assumed to be elastic. The results indicate that the bridging tractions significantly relax even with the creep deformation of the composite alone. A much weaker stress singularity than the ones described by K or C* occurs ahead of the bridged cracks in the creep regime. In this weak singularity region the cavities grow at comparably high rates to each other.

MODELLING OF TEXTURE AND ANISOTROPIC PROPERTIES IN STEEL AND ALUMINUM INDUSTRIES FOR OPTIMUM MATERIALS PERFORMANCE: Session II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Texture & Anisotropy Committee

Program Organizers: Hasso Weiland, Alcoa, Alcoa, PA 15069 USA; J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

Wednesday PM

Room: Madrid

October 14, 1998

Location: O'Hare Hilton Hotel

Session Chair: J. A. Szpunar, McGill University, Dept of Metallurgical Engrg, Montreal, Quebec H3A 2A7 Canada

2:00 PM INVITED PAPER

NEW PARAMETER MODEL FOR TEXTURE DESCRIPTION IN STEEL SHEETS: L. Delanny¹; P. Van Houtte¹; D. Vanderschueren¹; ¹Katholieke Universteit Leuven, Department MTM, de Croylaan 2, Heverlee B-3001 Belgium

A new model is proposed for the characterisation of steel sheet textures. This model relies on the identification of 25 relevant parameters in the Orientation Distribution Function (ODF). Textures consisting of alpha and gamma fibres and/or cube and Goss orientations can be generated. It was found that the model can quantitatively reproduce almost any industrial steel sheet texture. A first application which was conducted consisted in the study of the r-value sensitivity on the model parameters.

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THE DESIGN OF A BIAXIAL TENSILE TEST AND ITS USE FOR THE VALIDATION OF CRYSTOGRAPHIC YIELD LOCI: E. Hoferlin¹; A. Van Bael¹; P. Van Houtte¹; G. Steyaert²; C. De Mare²; ¹K.U. Leuven, Department MTM, de Croyoann 2, B-3001, Herverlee Belgium; ²OCAS nv, John Kennedylann 3, Zelzate B-9060 Belgium

A biaxial tensile test has been designed for the experimental determination of yield locus points of thin steel sheets. Using texture-based anisotropic finite element (FE) simulations, the geometry of the test sample has been optimised and the accuracy of the conversion procedure has been validated. Experimental tests have been carried out on low carbon steels and both yield stresses and ratios of plastic strains have been compared to the theoretical predictions obtained with the Taylor-Bishop-Hill (TBH) model using the experimentally determined crystallographic textures.

3:00 PM INVITED PAPER

THE INFLUENCES OF DEFORMATION BANDING AND SLIP SYSTEMS ON MODELING BCC ROLLING TEXTURES: Guanglei Liu¹; B. J. Duggan¹; Lixin Zhang²; ¹The University of Hong Kong, Department of Mechanical Engineering, Pokfulam Road, Hong Kong; ²Institute of Corrosion and Protection of Metals, Academia Sinica, 62 WenCui Road, Shenyang 110015 P. R. China

Deformation banding has been established in cold rolled iron by microstructure investigation with a series of SEM micrographs taken from progressively rolled pure iron. Such deformation modes should be included in texture modeling. Of particular significance to simulating bcc rolling texture is the occurrence of deformation banding in the rolling plane since this allows another degree of freedom, ϵ_{32} , in modeling texture evolution with DB model. Simulations including deformation banding and $\{111\} \langle 110 \rangle$ slip show a better agreement with experimental observations. On the other hand, $\{112\}$ slip yields peak type texture and $\{123\}$ slip supports the a-fiber.

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SIMULATION AND EXPERIMENTAL INVESTIGATION OF ROLLING TEXTURES OF FERRITIC STEELS: HOMOGENIZATION METHODS VERSUS Fe METHODS: *D. Raabe*¹; ¹CMU, Pittsburgh, PA USA

The paper focusses on two large-scale approaches to simulate the evolution of rolling textures of polycrystalline metals. The first part of the paper presents predictions obtained by various homogenization approaches, such as Taylor-Bishop-Hill-type and Self-Consistent Modeling. The simulations are conducted for simple plane-strain deformation modes. The texture predictions are conducted for a ferritic stainless steel. The simulations are compared with corresponding experimental data. The second part of the paper presents predictions obtained by the integrated use of viscoplastic finite element simulations in conjunction with Taylor-Bishop-Hill-theory. The simulation results are compared with experimental results for a commercial low-carbon steel.

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RESIDUAL STRESSES IN HY100 POLYCRYSTALS: COMPARISONS OF EXPERIMENTS AND SIMULATIONS: *Paul Dawson*¹; Donald Boyce¹; Ronald Rogge²; Stuart MacEwen³; ¹Cornell University, Sibley School of Mechanics and Aerospace Engineering, 196 Rhodes Hall, Ithaca, NY 14853 USA; ²Steacie Institute for Molecular Science, National Research Council, Ottawa, Ontario K1A 0R9 Canada; ³Alcan International Limited, Kingston Research and Development Center, P.O. Box 8400, Kingston K7L4L9 ONT Canada

Residual stresses arise in polycrystals when they are unloaded after being inelastically deformed. Inelastic deformations are inhomogeneous over the scale of polycrystals due to the anisotropy of individual crystals and to differences in lattice orientations from crystal to crystal. Some crystallographic orientations tend to exhibit greater residual stresses than do other orientations, again a consequence of the single crystal anisotropy both elastic and plastic. Residual stress levels in polycrystals can be determined from neutron diffraction measurements of elastic lattice strains. In this paper we discuss tensile testing of HY100 in which diffraction measurements were made after various levels of plastic strain, both while the specimens were loaded and while they were unloaded. Finite element simulations of the experiments are also presented which are based on an elastoplastic polycrystal constitutive theory. Identical sequences of loading and unloading of specimens have been simulated, allowing the residual elastic lattice strains to be computed at several stages in the process of applying a large overall plastic strain. Comparisons of the residual lattice strains are presented for crystals having particular planes aligned either with the specimen tensile axis or with a transverse axis. The levels of the corresponding residual stresses are discussed relative to aspects of the crystal orientations.

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COMPUTER MODELLING OF GOSS TEXTURE DEVELOPMENT IN Fe-Si STEELS: *N. Rajmohan*¹; J. A. Szpunar¹; Y. Hayakawa²; ¹McGill University, Department of Mining and Metallurgical Engineering, Montreal, Quebec H3A 2B2 Canada; ²Kawasaki Steel Corporation, Technical Research Laboratories, 1-Kawasakidouri, Mizushima, Kurashika, Okayama 712 Japan

Abnormal growth of Goss grain is studied in detail by various computer models which use computer specimens representing the orientation distribution function (ODF) of primary recrystallized Fe-3%Si steel. The computer models take into account the anisotropy of GB energy and mobility. Using various computer experiments conducted, the importance of initial matrix texture, percentage of mobile boundaries around a growing grain, the mobility differences between GBs and the time of release of various grain boundaries during annealing is discussed. Selection mechanism that operates among various Goss nuclei (which are deviated from the real Goss orientation by few degrees) in the initial primary recrystallized matrix, is discussed in detail using the results obtained.

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APPLICATION OF A STOCHASTIC 3D CELLULAR AUTOMATON FOR THE SIMULATION OF RECRYSTALLIZATION TEXTURES IN BCC STEELS: *Dierk Raabe*¹; ¹CMU, Pittsburgh, PA USA

The paper introduces a 3D stochastic cellular automaton for the spatial, kinetic, and crystallographic simulation of mesoscale transformation phenomena that involve non-conserved structural field variables and the motion of sharp interfaces, such as encountered in recrystallization and grain growth. The local transformation rule that acts on each lattice site consists of the probabilistic analogue of a classical linear symmetric rate equation for interface motion. All possible switches are simultaneously considered using a weighted stochastic sampling integration scheme. The automaton is discrete in time, real space, and orientation space. The required input parameter to such simulations (local texture and a local stored energy measure as a function of x,y,z in conjunction with mobility data for the grain boundaries) can be provided by FE simulations or by coupled local texture and microstructure experiments. The method is very simple to use and allows the user to predict texture and microstructure (and thus anisotropy) under consideration of local effects.

PROCESSING & FABRICATION OF ADVANCED MATERIALS VI: Metal-Matrix Composites - II

Sponsored by: Structural Materials Division, Structural Materials Committee

Program Organizers: T. Srivatsan, Univ of Akron, Dept. of Mech. Engr., Akron, OH 44325-3903; K. A. Khor, Nanyang Technological University, Sch of Mech & Prod Engrg, Singapore 639798 Singapore

Wednesday PM

Room: Chicago

October 14, 1998

Location: O'Hare Hilton Hotel

Session Chairs: Dr. R. A. Drew, McGill University, Department of Mining and Metallurgical Engineering, Montreal, Quebec H3A 2B2 Canada; Dr. Z. Xiao Guo, Queen Mary and Westfield College, London University, London United Kingdom

2:00 PM INVITED PAPER

COST-EFFECTIVE MANUFACTURE OF TITANIUM/SILICON CARBIDE (SiC) FIBRE COMPOSITES: *Z. Xiao Guo*¹; ¹Queen Mary and Westfield College, London University, Mile End Road, London United Kingdom

Composites involving continuous SiC fibres and titanium matrices can offer a very desirable combination of weight-specific properties and are ideal for many applications in gas and steam turbine engines. However, large-volume applications are currently hampered by the relatively high costs of the materials. This paper critically examines the characteristics of the manufacturing routes for such composites, including well-studied processes, such as Foil-Fibre-Foil (FFF), Physical Vapour Deposition (PVD), and Vacuum Plasma Spraying (VPS), and new Slurry Powder Metallurgy (SPM) processes currently being developed. For a given manufacturing route, composite properties are enhanced if the material possesses uniform fibre distribution and a low oxygen content and is free from: fibre/matrix interface reactions, residual voids and fibre damage. The capabilities of the above processes in satisfying these requirements are compared and discussed. Possibilities of reducing product costs are analyzed. Several techniques of improving the cost-effectiveness of the composites in relation to manufacture are outlined. Particular emphasis is placed on the study of the SPM routes for Ti/SiC MMCs.

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PROCESSING OF METAL-MATRIX COMPOSITES BY WETTING-ASSISTED INFILTRATION USING METAL-COATED REINFORCEMENTS: Carlos A. Leon¹; Robin A. L. Drew¹; ¹McGill University, Department of Mining and Metallurgical Engineering, 3610 University Street, Montreal, Quebec H3A 2B2 Canada

Metal matrix composites produced via infiltration of a porous preform has been investigated as a viable fabrication process. The combination of properties of SiC/Al and Al₂O₃/Al composites makes of them potentially important as structural materials. Electroless nickel and copper plating techniques were used to enhance the wetting of the solid by the liquid phase in order to promote infiltration. Initially the work focused on establishing the best processing route to achieve effective metal-deposition on the ceramic powders. The quantity and size of deposited material, and the uniformity and nature of the coating upon deposition of Cu and Ni on Al₂O₃ and SiC particles were studied. Microscopy and chemical analysis revealed a continuous and uniform nickel deposition on reinforcements with traces of phosphorus resulting from the electroless solution. Surface morphology of copper-plated substrates consisted of fine adhering particles forming thinner metal coatings. Furthermore, the microstructure, coating distribution and infiltration behaviour of the composites are also being investigated.

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PHASE TRANSITIONS IN THE FORMATION OF Ti₅Si₃/TiAl IN SITU COMPOSITES AND THEIR MULTIPLEX STRUCTURES: Yijun Du¹; K. P. Rao¹; X. D. Han²; K. C. Law²; J. C. Y. Chung²; ¹City University of Hong Kong, Department of Manufacturing Engineering, Tat Chee Avenue, Kowloon, Hong Kong; ²City University of Hong Kong, Department of Applied Physics and Materials Science, Tat Chee Avenue, Kowloon, Hong Kong

A mixture of elemental Ti, Al and Si powders with a stoichiometry of Ti₁₄Al₂₁Si has been ball-milled for producing metastable phases. Differential thermal analysis (DTA) and x-ray diffraction (XRD) have been used to identify the phase reactions and transitions occurring in the metastable phases during heating. It has been found that Ti₅Si₃ is in situ formed through a phase transition chain, i.e., from TiSi₂ to Ti₅Si₄ and finally to Ti₅Si₃. Also, it has been found that formation of Ti₃Al (or TiAl, or TiAl₃, depending on the milling time) competes against TiSi₂ in the metastable phases during heating. In practice, the metastable phases were incorporated into Ti-Al powder matrix, and a final Ti₅Si₃/TiAl composite was obtained when the constituent powders underwent a reactive sintering (RS) process followed by hot isostatic pressing (HIP). Transmission electron microscope (TEM) was employed for revealing the phase structure and distribution of various phases in the composite. A hierarchical structure including ductility-favored phases such as alpha-2 (Ti₃Al) has been observed between the Ti₅Si₃ reinforcement and TiAl matrix. It is thus possible that both room-temperature ductility and high-temperature strength can be enhanced under such a multiplex microstructure. In addition, elemental Si powders of two different sizes (71 μm and 50 nm) were tried to study the respective structure evolutions of the resultant composites.

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THE INFLUENCE OF FABRICATION MODE ON THE PROPERTIES OF HEIGHT DEFORMED Cu-Nb/Ti COMPOSITE WIRES: L. Brandao¹; Peter N. Kalu¹; ¹FAMU-FSU College of Engineering, National High Magnetic Field Laboratory, Department of Mechanical Engineering, Tallahassee, FL 32310 USA

The principal requirements for conductor wires used in high-field pulsed magnets are high mechanical strength and high electrical conductivity. In order not to decrease the conductivity, a combination of fiber strengthening plus work hardening was used to strengthen Cu-Nb/Ti material. Heavy deformation was used to produce the required level of work hardening. Three fabrication methods, swaging, drawing and swaging plus drawing were employed in producing 2.03 mm diameter Cu-Nb/Ti wires. Here, the effect of the mode of fabrication

on the properties and texture development in Cu-Nb/Ti composite is reported.

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DEVELOPMENT OF HIGH STRENGTH HIGH CONDUCTIVE COPPER-BASE IN SITU COMPOSITE: H. G. Suzuki¹; K. Mihara¹; K. Adachi¹; Y. Jia¹; T. Takeuchi¹; ¹National Research Institute for Metals, 1-2-1 Sengen, Tsukuba 305 Japan

Cu-15% Cr-C,-Zr in situ composites which have the strength level higher than 1100MPa and electrical conductivity exceeding 70%IACS, have been developed by optimizing the alloy compositions as well as thermomechanical process. Cold drawing is effective to refine lamellar spacing of second phase (Cr fiber) and aging treatment just after solutioning gives precipitation strengthening and recovery of electrical conductivity. The optimization of each process was conducted. The tensile strength follows Hall-Petch type equation with Cr lamellar spacing. The additions of C refines the solidification structure, while Zr increases the precipitation hardening. We will discuss the method for process optimization and the mechanism of strengthening of in situ composite.

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TRIBOLOGICAL PROPERTIES OF SELF-LUBRICATIVE Cu COMPOSITES WITH CLUSTER DIAMOND: Kotaro Hanada¹; ¹Mechanical Engineering Laboratory, Plasticity & Forming Division, Namikil-2, Tsukuba, Ibaraki Japan

Cluster diamond (CD) produced by a detonation method has attracted special interests in various fields because it has unique characterization such as (1) ultra-fine particulate (5nm in average), (2) single crystal, (3) almost a spherical shape, (4) excellent lubrication, (5) excellent abrasion. In this work, CD dispersed self-lubricative Cu composites are fabricated by powder metallurgy method, and the tribological properties are investigated. Pure Cu powder and CD powder are mechanically mixed at 500rpm for 40 hours under Ar atmosphere, and the mixed composite powders are hot pressed at 723°K in vacuum. X-ray analysis shows that CD is not changed to graphite in the compacted Cu/CD composite powders. The coefficient of friction and the linear wear of Cu/1vol%CD composite are superior to pure Cu under all testing condition in this work, and those improvements are 36% and 78% in maximum, respectively. However, as the volume fraction of CD is increased, the diffusion bonding between Cu matrix powders becomes weak because of the agglomeration of CD at the prior particle boundaries of Cu/CD powders, and it results in poor friction properties.

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SIMULATION OF METAL MATRIX COMPOSITE INFILTRATION PROCESSING: Th. Dopler¹; A. Modaresi¹; V. Michaud²; ¹Laboratoire de Mecanique des Sols, Structures et Materialux, CNRS URA 850, Ecole Centrale Paris, Chitenay-Malabry France; ²Laboratoire de Technologie des Composites et Polymers, Department des Materiaux, E-cole Polytechnique Federale de Lausanne Switzerland

Infiltration processing constitutes a simple method to produce metal matrix composites, whereby a liquid metal penetrates into a porous preform of the reinforcing phase. A 3-D finite element software is currently being developed to simulate this process, based on the similarity with drainage phenomena in soil mechanics. Capillarity is taken into account for isothermal infiltration under any increasing applied pressure function. Simulation of preform deformation is also possible. Saffil alumina preforms were infiltrated by molten aluminum. Comparison of simulation results with experimental data shows good agreement, in terms of infiltration kinetics and porosity distribution.

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EFFECT OF FLYASH PARTICLES ON THE WEAR BEHAVIOUR OF ZA-27 ALLOY COMPOSITES: S. C. Sharma¹; B. M. Girish¹; R. Kamath¹; B. M. Satish¹; ¹R. V. College of Engineering, Department

of Mechanical Engineering, Division of Research and Development, Bangalore, Karnataka 560059 India

In the present study, the effect of zircon reinforcement on the unlubricated sliding wear behaviour of ZA-27 alloy composites was investigated using a pin-on-disc sliding wear testing machine. The composite specimens were prepared using the liquid metallurgy technique and the percentage of zircon was varied from 1-5% in steps of 2% by weight. The sliding wear tests were conducted at loads of 3, 4, 5 and 6 Kg (29.4, 39.2, 49.1 and 58.9 N respectively) and rotational speeds of 200, 250 and 300 rpm (i.e., tangential speeds of 1.25, 1.56, 1.87 m/s respectively). The unreinforced alloy was also tested under identical conditions in order to enable comparison. The results of the tests revealed that the zircon reinforced composites exhibited a lower wear rate compared to the unreinforced alloy specimens. Increase in the applied load increased the wear severity by changing the wear mechanism from abrasion to particle cracking induced delamination wear. It was found that with the increase in zircon content, the wear resistance increased monotonically with hardness. SEM micrographs indicated the effect of zircon particles in reducing the wear rate of the composite material.

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SHORT GLASS FIBRES REINFORCED ZA-27 ALLOY COMPOSITES FOR JOURNAL BEARING APPLICATIONS: S. C. Sharma¹; B. M. Girish¹; R. Kamath¹; B. M. Satish¹; ¹R. V. College of Engineering, Division of Research and Development, Department of Mechanical Engineering, Bangalore, Karnataka 560059 India

The bearing performance of cast ZA-27 alloy composite materials reinforced with short glass fibres have been investigated under lubricated, semi-dry and dry conditions. The composite materials were fabricated employing the compocasting technique. The journal bearing in their as-cast conditions were tested using a computer interfaced bearing test rig. The test bearings press against the shaft (EN24) under a radial vertical loading. It followed from the results obtained that the composite bearings exhibited lower friction in comparison with the unreinforced alloy bearings. The composites as well as the alloy bearings tested under lubrication exhibited least coefficient of friction in comparison with those tested under semi-dry and dry tests. In the lubricated tests, the composite bearings were able to run without seizure up to the regimes of boundary lubrication with less friction. In the semi-dry and the dry tests, the composite bearings exhibited lower friction which further reduced with the increase in the fibres content but increased with the increase in load. Under the semi-dry and dry conditions, the unreinforced alloy bearings seized at much lower loads than the composite bearings.

PROCESSING & PROPERTIES OF ADVANCED STRUCTURAL CERAMICS: Advanced Structural Ceramics: Properties

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Materials Design and Manufacturing Division, Structural Materials Division,

Program Organizers: Rajiv S. Mishra, University of California, Dept of Chemical Engineering & Materials Science; Amiya K. Mukherjee, University of California, Dept of Chemical Engineering & Materials Science

Wednesday PM
October 14, 1998

Room: London
Location: O'Hare Hilton Hotel

Session Chair: Katherine T. Faber, Northwestern University, Materials Science and Engineering, Evanston, IL 60208 USA

2:00 PM KEYNOTE

A SYSTEMS APPROACH TO THE DESIGN OF STRUCTURAL CERAMICS: Rishi Raj¹; Ganesh Subbarayan¹; ¹University of Colorado, Department of Mechanical Engineering, Boulder, CO 80309-0427 USA

A systems approach in the analysis and design of structural ceramics is used to identify the critical parameters in the development of a ceramic for specific applications. In this instance we shall consider ceramic coatings on metallic turbine blades. The presently used zirconia based thermal barrier coatings will be compared with other candidate materials. A rational process for identifying the key issues in each instance will be articulated. Environmental issues and the mechanical chemical properties of the ceramic-metal interface merges as one of the critical areas from the point of view of failure prediction. However, the total performance of the coating also depends on the thermal diffusivity and on the surface roughness of the coating.

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THERMAL AND MECHANICAL PROPERTIES OF NANOSTRUCTURED ZIRCONIA THIN FILMS*: J. A. Eastman¹; G. Soyoz¹; R. J. DiMelfi²; L. J. Thompson¹; ¹Materials Science Division, Argonne National Laboratory, Argonne, IL 60439 USA; ²Reactor Engineering Division, Argonne National Laboratory, Argonne, IL 60439 USA

Stabilized zirconia thermal barrier coatings are used in a number of important applications, such as in protecting gas turbine components in high temperature environments. Improved thermal barrier coatings are required to attain desired higher, and thus more efficient, operating temperatures. Improvements in service lifetimes of components are also desirable and require the development of coatings with improved damage tolerances both at ambient and elevated temperatures. Nanocrystalline coatings are expected to exhibit reduced thermal conductivities compared to coarse-grained coatings because of a reduction in the mean free path of phonons due to presence of grain boundaries. This behavior, combined with expected improved mechanical properties, makes nanostructured zirconia coatings excellent candidates for future applications as thermal barriers. Experiments are underway to produce yttria-stabilized zirconia thin films by metal-organic chemical vapor deposition techniques and to correlate thermal and mechanical properties with film microstructure. *This work is supported by the U. S. Department of Energy, BES-DMS, under Contract W-31-109-Eng-38 and by a grant from Argonne's Coordinating Council for Science and Technology

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MECHANICAL PROPERTIES OF SOME OXIDE SINGLE CRYSTALS AT HIGH TEMPERATURES: T. E. Mitchell¹; ¹Los Alamos National Laboratory, Center for Materials Science, MS-K765, Los Alamos, NM 87545 USA

The mechanical properties of sapphire and spinel at high temperatures are described and compared with other oxides. The critical resolved shear stress is found to decrease logarithmically with increasing temperature for both basal and prism plane slip in sapphire and for {110} and {111} slip in spinel. The CRSS of magnesium aluminate spinel ($MgO \cdot nAl_2O_3$) decreases by almost orders of magnitude as n increases from 1 (stoichiometric spinel) to 3.5; at the same time the minimum temperature for compression ductility decreases from $\sim 1800^\circ C$ to $\sim 1300^\circ C$. Deviations from stoichiometry are compensated by the introduction of cation vacancies with a concentration given by $[V_c] = (n-1)/3(3n+1)$. The solution softening effect resulting from increasing values of n and $[V_c]$ is found to be such that the CRSS is proportional to $[V_c]^2$. The temperature dependence and the concentration dependence suggest that the CRSS is controlled by a Peierls stress which is decreased by kink nucleation at cation vacancies. In addition, hydrolytic weakening is observed in sapphire which also may be due to double kink nucleation at point defects, possibly hydrogen. These effects are discussed in terms of their crystal structures.

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THE EFFECT OF HIGH FREQUENCY FATIGUE ON THE INTERFACIAL PROPERTIES OF NICALON FIBER REINFORCED SICON CERAMIC MATRIX COMPOSITES: *Nikhilesh Chawla*¹; Jeffrey Eldridge²; ¹University of Michigan, Department of Materials Science and Engineering, 2300 Hayward Dr., Ann Arbor, MI 48109-2136 USA; ²NASA Lewis Research Center, Cleveland, OH 44135 USA

In a variety of applications, continuous fiber reinforced ceramic matrix composites (CFCMCs) are likely to encounter high frequency fatigue loading. Most CFCMCs have been tailored with relatively weak fiber/matrix interfaces that are conducive to crack branching, crack deflection, and fiber pullout, that substantially enhance the toughness of these materials. During high frequency fatigue, however, it has been shown that wear of the weak interface takes place, contributing to premature failure in these materials. In this work, we have studied a woven Nicalon fiber reinforced SiCON matrix composite, processed by polymer infiltration and pyrolysis (PIP), with a relatively strong fiber/matrix interface to increase the fatigue resistance of the composite. The composite was fatigued at a loading frequency of 100 Hz. The evolution of interfacial shear stresses was measured using fiber pushout technique and related to the macroscopic fatigue behavior of the composite. Finally, modulus reduction, stress-strain hysteresis, and temperature rise in the composites were measured with increasing cycles to provide an estimate of evolution of damage in the composite.

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TOUGHENING IN CERAMIC LAMINATES: *K. T. Faber*¹; D. M. Baskin¹; E. R. Fuller²; ¹Northwestern University, Department of Materials Science and Engineering, 2225 North Campus Drive, Evanston, IL 60208 USA; ²National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

In both laminate and fiber-reinforced ceramics, the interface toughness relative to the toughness of the layer or fiber dominates the mechanical behavior and determines whether crack deflection or penetration occurs. We explore, here, laminates of textured and non-textured iron titanate, a highly anisotropic ceramic, in which various combinations of elastic and thermal mismatch can be produced using a single material. Layers as thin as 0.6 mm are made via gel-casting and sintered together to form laminates. Mechanical properties are evaluated using conventional fracture mechanics techniques. Laminate properties are compared to those simulated from microstructures using an object oriented finite element code (OOF). Design concepts for ceramic laminates are confirmed.

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CERAMIC NANOCOMPOSITES: MECHANISMS FOR STRENGTH ENHANCEMENT: *B. Derby*¹; C. W. Lawrence¹; S. G. Roberts¹; H. Z. Wu¹; ¹University of Oxford, Department of Materials, Parks Road, Oxford OX1 3PH UK

Ceramic nanocomposites consisting of oxide matrices reinforced with submicron intragranular dispersions show an enhanced strength when compared to unreinforced matrix material of the same grain size. This increase in strength cannot be explained by an increase in toughness of these materials. From our understanding of the mechanisms of brittle fracture, this strength increase must be associated with a decrease in the maximum (Griffiths) flaw present in the test pieces. An analysis of surface flaw densities using Hertzian indentation, acoustic measurements and microstructural examination will be presented to demonstrate a lower surface defect density in these materials. This is also evident in a greater resistance to erosive wear shown by these materials. Possible mechanisms for this reduction in surface damage will be discussed.

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ELECTRIC FIELD ENHANCED PLASTIC DEFORMATION KINETICS OF FINE-GRAINED Al_2O_3 : James Campbell¹; *Hans Conrad*¹; Yusef Fahmy¹; ¹North Carolina State University, Materials Science and Engineering Dept., Raleigh, NC 27695-7907 USA

The influence of a contacting, axial electric field ($E=1-1.5$ kV/cm) on the plastic deformation kinetics in tension of fine-grained ($d_0=1.5$ mm) Al_2O_3 was determined at 1450°-1600°C by constant strain rate and stress relaxation tests. The electric current (6-16 mA) and temperature rise (10°-40°C) due to Joule heating were measured. It was found that application of the electric field during the course of a test reduced the flow stress by ~80%, of which approximately one-half was due to Joule heating and the remainder a direct effect of the field and/or current. The electric field had no detectable effect on the stress exponent $n = \ln(\text{strain rate}) / \ln \sigma$, which had a value of ~2 in all tests. The parameters and mechanism by which the electric field and/or current affected the plastic deformation kinetics are discussed.

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MECHANICAL PROPERTIES OF DENSE Al_2O_3 -ZrO₂ NANOCOMPOSITES: *S. Bhaduri*¹; D. F. Bahr²; ¹University of Idaho, Metallurgical and Mining Eng., Moscow, ID 83844-3024 USA; ²Washington State University, Pullman, WA 99164-2920 USA

Nanocrystalline Al_2O_3 -ZrO₂ powders were synthesized using a novel 'Auto Ignition' synthesis process. The respective nitrates and urea were used as precursors. The powders were hot isostatically pressed at 1200C for 2 hours at 175 MPa. Initial mechanical properties were determined by an indentation technique using a Vickers indenter. The results indicated that the hardness value was low and toughness was in the range for toughened ceramics. In order to check if phase transformation of ZrO₂ or ductility was involved, the samples were further tested using a nanoindenter in conjunction with an AFM. Load displacement curves did not indicate any phase transformation in these materials. Considerable pile up of materials was observed around the indentation. This is an evidence of ductility in these materials.

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