# SYMPOSIUM V

## Size Effects in Plasticity

November 29 - December 2, 2004

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SESSION V1: Small Scale Plasticity, Strain Gradient Theory/Expt
Chairs: William Nix and Hussein Zbib
Monday Morning, November 29, 2004
Room 206 (Hynes)

**8:30 AM *V1.1**
**Deformation at the Nanometer and Micrometer Length Scales: Dislocation Starved Plasticity.** William D. Nix, Gang Feng and Julia R. Greer; Materials Science and Engr., Stanford University, Stanford, California.

Size effects in plasticity are now well known. Plastic deformation in small volumes requires higher stresses than are needed for plastic flow of bulk materials. Here we review the various effects that appear to be responsible for this, focusing mainly on the role of dislocation starvation. The size dependence of the hardness of metals can be described in terms of the geometrically necessary dislocations or, correspondingly, the strain gradients, created in small indentations. But such accounts break down when the size of the deformation volume begins to approach the spacing of individual dislocations or when the crystal becomes dislocation starved. Nanoindentation of epitaxial films at the nanometer depth scale reveals irregular load-displacement curves. In this domain the nucleation of dislocations and plasticity under dislocation-starved conditions appears to be more important than strain gradients. Recent uniaxial compression experiments on tiny samples of gold made by focused ion beam machining and integrated circuit fabrication methods show strong size effects on plasticity, with sub-micron sized crystals showing remarkable strengths after plastic deformation. These experiments involve small deformation volumes and minimal strain gradients. These size effects may be explained by considering a process of strain hardening by dislocation starvation, wherein existing dislocations leave the crystal more frequently than they reproduce themselves by multiplication. Efforts to model this behavior using the breeding factor for dislocation multiplication as it applies to small crystals will be described.

**9:00 AM V1.2**
**Micron-Scale Plasticity of Gold in the Absence of Strain Gradients.** Julia Rosolovsky Greer and William D. Nix; Materials Science and Engineering, Stanford University, Stanford, California.

The classical laws of materials science dictate that the mechanical behavior is independent of the sample size, however the results of recent experiments in nanoindentation and of the molecular dynamics simulations display a strong size effect at the micron and below scales. In nanoindentation experiments, the non-uniformity of stresses and strains within the test sample is responsible for the so-called indentation size effect (ISE). ISEs have been widely observed in bulk materials and in thin films and manifest themselves as the apparent increase in hardness at shallower indentation depths. The observed ISE is explained in part by the strain gradient plasticity theory developed by Nix and Gao, which demonstrates a linear dependence between the square of the indentation hardness and the inverse of the indentation depth for single crystals. The model utilizes Taylors relation between the flow stress and the dislocation density and makes use of the concept of the evolving density of the so-called geometrically necessary dislocations. While the Nix-Gao model explains the depth-dependent hardness variation in single crystals well for indentation depths above 100nm, it cannot predict the observed discrete strain bursts characteristic of the elastic-to-plastic transition observed when infinitesimal volumes are tested. The discrete displacements found in single crystalline materials during the initial stages of nanoindentation are attributed to the nucleation of dislocations, the primary mechanism of plasticity on the nanometer scale. Recently, atomistic behavior during mechanical deformation was studied via MD simulations, which indicated that plastic deformation was intrinsically inhomogeneous, that the yield strength depended on the sample size even in the absence of a strain gradient, and that for small single crystals, the yield strength scaled with the volume-to-surface area ratio of the sample. Results of an experiment testing mechanical properties of gold at the micron scale without the associated strain gradients are presented in this paper. The test methodology consists of two unique fabrication processes of single-crystalline and polycrystalline free-standing gold cylinders of sub-micron dimensions and the subsequent analysis of their responses to uniaxial mechanical deformation. Specimens of varying aspect ratios fabricated by both techniques are compressed via MTS Nanoindenter with a custom-machined flat punch tip. Stress, strain, and instantaneous stiffness of the pillars are reported here. Test results indicate a significant increase in flow stress up to several GPa, reaching the order of theoretical shear strength of the material as the pillars are compressed. Dislocation behavior is modeled and observed via TEM analysis performed on the non-deformed as well as on the compressed pillars. No apparent correlation between flow stress and volume-to-surface area of the specimen was detected.

**9:15 AM V1.3**
**Measurement of Geometrically Necessary Dislocation Density Based Upon Lattice Rotation Measurements in FCC Single Crystals.** Jeffrey William Kysar and Yong X. Gan; Mechanical Engineering, Columbia University, New York, New York.

Strain gradient plasticity theories often invoke the density of geometrically necessary dislocations on individual slip systems of a plastically deforming crystal. In this talk, a technique of measuring the density of geometrically necessary dislocations based upon lattice rotation measurements is discussed. A specially oriented single crystal of a face-centered cubic material is plastically deformed such that it experiences plane strain deformation. The as-deformed orientation of the crystalline lattice is measured under plane strain conditions via the electron backscatter diffraction technique (EBSD). Subsequently the gradient of the experimentally determined lattice rotation is determined numerically in order to calculate all non-zero components of the lattice curvature tensor as well as the Nye dislocation density tensor. Then, minimization schemes are employed to determine the density of geometrically necessary dislocations on each slip system of the crystal. The technique is applied to indentations in single crystals and the measurements are made with a submicron spatial resolution.

**9:30 AM V1.4**
**What Size Effect?** John Joseph Gilman, MSE, UCLA, Los Angeles, California.

That the sizes of solids affect their behavior is unquestionable. It results from the inseparability of size and surface area, plus the Heisenberg Principle which limits the magnitudes of gradients. These factors generate a great variety of surface effects. Of late, however, the idea that size effects arise from strain gradients in continuous matter has been resurrected. This metaphysical idea has long ago been shown to be inconsistent with both experimental facts, and with established physical theory, including the fact that matter is discontinuous. These inconsistencies are the subject of this paper. The principle faults of the strain gradient theory are that it is unnecessary to understand known size effects; and that it does not lead to sensible results. It is not necessary because there are several well-known sources of scale effects in the mechanics of solids. These include: grain boundaries; domain sizes, twins; crack lengths; Debye lengths; electron cyclotron radii; electron and phonon mean-free-paths; electron wavelengths; plasmon wavelengths; surface relaxation lengths; skin depths; adsorped films; surface reaction products; Casimir distances; and more. It's a quantized world. The validities of many theories can be tested by considering their limits. The expression for size effects given by the strain gradient theory becomes infinite at small distances; thereby establishing its lack of validity. Also, the theory lacks generality. Consider a direction normal to an interface between two dissimilar solids. A continuous strain gradient cannot even be defined at the interface! However, the interfacial structure can be described in considerable detail using quantum electrodynamics.

**9:45 AM V1.5**
**X-ray Diffraction Investigation of Strain Gradient Plasticity Theories.** M. M. Barney[1,2], G. H. Campbell[1], J. S. Stolken[1] and R. O. Ritchie[2]; [1]Chemistry and Materials Science, Lawrence Livermore National Laboratory, Livermore, California; [2]Materials Science and Engineering, University of California, Berkeley, Berkeley, California.

Non-uniform plastic deformation occurring in small volumes results in large gradients in strain and has been shown to affect the mechanical behavior in metals. Because conventional continuum plasticity theories are size invariant, and therefore cannot capture this effect, different types of strain gradient plasticity theories have been developed by including a strain gradient term in the governing field equations. While there has been considerable work done on developing various models based on this theory, no experiments have been done to investigate which type of modeling approach describes the microscopic behavior accurately. The present study proposes to address this issue by conducting a set of deformation experiments and directly measuring the material response on the micron scale. Specifically, sapphire/aluminum/sapphire layered sandwich beams are deformed in simple shear using the asymmetric four-point bend test configuration. After deformation, X-ray micro-diffraction experiments using synchrotron radiation are conducted to measure lattice rotations in the deformed aluminum layer, which can be related to the strain. Because simpler strain gradient plasticity models predict uniform strain near the interface, and more complex models allow for the presence of a strain gradient very close to the boundary (called a boundary layer), this experiment will be able to determine which type of model best describes the true material behavior. Using the procedure described above, evidence of a boundary layer has been found, suggesting that the more complex stain gradient plasticity models are needed. This work performed under the auspices of the U.S. Department of Energy by University of California, Lawrence

Livermore National Laboratory under Contract W-7405-Eng-48. UCRL-ABS-204674

**10:30 AM *V1.6**
**Size Effects in Discrete Dislocation Plasticity.** Alan Needleman, Brown University, Providence, Rhode Island.

Plastic flow in crystalline materials is size dependent over length scales of the order of tens of microns and smaller. This size dependence arises in a variety of contexts; e.g. micro- and nano- indentation and the thermo-mechanical response of thin films. One well-appreciated origin of size effects is associated with imposed plastic strain gradients and geometrically necessary dislocations. In addition, strain gradients and boundary layers leading to size-dependent response can occur in circumstances where, at least in principle, a more or less homogeneous response is possible but where the physics of dislocation motion prevents it. Furthermore, in small volumes plastic flow can be source limited. Discrete dislocation plasticity analyses of various plastic flow processes will be used to illustrate a range of size effects and the scaling that is predicted by the calculations. Implications for deformation and fracture will be discussed.

**11:00 AM V1.7**
**Mechanical Property Measurements of a Near Alpha Titanium Alloy at the Micro-Size Scale.** Dave Norfleet[1], Michael Mills[1], Jim Williams[1] and Michael Uchic[2]; [1]The Ohio State University, Columbus, Ohio; [2]Air Force Research Laboratory, Dayton, Ohio.

The constant strain rate deformation behavior of individual $\alpha$-$\beta$ colonies of the titanium aeroengine alloy Ti-6Al-2Sn-4Zr-2Mo-0.1Si (composition in wt.%) has been studied through an ultra small-scale compression technique. Using an FEI Dual Beam Focused Ion Beam (DB-FIB), cylindrical compression samples were micromachined into six single colony crystal orientations. Each of the samples were oriented for single slip in the HCP alpha phase along one of the three distinct a-type slip systems on both basal and prism planes. The compression samples having diameters ranging from 10 to 35 microns, were mechanically tested using an MTS Nano Indenter XP fitted with a flat tip to apply uniaxial compression at a constant strain rate. The effect of sample size on the flow properties will be discussed. The resulting data will be correlated with macrosample measurements, as well as, tensile results for similar orientations. The deformation mechanisms will be discussed in light of TEM studies on samples extracted from the micro-compression pillars using the DB-FIB.

**11:15 AM *V1.8**
**Elements of a Micromechanical Theory of Constrained Crystal Plasticity: Energy, Configurational Forces and Characteristic Lengths.** Sinisa Dj Mesarovic, School of Mechanical and Materials Engineering, Washington State University, Pullman, Washington.

To formulate a continuum theory of crystal plasticity we first define the portion of work on plastic deformation of crystals associated with the existence of geometrically necessary (GN) dislocations, on the basis of the mechanics of dislocations. Then, we express this microstructural work in terms of the continuum representation of GN dislocations - the Nye's dislocation density tensor, and thus establish a firm connection between the mechanics of dislocations and a continuum theory. With this connection in place, the nature of the resulting characteristic lengths becomes apparent. Analysis of elementary energy sinks in dislocation mechanics leads to the conclusion that (a) the interaction energies amongst GN dislocation segments, and, (b) the interaction energies between the GN segments and the boundaries, must be identified with the microstructural work. After establishing a connection between the discrete and the continuum descriptions of the state of dislocations in a solid, the interaction energies - both, amongst the GN segments and between the GN segments and the boundaries - are expressed as functions of Nye's dislocation densities. Configurational forces are defined as work-conjugates to Nye's densities. The resulting microstructural work density and the microstructural constitutive relations are nonlocal. The microstructural constitutive law takes the form of a convolution integral over the domains with non-vanishing Nye's densities. This is a direct consequence of the long-range interactions in dislocation mechanics. Continuum characteristic lengths are intimately connected with the nonlocal nature of the microstructural work density. They represent the dimensions of domains of integration in the convolution integrals associated with the nonlocal configurational forces. No characteristic length can be described as a material length. Dislocation mechanics lengths are absorbed into continuum fields. The continuum lengths are identified as problem-dependent (dimensions of obstacles to dislocation motion) and solution-dependent (the widths of boundary layers associated with each slip system).

**11:45 AM V1.9**
**On Boundary Conditions and Plastic Strain-Gradient Discontinuity in Lower-Order Gradient Plasticity.** Amit Acharya[2], Huang Tang[3], Sunil Saigal[2] and John L. Bassani[1]; [1]Mechanical Engineering and Applied Mechanics, University of Pennsylvania, Philadelphia, Pennsylvania; [2]Civil and Environmental Engineering, Carnegie Mellon University, Pittsburgh, Pennsylvania; [3]Mechanical Engineering, MIT, Cambridge, Massachusetts.

Through linearized analysis and computation, we show that lower-order gradient plasticity is compatible with boundary conditions, thus expanding its predictive capability. A physically motivated gradient modification of the conventional Voce hardening law is shown to lead to a convective stabilizing effect in 1-d, rate-independent plasticity. The partial differential equation is genuinely nonlinear and does not arise as a conservation law, thus making the task of inferring plausible boundary conditions a delicate matter. Implications of wave-type behavior in rate-independent plastic response (under conditions of static equilibrium) are analyzed with a discussion of an appropriate numerical algorithm. Example problems are solved numerically, showing the robustness and simplicity of physically-motivated lower-order gradient plasticity. The 3-d case and rate-dependent constitutive assumptions are also discussed.

SESSION V2: Multiscale Phenomena and Modeling
Chairs: Peter Anderson and William Gerberich
Monday Afternoon, November 29, 2004
Room 206 (Hynes)

**1:30 PM *V2.1**
**Thin Film Delamination: A Discrete Dislocation Model.** William A. Curtin and Michael P. O'Day; Division of Engineering, Brown University, Providence, Rhode Island.

The onset of interface delamination during the indentation of thin ceramic films on metal substrates is studied within the discrete dislocation (DD) plasticity framework. In this method, plasticity in the metal substrate occurs directly via the motion of dislocations embedded in a linearly elastic medium. A set of physically-based constitutive rules governs the nucleation, motion and annihilation of dislocations; the long range interactions occur through their singular elastic fields. A cohesive law characterizes the traction-separation response of the interface; no initial interface imperfections or damage exist in the unloaded configuration. The indenter is a rigid, flat punch and plane strain deformation is assumed. Shear delamination occurs during deep indentation loading of relatively weak interfaces while tensile cracking underneath the indenter is found for considerably stronger interfaces and at lower maximum indentation depths, in qualitative agreement with continuum plasticity. However, continuum plasticity analyses only predict separation for interfaces with strengths equal to, or less than, the material yield strength, while the DD simulations predict crack nucleation for interface strengths up to three times the yield strength. This is due to the large interface stresses that arise from nearby dislocations and pileups which are not captured in standard continuum plasticity models. Such tensile crack nucleation is evident as a kink on the force-indentation depth curve. With increasing interface strength the kink becomes rather sharp, denoting the unstable snapping-open of the crack surfaces.

**2:00 PM V2.2**
**Bridging from Ab Initio Atomistic Simulations to Finite Element Modeling.** Lyle E. Levine, F. Tavazza, R. J. Wagner, L. Ma, Shafique M. A. Khan and A. M. Chaka; National Institute of Standards and Technology, Gaithersburg, Maryland.

Ab initio (density functional theory) simulations can only handle a few hundred atoms using today's computers. Conversely, finite element modeling (FEM) is typically used to model the mechanical behavior of macroscopic structures. We describe a methodology we have developed for bridging this gulf for quasi-static applications. At the small scale, we use a direct coupling of ab initio simulations with the embedded atom method (EAM). This has the advantage of allowing us to conduct large-scale atomistic simulations with simple classical potentials while reserving the more accurate ab initio approaches for critical regions. The boundary conditions and initial atom positions for the EAM simulations come from either small-scale finite element models or from 3D dislocation dynamics simulations. The information flow goes both directions between the macroscopic and atomic size scales.

**2:15 PM V2.3**
**Simulation Studies of Ideally Ductile Cracks: the Role of Temperature and Size Effects.** Robin L. B. Selinger and Matthew A. Davidson; Physics Dept., Catholic Univ., Washington, District of Columbia.

Temperature is an important factor controlling the dislocation nucleation rate at a crack tip under stress, but its role is not completely understood. To examine temperature effects, we perform computer simulations of an ideally ductile straight crack deformed in mode III in two dimensions. The simulation model is essentially molecular dynamics with a highly idealized potential and with only antiplane displacements allowed, and is an analog of the XY model from statistical physics. We apply an external shear load to a crack and measure the resulting strain rate as a function of temperature; the strain rate is directly proportional to the rate at which screw dislocations nucleate at the crack tip and annihilate on the opposite free surface. At low temperature we observe Arrhenius behavior: the log of the measured strain rate is linear in 1/T, indicating that the rate of dislocation production is controlled by thermal activation. However at higher temperature we observe a crossover to nonlinear super-Arrhenius behavior, with a dislocation nucleation rate well in excess of that expected from thermal activation alone. We argue that this finding is direct evidence of the cooperative instability in dislocation nucleation first proposed by Khantha et al [Phys. Rev. Lett. 73, 684 (1994).] We also examine the mechanical response of an ideally ductile crack under a constant applied shear velocity and measure the resulting stress-strain behavior as a function of crack size, at fixed temperature. In each case we scale both the crack length and simulation cell size by the same factor. We observe size effects both in the stress/strain at first dislocation nucleation and in the equilibrium flow stress.

**2:30 PM *V2.4**
**Recent Advances in Multiscale Modeling of Materials Behavior.** Hussein M. Zbib, School of Mechanical and Materials Engineering, Washington State University, Pullman, Washington.

Deformation and strength of crystalline materials are determined to a large extent by underlying mechanisms involving various crystal defects, such as vacancies, interstitials and impurity atoms (point defects), dislocations (line defects), grain boundaries, heterogeneous interfaces and microcracks (planar defects), chemically heterogeneous precipitates, twins and other strain-inducing phase transformations (volume defects). Most often, dislocations define plastic yield and flow behavior, either as the dominant plasticity carriers or through their interactions with the other strain-producing defects. Over the past seven decades, experimental and theoretical developments have firmly established the principal role of dislocation mechanisms in defining material strength. It is now understood that macroscopic properties of crystalline materials are derivable, at least in principle, from the behavior of their constituent defects. However, this fundamental understanding has not been translated into a continuum theory of crystal plasticity based on dislocation mechanisms. The major difficulty in developing such a theory is the multiplicity and complexity of the mechanisms of dislocation motion and interactions that make it impossible to develop a quantitative analytical approach. The problem is further complicated by the need to trace the spatiotemporal evolution of a very large number of interacting dislocations over very long periods of time, as required for the calculation of plastic response in a representative volume element. However, bridging the gap between dislocation physics and continuum crystal plasticity has become possible with the advancement in computational technology with bigger and faster computers. To this end, new computational methods in discrete dislocation dynamics (DD) have been developed. In these new models, dislocation motion and interactions with other defects, particles and surfaces are explicitly considered. However, complications with respect to dislocation multiplications, self-interactions and interactions with other defects, and keeping track of complex mechanisms and reactions have provided a new set of challenges for developing efficient computational algorithms. In this presentation, the principles of DD analysis will be presented followed by the procedure for the measurement of local quantities such as plastic distortion and internal stresses. The incorporation of DD technique into the three-dimensional plastic continuum mechanics-based finite elements modeling will then be described. Finally, examples are provided to illustrate the applicability of this powerful technique in material engineering analysis.

**3:30 PM V2.5**
**Multiscale Modeling of Dislocation/Grain Boundary Interactions.** Michael Peter Dewald and William A. Curtin; Engineering, Brown University, Providence, Rhode Island.

Dislocation and grain boundary migration contribute significantly to plasticity in metals, but little is understood as to how the interaction between them influence plastic response. A multiscale computational method (CADD) is used to study the effects of dislocation pile-ups on the grain boundary deformation, initiation of failure, and overall mechanical response. CADD couples discrete dislocation plasticity to fully atomistic models wherein domains are treated as atomistic or continuum regions that communicate across a coherent boundary. Dislocations can either have atomistic or continuum descriptions within a single computational framework that allows each dislocation to seamlessly convert from one description to the other. Dislocations are generated from a nucleation source in the continuum region and are driven into the atomistic region within which resides a twin boundary. Use of CADD preserves accurate atomistic details while allowing a large number of dislocations to pile up near this twin boundary, thus permitting a precise yet computationally efficient study of this phenomenon. The effects of applied loading, source strength and location, pile-up densities, and size on absorption, transmission, and damage initiation at the grain boundary as well as loading response are studied.

**3:45 PM V2.6**
**Continuum Simulation of the Anomalous Size-Dependence of the Strength of Nanocrystals.** Raul Radovitzky and Antoine Jerusalem; MIT, Cambridge, Massachusetts.

The distinctive mechanism of grain boundary sliding and accomodation observed to be operative in the deformation of nanocrystalline metals is modeled with a continuum finite element crystal plasticity formulation of the bulk material inside the grain and with a special model accounting for the grain boundary behavior. Despite its phenomenological character, the model proposed succeeds in describing the main feature of the effective behavior afforded by atomistic descriptions at a much lower cost, i.e., without the need of tracking the evolution of individual atoms. The model proposed does not suffer from the time scale limitations affecting atomistic descriptions and, therefore, enables the analysis of the mechanical response to slow deformation rates. The model parameters are calibrated to published experimental results at low strain rates. The calibrated model is used to predict the behavior at deformation rates comparable to published atomistic simulations. It is found that the model reproduces both (low-rate) experimental results and (high-rate) atomistic calculations with a unique set of parameters , thus suggesting that the significant discrepancy between experiments and atomistic descriptions may be partly due to rate dependency effects.

**4:00 PM V2.7**
**Mechanical Behavior of Copper Interconnect.** Sidonie Lefebvre, Benoit Devincre[3], Ladislas Kubin[3], Pierre Vekeman[2] and Thierry Hoc; [1]Ecole Centrale Paris, Chatenay Malabry; [2]Altissemiconductor, Corbeil Essones, France; [3]LEM/ONERA, Chatillon, France.

Mechanical properties of nanocrystalline metals differ dramatically from their bulk counterparts and have attracted a great deal of interest in recent years, but knowledge of the underlying mechanisms is still incomplete. Miniaturization of micro electronics components, which enables to decrease costs, is a necessity for the semiconductor industry: the thickness of interconnects is now around 150 nm and must soon pass below 100 nm. Unfortunately, at this length scale, mechanical stresses generate a loss of reliability. The aim of this work consists in developing a finite element model of copper behavior which takes size effects into account. We only consider grains larger than 100 nm. In order to properly understand deformation transmission in a polycrystal, we use a 2D simulation of dislocations dynamics. We first wanted to find out what is predominantly responsible, according to grain size, for slip nucleation at the interface: dislocations pile-ups at the boundary or confined geometry and decrease of length scale? In this simulation, we assumed that interfaces act as barriers to dislocation glide and also examined the possibility of dislocation emission at the grain boundary. At the same time, an uniaxial compression test on a bulk copper bicrystal has been performed. The local strain field, the crystal orientation and the stress, obtained respectively by microextensometry, electron beam scattering and X-Ray techniques, have been recorded close to and far from the grain boundary. We noticed a high heterogeneity near the interface and simulated by finite element method the hardening behavior, assuming that the mean free-path is related to the interface distance. The results showed a good agreement between numerical and experimental data.

**4:15 PM *V2.8**
**Multi-Scale Experiments and Modeling of Friction and Wear in MEMS from 1 to 1,000,000 Nanometers.**
Robert W. Carpick[1], Erin E. Flater[1], Mark D. Street[1], Can K. Bora[1], Michael E. Plesha[1], Anirudha V. Sumant[1], David S. Grierson[1], Uday D. Lanke[3], Stephen G. Urquhart[3], E. David Reedy[2], Alex D. Corwin[2] and Maarten P. de Boer[2]; [1]Engineering Physics, University of Wisconsin - Madison, Madison, Wisconsin; [2]Sandia National Laboratories, Albuquerque, New Mexico; [3]Chemsitry, University of Saskatchewan, Saskatoon, Saskatchewan, Canada.

The design of reliable MEMS devices that involve sliding surfaces requires a predictive capability for friction and wear across length

scales. We use atomic force microscopy (AFM) to resolve roughness features of actual silicon-based MEMS surfaces from the nm-to-um scale, as well as to derive the tribological behavior of nanometer-scale single asperity contacts. Various self-assembled monolayer (SAM) coatings, applied to both the sample and the AFM tip, are also studied, so that the AFM mimics the actual asperity size, shape, and composition found in MEMS devices. Agreements as well as deviations from continuum mechanics models of adhesive contact are observed, and we find that incorporation of the molecular-scale mechanical behavior of the SAMs, based on both experiments and molecular dynamics modeling, becomes essential. These results are then used as inputs to models of contacts with multi-scale roughness which allow us to predict critical contact, friction, and wear parameters for the interfaces. Our predictions are compared with experimental results from a real MEMS actuator designed for quantitative friction and wear tests, whose sliding interfaces are nearly 1 mm in length. We determine the extent to which our multi-scale model, based on experimental inputs, can predict friction and failure. Finally, we will discuss new results aimed at determining the structural and chemical effects of wear in the MEMS device. AFM images reveal extremely smooth plastically-deformed material deposited in the wear track, as well as significant contrast in dynamic AFM phase images between the wear track and the unworn surroundings, which indicates compositional heterogeneity. High-resolution chemical imaging of the damaged silicon and self-assembled monolayers is then conducted by using synchrotron-based photoemission electron microscopy (PEEM) combined with near-edge x-ray absorption fine structure (NEXAFS) spectroscopy. Part of this work was funded by Sandia. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

**4:45 PM V2.9**
**High-Resolution Measurements of The Local Rotation Matrix and The Nye Dislocation Tensor for Quantitative Testing of Deformation Theory and Modeling.*.** B. C. Larson[1], Wenge Yang[1] and Anter El-Azab[2]; [1]Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; [2]Florida State University, Tallahassee, Florida.

Establishing a direct, quantitative, 3D, spatially-resolved linkage between experimental measurements of crystal plasticity and the results of theory, computer simulations, and multi-scale modeling has been a major obstacle for fundamental investigations of deformation on mesoscopic length scales (tenths to hundreds of microns). However, the inherent spatial confinement of deformation volumes below nano- and micro-indents provides controlled deformation conditions and known boundary conditions over confined (mesoscale) volumes for which the microstructure can now be probed quantitatively and nondestructively using 3D x-ray structural microscopy. In this presentation, micron-resolution, 3D x-ray structural microscopy measurements of the local rotation (deformation) matrix under spherical indents in single crystal Cu will be discussed and used to generate micron resolution measurements of the full Nye dislocation tensor over mesoscopic length scales. The spatially resolved structure of the individual components of both the local rotation matrix and the local dislocation tensor provide direct visual insight into the complex and collective 3D lattice rotations and gradients generated by spherical indentations. Moreover, the well-defined patterns of rotations and gradients observed within the measured dislocation tensor distributions under indents provide a direct, quantitative linkage with theory, simulation, and multi-scale modeling with no adjustable parameters. Accordingly, continuum, discrete-dislocation, finite-element crystal plasticity, strain-gradient, statistical dislocation dynamics, etc. based models of deformation induced lattice rotations can be compared quantitatively with such measurements after convolution with the micron spatial resolution of the measurements. The implications of extending such measurements to bi-crystals, polycrystals, and composite materials will be discussed. *Research supported by the DOE Office of Science, Division of Materials Sciences under contract with ORNL, managed by UT-Battelle, LLC; UNI-CAT is supported by UIUC, ORNL, NIST and UOP Res., Inc; the APS is supported by the DOE.

SESSION V3: Nanowires, Single Crystal Plasticity
Chairs: Lyle Levine and Patrick Veyssiere
Tuesday Morning, November 30, 2004
Room 206 (Hynes)

**8:30 AM *V3.1**
**A Novel In-situ Mechanical Testing Device and Its Application to the Testing of Nanowires.** Yong Zhu and Horacio D. Espinosa; mechanical engineering, northwestern university, evanston, Illinois.

While in-situ measurement of mechanical properties of nanostructures (e.g. nanotubes, nanowires, and ultra thin films) is challenging, significant research efforts are being conducted in this area. The major limitation of the current experimental techniques is that both deformation and force are deduced from the microscopic imaging of specimen and testing structure. This scheme results in the impossibility of continuous observation of the specimen deformation and failure while independently measuring the applied load. In order to overcome this limitation, another approach to measure electronically the applied force has been developed by our group. In this paper, we present a novel MEMS testing stage consisting of actuator, specimen and force sensor possessing nano-Newton resolution. Two types of actuator: thermal and electrostatic (comb drive) are employed for testing stiff and compliant structures, respectively. The thermal actuator works in a displacement-controlled fashion while the comb drive actuator is force-controlled. The force sensor is based on differential capacitance measurement, from which sensor displacement is measured. Through identification of sensor stiffness, the force is obtained. In the MEMS testing stage, a controlled gap (as small as 2 micrometer) is microfabricated between actuator and sensor for mounting nanowires. Characterization and calibration of the devices has been performed. The thermal actuator has a motion range from tens of nanometers up to 1 micrometer while the comb drive actuator can move up to 4 micrometers. The motion was detected using optical microscopy and digital image analysis software. A resolution of 15 nanometers was achieved. The capacitance difference of the force sensor was measured using an integrated circuit (IC) chip, with a resolution of 0.2 femtoFarads. A calibration curve for the force sensor was obtained based on the capacitance and motion measurements. The stiffness of the force sensor was identified by resonance of the sensor structure completing the calibration process. The measurements were found to be in close agreement with the lumped analytical model used in the design stages. Gold nanowires were synthesized using anodized aluminum oxide as template. The nanowires were mounted on the testing device using a nanomanipulator from Klocke Nanotechnik. Preliminary results on the in-situ mechanical testing of these nanowires inside a scanning electron microscope (SEM) will be reported. Observations of surface texture evolution and fracture will be reported. Plasticity size effects of these 1-D structures will be discussed in relation to previous work by our group on plasticity size effects in submicron freestanding thin films. Unresolved issues concerning scaling effects will be highlighted and the potential of these new experiments in addressing these issues will be delineated.

**9:00 AM *V3.2**
**The Yield of Atomic and Nanometer Scale Metal Wires.** Ken Gall[1], Jiankuai Diao[1], Martin L. Dunn[1] and Jonathan Zimmerman[2]; [1]Department of Mechanical Engineering, University of Colorado, Boulder, Colorado; [2]Science-Based Materials Modeling Department, Sandia National Laboratory, Livermore, California.

We use atomistic simulations to systematically investigate the strength of gold wires that span atomic and nanometer size scales. The results have ramifications on the basic science of small-scale metals and the potential use of such materials in nanotechnology. We have investigated the effects of wire size, orientation, structure, and applied stress state (tension vs. compression). We discuss several critical factors that influence the yielding of metal nanowires and lead to a much different yield behavior relative to bulk single crystal and polycrystalline metals: (1) intrinsic stress and strain in the wire caused by surface stress, (2) active slip systems for the nucleation and propagation of dislocations, (3) possible preexistence or complete absence of defects such as stacking faults or dislocations, and (4) potential non-fcc wire core and surface structures. The aforementioned effects lead various interesting phenomenon such as tension-compression strength asymmetry, size-scale dependence of yield, yield above theoretical values, and extreme non-Schmid type behavior. The atomistic results are quantitatively consistent with certain experimental observations and they reveal the mechanisms for increasing strength with decreasing dimensional scale. At nanometer scales, the mechanism for strengthening involves the scarcity and low mobility of dislocations coupled with constraint from surface stresses. As the wires approach the atomic scale, the wire structure changes, dislocations cease to exist, resulting in further strengthening and finally perfect atomic separation without yield. Limitations and strengths of the current work are discussed along with future directions for nanowire modeling efforts.

**9:30 AM V3.3**
**Molecular dynamics simulation of deformation of metallic nano-wires.** Tahir Cagin, Hyon-Jee Lee, Goyung Zhang, William A. Goddard and William L. Johnson; MSC, Caltech, Pasadena, California.

We studied the deformation of crystalline and amorphous metalic

nano-wires under uni-axial tension using molecular dynamics simulations. We find that the stress, after increasing in a linear fashion until the elastic limit, drops and then increases repetetively as the strain increases. The sharp drops in the stress-strain curve are due to cooperative shear events, which result in local structural defects, such as slip or twin in the crystalline case and shear bands in the amorphous case. We introduce an improved generic method to capture the local defects, which is universally applicable to glass and crystalline materials. Using this method, we successfully capture the local defects and demonstrate the strain localization and local structure in nano-wires as a function of strain. We also study the failure of amorphous nano-wire where we found formation of single icosahedral chain before failure.

**9:45 AM V3.4**
**Atomistic Finite Deformation Simulation: A Discussion on Yield Surfaces of Single Crystalline Al under Multi-Axial Loading.** Yibin Xue, Youssef Hammi and Mark F. Horstemeyer; Center for Advanced Vehicular Systems, Mississippi State University, Starkville, Mississippi.

Atomistic simulation on finite deformation of single crystalline aluminum was performed with the Embedded Atom Method (EAM) to examine the plastic flow and determine the yield surface under the combination of tension and simple shear loads. The plastic strains were identified based on the formation of dislocation and non-proportional stress-strain behavior. The plastic strain increments were induced under incremental loading conditions. Thus, the yield surfaces were obtained both in stress space and strain space at deference stages of plastic deformation. In particular, we focused on studying the effects of spatial size scales, temperature and strain rate on yielding and hardening. The yield surfaces were found to change shapes considerably with deformations. The effect of spatial sizes, strain rates, and temperature on yield surfaces will be discussed in the context of serial and parallel computing.

**10:30 AM V3.5**
**Smaller is softer: hardness in a single ZnO nanobelt.** Minhua Zhao[1], Chuanbin Jiang[2], Shouxin Li[2], Lei Lu[2] and Scott X. Mao[1]; [1]Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; [2]Shenyang National Laboratory for Materials Science, Institute of Metal Research, Shenyang, China.

ZnO nanobelt , a quasi-one-dimensional nanostructure with rectangular cross section and perfect crystallinity, is ideal for the study of its plasticity by nanoindentations. Hardness of a single ZnO nanobelt lying on the silicon substrate was characterized by Hysitron nanoindenter with homemade sideview CCD camera in the indentation depth of 10-100nm. For comparison, similar work was done on the (0001) ZnO bulk sample. We find the hardness of the ZnO nanobelt is much smaller than that of the bulk sample, contrary to the conventional believing of "smaller is stronger" based on continuum considerations. The mechanism for the observed phenomena is analyzed in terms of discrete dislocations. This finding has significant effect on the application of quasi-one-dimensional nanostructures in micro-electro-mechanical systems (MEMS) and nano-electro-mechanical systems (NEMS).

**10:45 AM V3.6**
**Atomistic Simulation Studies of Dislocation Pattern Formation and Size Effects in Plasticity.** N. Scott Weingarten and Robin L. B. Selinger; Physics Dept., Catholic Univ., Washington, District of Columbia.

Coalescence of dislocation patterns plays a crucial role in the mechanical response of crystalline solids and in setting the characteristic length scale associated with size effects in plasticity. We present the first (to our knowledge) fully atomistic simulation study of dislocation pattern formation in a two-dimensional crystal. The simulation shows both the initial coalescence of edge dislocations to form tilt grain boundaries and subsequent microstructural evolution under increasing strain. The simulation model is a two-dimensional Lennard-Jones crystal which evolves via off-lattice Monte Carlo simulation at finite temperature. The sample, a single crystal, is confined between two hard walls which gradually bend inwards to induce bending strain, while the other two boundaries of the sample are free surfaces. Edge dislocations nucleate at the corners of the sample and glide into the interior. Dislocations annihilate on the opposite free surface or arrest near the hard walls, or they may react or combine with one another. Because the boundary conditions induce a strong strain gradient, a net density of geometrically necessary dislocations remains in the sample's interior. When the dislocation density reaches a threshold value, tilt boundaries coalesce. As strain increases, the tilt boundaries diffuse and occasionally combine. When the applied strain is held fixed, we observe stress relaxation via both dislocation glide and climb. Finally, we explore the influence of sample size and strain rate on mechanical response under bending. We demonstrate that when plastic flow is nucleation limited, constant strain rate comparisons produce an apparent reverse size effect.

**11:00 AM *V3.7**
**A New Look at Size-Affected Early-Stage Single-Crystal Plasticity for FCC Metals.** Dennis M. Dimiduk[1], Michael D. Uchic[1] and Triplicane A. Parthasarathy[2]; [1]Materials and Manufacturing Directorate, Air Force Research Laboratory, Wright-Patterson, AFB, Ohio; [2]UES, Inc., Dayton, Ohio.

There is a widespread understanding that size effects in single-crystal deformation can be attributed to a number of known sources: alterations in the kinematic barriers to glide, an enhanced dislocation density in the presence of strain gradients, or nucleation limited flow such as for metal whiskers. Here we report evidence for an important new regime of behavior that is likely affiliated with limited multiplication of dislocations resulting from physical constraints at small sample sizes. We have made novel measurements of plastic yielding in annealed single crystals, under uniaxial compression, for sample dimensions that range from 40 to 0.5 mm. These tests have been carried out on three fundamentally unique types of metals within the common Ni-alloy class; pure Ni, Ni3Al alloys, and common superalloys. The restricted sample dimensions directly limit the length scales available for dislocation processes and substructure evolution, but without imposing external barriers to flow. The results show dramatic size effects at surprisingly large sample dimensions. Further, they suggest that the classical understanding of whisker experiments may be incomplete. Importantly, the results emphasize that at the micron-size scale one must define both the external geometry and internal structure to characterize the strength of a material

**11:30 AM V3.8**
**Mechanism of Superplastic Behavior of Mg Single Crystals at Elevated Temperature.** Hiromi Miura[1], T. Sakai[1], X. Yang[1], Y. Watanabe[2] and S. Miura[3]; [1]Mechanical Engineering and Intelligent Systems, University of Electro-Communications, Chofu, Tokyo, Japan; [2]Department of Functional Machinery and Mechanics, Shinshu University, Ueda, Nagano, Japan; [3]Division of Materials Science and Engineering, Hokkaido University, Sapporo, Hokkaido, Japan.

Pure Mg single crystals were tensile tested at temperatures between 473 K and 673 K at an initial strain rate of 4.2 x 10-4 s-1 in vacuum. All the single crystals showed high ductility. Some of them showed extraordinary large elongation over 600 % in nominal strain, though the tensile tests were terminated before fracture because of shortage of the stroke capacity of tensile machine. On the other hand, the others showed much lower ductility, ranging around 200 % in nominal strain, even while it was much larger than that of polycrystals. The observed strong orientation dependence of ductility of the Mg single crystals during high-temperature deformation will be discussed in relation to slip-plane behavior and occurrence of dynamic recrystallization.

**11:45 AM V3.9**
**Void Growth and Coalescence in Solids Driven by Diffusion and Plasticity.** Kinjal Dhruva[1], Alberto Cuitino[1] and Michael Ortiz[2]; [1]Mechanical & Aerospace Engineering, Rutgers University,NJ, Piscataway, New Jersey; [2]Graduate Aeronautical Laboratories, California Institute of Technology, Pasadena, California.

We study the process of void growth and coalescence in plastically deforming solids driven by vacancy diffusion and dislocation emission. A numerical scheme based on a eulerian description of the elasto-plastic and vacancy concentration fields over a regular finite difference grid is utilized. The procedure is augmented with a level-set description of the void interfaces and immersed boundary approach, which is based on the ghost method used in fluid problems. This approach allows us to track the complex topological changes due to void coalescence without the need for remeshing. The level-set method involves the evolution of interface under a non-uniform velocity field. The velocity field in turn depends on concentration gradients as well as dislocation densities. The vacancy concentration gradients are obtained by explicitly solving time dependent diffusion equation. The dislocation field on the other hand is obtained by solving elasto-plastic equations governing dislocation motion. Once the velocity field is completely determined, the new void location is updated accordingly. The evolution and rate of growth of voids are analyzed using techniques of image analysis. The model ensures that the computational regime can be used as a representative cell of the entire solid by imposing implicit periodic boundary condition at the domain. A curvature dependent boundary condition is used at void interface.

SESSION V4: Nanocrystals, nanofilms, and multilayers
Chairs: Michael Baskes and Dieter Wolf
Tuesday Afternoon, November 30, 2004
Room 206 (Hynes)

**1:30 PM V4.1**
**A Dislocation Dynamics Study of Initial Yield, Hardening, and Macroscopic Yield in Multilayer Thin Films.**
Peter M. Anderson and Qizhen Li; Materials Science and Engineering, Ohio State University, Columbus, Ohio.

A 3D Dislocation Cellular Automaton model is employed to simulate yield and hardening in nanostructured metallic multilayer thin films. The films consist of 2 types of alternating single crystalline FCC layers with (001) epitaxy, a mismatch in stress-free lattice parameter, but no elastic modulus mismatch. The simulations monitor the operation of interfacial and threading sources during in-plane, biaxial tensile loading. At larger layer thickness, strength increases with decreasing layer thickness. This occurs since slip is confined to individual layers, prior to macroscopic yield. Hardening is also significant here, due to deposition of dislocation content at interfaces. At sufficiently small layer thickness, however, strength reaches a plateau that is primarily dictated by the mismatch in stress-free lattice parameter and internal length scale of interfacial or threading sources. This plateau occurs since slip is not confined to individual layers in this regime, even for operation of the first interfacial or threading dislocation source. The simulation results motivate a theory that captures experimental trends of multilayer yield strength versus layer thickness. They also suggest particular features of thin film systems that may allow for an increased plateau strength.

**1:45 PM V4.2**
**Dislocation Dynamics Simulations of Plastic Deformation and Strain Hardening in Thin Films.** Prita Pant[1], Klaus W. Schwarz[2] and Shefford P. Baker[1]; [1]Materials Science and Engineering, Cornell University, Ithaca, New York; [2]IBM Research, Yorktown Heights, New York.

Dislocation dynamics simulations were carried out in FCC films involving dislocation densities comparable to those observed experimentally in thin metal films (about $10^{13}/m^2$ and higher). In order to understand strengthening mechanisms in thin films, we identified the conditions that led to dislocations being stopped during cyclic loading and unloading. At low dislocation densities, threading dislocations are blocked due to short-range interactions with individual dislocations. However, as the applied strain and the corresponding dislocation density increase, the stress fields of the dislocations sum over longer distances so as to create regions where the stress is much higher or lower than the mean stress in the film. Threading dislocations now stop when they encounter a blocking interaction in a region where the stress is low. The strength of the film then depends both on the stress fluctuations and on dislocation blocking interactions. Thus, stress distributions play a significant role in trapping dislocations and must be taken into account while modeling the strength of thin films. Simulations were carried out in (001) and in (111) oriented films. A comparison of the results shows significantly different dislocation structures and higher strain hardening rates in (111) than in (001) films. This is qualitatively consistent with experimental observations in FCC metal films.

**2:00 PM V4.3**
**Characterization of Inhomogeneous Stresses and Dislocation Trapping in Passivated Thin Copper Films.** Ray S. Fertig[1], Prita Pant[1], Klaus W. Schwarz[2] and Shefford P. Baker[1]; [1]Materials Science and Engineering, Cornell University, Ithaca, New York; [2]IBM T. J. Watson Research Center, Yorktown Heights, New York.

Dislocation dynamics simulations have been used to model the evolution of the stress and dislocation structure in passivated thin copper films in response to the application of a uniform biaxial strain. The results reveal that at high dislocation densities inhomogeneous stresses arise from superposition of dislocation stress fields. These stress fluctuations, in addition to local dislocation interactions, play a significant role in stopping dislocation motion. In this report, the inhomogeneous stress fields are characterized in detail and criteria for determining where dislocations will be trapped are presented. The effect of different boundary conditions and model parameters on the inhomogeneous stress fields are also examined.

**2:15 PM *V4.4**
**When is Smaller Weaker?** Frans Spaepen, Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

The strengthening effect of decreasing sample size or microstructural length scale is well known ("Smaller is stronger"). In some nanocrystalline materials, lowering of the strength with decreasing length scale is reported. The experiments are critically reviewed with special attention for possible artifacts. Mechanisms for such softening are considered, and their experimental signatures are identified.

**2:45 PM V4.5**
**Size Effects on the Plastic Stability of Bimodal Nanolaminate Materials.** Amit Misra, Richard G. Hoagland, Duncan L. Hammon, Xinghang Zhang, J. David Embury and John P. Hirth; Materials Science and Technology Division, Los Alamos National Lab, Los Alamos, New Mexico.

Sputter deposited metallic nanolaminates exhibit unusually high hardness when the bilayer periods approach nanometer dimensions. Self-supported Cu-Nb multilayered foils were room temperature rolled to study the effect of nanostructured length scales on the deformation and fracture behavior at large plastic strains. At layer thickness of a few tens of nanometers with as-deposited yield strengths exceeding 1 GPa, Cu-Nb multilayers exhibited extraordinary plastic stability undergoing uniform reduction in layer thickness to high levels of plastic strain. However, at layer thickness of a few nanometers with as-deposited yield strengths exceeding 2 GPa, fracture by shear localization was observed at rolling reductions of only a few percent. Plastic stability was observed in bimodal multilayers with alternating bilayer periods of 80 nm and 8 nm indicating compatible deformation of the 8 nm bilayers constrained by the 80 nm bilayers. These observations are interpreted in terms of the effect of length scale on the dislocation mechanisms of deformation in nanolaminate composites. This research is funded by DOE, Office of Science, Office of Basic Energy Sciences.

**3:30 PM V4.6**
**Strength Plateaus in Thin Metal Films Approaching 100 nm Thickness.** T. John Balk, Linda Sauter and Eduard Arzt; Max-Planck-Institut fuer Metallforschung, Stuttgart, Germany.

With decreasing thickness, thin metal films typically exhibit higher strength. However, as film thickness approaches 100 nm, the room temperature flow stress of thin Cu and Au films exhibits a plateau at constant stress level. Strength plateaus thus exist in the nanoregime, for both bare and passivated thin metal films. Deformation in both Cu and Au films appears to be dominated by diffusion, which in turn appears to cause the plateaus in room temperature flow stress. However, while dislocation activity in Cu films is dominated by parallel glide in the plateau regime, Au films exhibit very little dislocation motion. Moreover, Au exhibits a two-step plateau, with a constant stress for films between 600 and 200 nm thick and another, higher stress for films 100 nm and thinner. This change in plateau height correlates with a change in grain aspect ratio. Finally, when normalized by their respective (isotropic) shear moduli, the strength plateaus of Cu and Au overlap between 400 and 200 nm, i.e. in the first plateau region for Au. The thermomechanical behavior of Cu and Au films will be discussed and compared to that of Ni and Ag films. These results will be explained in light of transmission electron microscopy observations, especially those from in-situ thermal cycling experiments.

**3:45 PM V4.7**
**Plasticity Size Effects in Thin Nickel Foils in Flexure.** Pascal Moreau[1,3], Matthieu Raulic[1,3], Ken M. Y. P'ng[2], Andy J. Bushby[2] and David J. Dunstan[1]; [1]Physics Department, Centre for Materials Research, Queen Mary, University of London, London, United Kingdom; [2]Materials Department, Centre for Materials Research, Queen Mary, University of London, London, United Kingdom; [3]Materials Department, Ecole Polytechnique de l'Universite de Nantes, Nantes, France.

Size effect in plasticity has a strong mechanical dependence on sample size for length scales below 100 microns. Size effects are observed in deformation geometries with gradients of applied strain. Fleck et al. (Acta Metall. Mater. 42, 2, 475, 1994) saw this through torsion tests on thin copper wires (less than 50 microns diameter) where thinner wires required significantly higher normalised torsions (a factor of two at around 20 microns diameter) than thicker ones. This effect was explained using the strain gradient plasticity theory. Recently, Dunstan and Bushby (Proc. Roy. Soc. A, in press) found a requirement that a yield criterion must be met over a finite thickness which implies a higher yield value when the strain gradient is high. They adopted Matthew's critical thickness theory that was developed to explain strained layer growth to predict the strength of thin copper wires and showed good agreement with Fleck's data at low strain gradient plasticity. Stolken and Evans (Acta Mater., 46, 5109, 1998) developed a versatile microbend test method that only relies on the measurement of curvature to investigate the stress-strain state of thin foils. They presented results of bending thin nickel foils which showed, in good agreement with Fleck, that the strength increases as the thickness of the foil is decreased, i.e. the size effect. However, Stolken and Evans' test, like Fleck's, only measured high values of plastic strain and they explain the observed behaviour using strain gradient plasticity theory. Here, we modified the microbend test method to measure the curvature at the onset of plasticity using a profilometer for a series of high purity thin nickel foils (10 to 125 microns thickness). By using rapid thermal annealing to control the microstructure of the foils; we distinguish between contributions from

geometrical considerations of foil thickness, the Hall-Petch effect and strain gradient plasticity. This enables a unified approach to understanding strength in small volumes.

**4:00 PM V4.8**
**Mechanical Properties of Nano-Crystalline Iron Thin Films.** Nail Chamsoutdinov, Amarante Bottger and Rob Delhez; Department of Materials Science and Technology, TU Delft, Delft, Netherlands.

The stress states of nano-crystalline magnetron sputtered iron films (grain sizes between 20 and 300 nm) were studied using wafer curvature and X-ray diffraction stress analysis. Transmission and scanning electron microscopy was applied to investigate the role of film morphology on mechanical properties. A wealth of data was gathered about macro- and micro-stress behavior dependence on grain size, porosity and heat treatment up to 800 K. It was demonstrated that the mechanical properties of nano-crystalline iron depend on porosity and defects. Due to porosity and limited grain size a mechanism of grain coalescence could result to tensile stress increase upon annealing. This mechanism in a combination with stress relieve by creep leads to a complex mechanical behavior, which deviates from the Hall-Petch (or power-law) dependence. It is concluded that, in nano-crystalline materials due to the increased role of grain boundaries and the reduced ability to relieve stresses, the defects and nano-voids are crucial for understanding their mechanical behavior.

**4:15 PM V4.9**
**Plasticity in Ni-Cu Nano-layered Systems during Indentation: a Molecular Dynamics Study.** Denis Saraev and Ronald E. Miller; Mechanical and Aerospace Engineering, Carleton University, Ottawa, Ontario, Canada.

Nanolayered materials and nanostructured films with coatings may possess interesting mechanical properties, including superhardness, due to both size and interface effects. Experimental observations show that a reduction in layer thickness can dramatically strengthen a nanolayered composite structure made of two kinds of metals. This strengthening is due to different mismatches on the interface, such as the elastic modulus, lattice parameter, chemical mismatch, and interface coherency. At the nanometer scale, the layer thickness becomes much less than the distance required for dislocations to propagate and interact. Thus, the interaction of dislocations and interfaces becomes the controlling parameter of plasticity in such systems. The detailed understanding of this problem includes creation of dislocations at interfaces, transmission of dislocations through interfaces and emission of dislocations from interfaces. Such complexity requires the necessity of atomistic modeling to elucidate the nature of the interface-controlled plasticity in nanolayered materials. In this work we employ embedded-atom simulations to provide an insight into plastic deformation of coherent Ni-Cu layer system during nanoindentation. Prior to indentation, the Ni-Cu interfaces are relaxed to make them coherent using the conjugate gradient method. This relaxation results in nucleation of misfit dislocations in the interface. Indentation is conducted at a constant velocity using a spherical indenter, simulated by a repulsive potential. During simulation the transition from reversible to permanent deformation, which corresponds to the nucleation and motion of lattice dislocations, is analyzed. Different layer order and thickness are considered. Several deformation mechanisms, such as dislocation pile-up on the interface, dislocation cross-slip and movement of misfit dislocations, are observed. Dislocations nucleated in nickel can easily transfer into copper, but not vice versa. Copper films are significantly strengthened by thin nickel coatings. These systems show the same enhanced hardness for a wide variety of coating thickness, even though the operating deformation mechanism is unique in each considered case.

**4:30 PM *V4.10**
**Deformation-Mechanism Map and Grain-Size Scaling in Nanocrystalline FCC Metals by Molecular-Dynamics Simulation.** Vesselin Yamakov, National Institute of Aerospace, Hampton, Virginia.

The mechanical behavior of nanocrystalline materials (i.e., polycrystals with a grain size of less than 100 nm) remains controversial, the observations ranging from greatly enhanced ductility to dramatically increased strength and hardness compared to coarse-grained polycrystals. We review large-scale molecular-dynamics simulations of nanocrystalline model microstructures to demonstrate how and why, at some critical grain size of the order of about 20 nm, the conventional dislocation-slip mechanism shuts down in favor of a grain-boundary based deformation mechanism. These simulations reveal that for the larger grain sizes, complete dislocations nucleate from the grain boundaries and grain junctions. Following their nucleation, these extended dislocations travel across the grains until they annihilate in some other grain boundary. A variety of well-known dislocation-dislocation interaction processes are identified and, for larger plastic strains (of typically > 5%), extensive deformation twinning is observed. As the grain size decreases, the magnitude of the stress-dependent splitting distance of these dissociated dislocations eventually becomes comparable to the grain size, preventing their complete nucleation and hence contributing ever less to the total strain. These simulations also reveal that for the smallest grain sizes and in the absence of grain growth and any dislocation activity, nanocrystalline fcc metals deform via a mechanism involving an intricate interplay between grain-boundary sliding and grain-boundary diffusion. It is shown that in the absence of grain-boundary diffusion or dislocation nucleation as accommodation mechanisms, grain-boundary sliding can cause high stress localizations, which may initiate fracture. The transition between these various deformation mechanisms is captured by a generalized deformation-mechanism map which reveals the scaling of the mechanical properties of nanocrystalline fcc metals with grain size and stacking-fault energy.

SESSION V5: Poster Session: Size Effects in Plasticity
Chairs: William Gerberich, Lyle Levine, Robin Selinger and Dieter Wolf
Tuesday Evening, November 30, 2004
8:00 PM
Exhibition Hall D (Hynes)

**V5.1**
**Length Scale Effects on Yield Phenomena During Nanoindentation.** Coralee McNee and David F. Bahr; Mechanical and Materials Engineering, Washington State University, Pullman, Washington.

Instrumented indentation to quantitatively measure the mechanical properties of small volumes of materials is a relatively standard test, and the analysis of nanoindentation behavior is extensively discussed in the literature. Two very interesting phenomena have come to light when testing materials on the sub- m scale, the first of which is commonly referred to as an excursion or "pop-in". This anomaly usually occurs during the loading portion of the test and is characterized by a sudden increase in depth without a concurrent increase in load. The second phenomenon discussed here pertains to the relation of bulk properties to sub-micron scale properties. When testing small volumes of material, the material appears much harder than compared with larger volumes of the same material, the Indentation Size Effect (ISE). With the interest in modeling deformation behavior on a small scale using discreet dislocation dynamics models, the ability to easily provide links between computational models and experiments on the same length scale suggests the extent of deformation during yield could help couple these areas of interest. A previous model for predicting the length of an excursion has been altered to accomodate descriptive analytical models for the ISE. A model which relates the deformation which occurs during the "pop in" and "excursion" phenomena noted during nanoindentation in many metals has been improved to account for scale dependent hardness (the indentation size effect). The model assumes the initiation of yielding is controlled by the start of dislocation motion, and that the tip arrest is controlled by a size dependent flow stress. Comparisons to nanoindentation data in iron are presented, and the model is shown to accurately predict the extent of plasticity which occurs in a discreet burst during an excursion at loads below 5 mN and depths below 150 nm.

**V5.2**
**Abstract Withdrawn**

**V5.3**
**Measurements and Characterizations for Microindentation Size Effects.** Yueguang Wei, Haifeng Zhao and Ying Du; Institute of Mechanics, Chinese Academy of Sciences, Beijing, Beijing, China.

Micro-indentation test at scale of hundreds of nano-meters has shown that measured hardness increases strongly with decreasing indent depth, which is frequently referred to as the micro-indentation size effect. Usually the size effect is displayed in the hardness-depth curve, however, due to the complicated definition of contact area in calculating the hardness, there are some critical disputes on the micro-indentation size effect. In the present study, the micro-indentation size effect is characterized in both the load-displacement curves and the hardness-depth curves. The experimental measurements are performed for several typical single-crystal metals, which include Al, Ag, Cu, Ni, Ti, and W, across the weakly hardening metals to the hardening metals. In order to experimentally measure the size effect, the measured loading-displacement curves and the hardening-displacement curves will be compared directly with a parabola and a constant (no size effect solution), respectively. Moreover, the micro-indentation size effect is characterized by using the dislocation density theory and the

strain gradient plasticity theory, respectively. Through comparing the theoretical predictions based on both the dislocation density theory and the strain gradient plasticity, a connection between the geometrically necessary dislocation density with the length parameter in the strain gradient plasticity theory is set up. Additionally, in order to investigate effects of some environment factors, such as, the effect of surface roughness, the effect of indenter tip curvature, etc., the specimen surface profile and the indentation imprint profile for single-crystal metal specimen are scanned and measured by using the atomic force microscopy technique. Furthermore, the micro-indentation size effect is characterized and analyzed for considering the effect of the specimen surface roughness.

**V5.4**
**The Effect of Grain Size on the Dynamic Mechanical Response of Copper; Deformation, Ductility, and Strength Model Validation.** George Thompson Gray, Ellen Cerreta, Laura Beth Addessio, Shuh-Rong Chen and Benjamin Henrie; MST-8, Los Alamos National Laboratory, Los Alamos, New Mexico.

The mechanical behavior and damage evolution of high-purity copper, a face-centered cubic metal, can be influenced by strain rate, temperature, stress state, and the effects of microstructure such as grain size. The yield strength of copper has been previously shown to be strongly dependent upon the average grain size of the material. Yield stresses were seen to decrease for increasing grain sizes as were the work hardening rates exhibited. The effects of grain size on the mechanical response of high-purity copper tested in compression are probed and the flow stress and work hardening behavior is correlated with the substructural evolution during deformation. The integrated influence of grain size on the overall constitutive response of copper is quantified utilizing Taylor cylinder impact testing coupled with 3-D finite-element simulations. Finally, dynamic extrusion experiments will be presented that demonstrate a significant influence of grain size on the large-strain dynamic tensile ductility of high-purity copper. The texture evolution in the extrusions, quantified using orientation imaging microscopy will be discussed.

**V5.5**
**The dependence of Deformation and Fracture Behaviors on Grain Size in Nanocrystalline Face-Centered Cubic Metals.** Hongqi Li and Fereshteh Ebrahimi; Materials Science and Engineering, University of Florida, Gainesville, Florida.

Recently, both experimental and computer simulation results have shown that the deformation mechanism depends on the grain size and transits at a critical grain size value. In addition, documented results propose that nanocrystalline (<100nm) metals are inherently brittle. However, no systematic experimental results are available to characterize how the tensile behaviors change with the grain size. In present study, the deformation and fracture behaviors of electrodeposited single-phase nanocrystalline face-centered cubic (FCC) Ni (44nm) and Ni-15%Fe alloy (9nm) were investigated. The grain size distribution was evaluated using the transmission electron microscopy (TEM) technique. The tensile test of the dog-bone shaped specimens having a gauge length of 5mm was conducted at room temperature. Fracture surface and the necking geometry of the samples were examined using scanning electron microscopy (SEM). Tensile stress-strain curves demonstrate that nanocrystalline FCC metals are intrinsically ductile and their failure begins with necking, regardless of the grain size. However, the area reductions and the fracture behaviors were found to be dependent on the grain size. For pure Ni, whose grain size was larger than the critical value ( 10nm), the plastic deformation is controlled by dislocation activity and the material showed large reduction in area, typical of conventional FCC metals. Consistently, the fracture surface displayed deep microvoids, strongly suggesting that pure Ni fractured by the microvoid coalescence mechanism. Interestingly, as the grain size was reduced to the regime where grain boundary sliding dominates, i.e. in the case of Ni-15%Fe alloy, the material showed very high strain-hardening rate and extensive microcracking preceded the final failure with no noticeable reduction in area. Furthermore, the fracture surface exhibited shallow microvoids and careful examinations in SEM likely confirmed the computer simulation results that the shallow microvoids are developed by detaching clusters of grain along the local shear planes formed around them. Finally, close observations in TEM illustrated that the fracture at nanoscale occurred intergranularly.

**V5.6**
**Numerical Investigation of fracture in Bimodal Nano-structured Al Alloys.** Riqing Ye, Bingqiang Han and Enrique J. Lavernia; University of California, Davis, Davis, California.

The nanostructured materials have shown many attractive characteristics, such as high strength and high hardness. However, low ductility and toughness are also observed in the nanostructured materials. Available experimental and theoretical studies attribute the low ductility of nanostructured materials to the deficit of dislocation activity. Nanostructured Al alloys with a microstructure that contains multiple length scales, from tens of nanometers to hundreds of nanometers, have demonstrated an excellent combination of strength and ductility. The present work involves a numerical simulation on the fracture process of nanostructured aluminum alloys with a bimodal structure via a finite element model. The influence of coarse grain size on the cracking, debonding and the dynamic fracture process of bimodal materials were then studied. The numerical results are in good agreement with experimental data.

**V5.7**
**Hierarchical Modeling of Grain-Boundary Effects and Failure Evolution in Nanocrystalline Systems.** T. Kameda[1], Mohammed A. Zikry[1], A.M. Rajendran[1] and Donald Brenner[2]; [1]Mechanical and Aerospace Engineering, North Carolina State University, Raleigh, NC, North Carolina; [2]Department of Materials Science and Engineering, North Carolina State University, Raleigh, NC, North Carolina.

New hierarchical computational methodologies have been developed to predict dominant material behavior and mechanisms at scales ranging from the nano to the macro. Physically based scaling relations have been developed to characterize mechanisms and grain-boundary effects in nanocrystalline materials. These scaling relations have been used to link molecular dynamic and microstructural modeling to delineate the interrelated effects of grain boundary orientation and structure, dislocation transmission, absorption, and blockage through GBs, such that dominant failure mechanisms can be accurately identified and predicted from initiation to unstable growth.

**V5.8**
**Intermittent Plastic Flow and Dislocation Patterning in Metallic Single Crystals.** Masato Hiratani and Vasily Bulatov; CMS/MSTD, Lawrence Livermore National Laboratory, Livermore, California.

Spatio-temporal patterning of collective dislocation behavior is investigated for single crystals with/ without preexisting microstructures by using discrete dislocation dynamics scheme. Under constant strain rate load, large fluctuations of dislocation velocity and the load stress are observed at low rate regime in both BCC and FCC metals. Pair correlation analysis reveals rapid dislocation clustering or strain localization in FCC metals during low strain rate loading or relaxation while more random structures are developed at higher strain rate. The time series of stress and local strain signals at the low rate indicate self-affine or multifractal behavior, which is reminiscent of the self-organized criticality. When weak local obstacles exist, glassy dislocation motion and dislocation avalanches are seen in early deformation stage, and the dynamical quantities are found to follow a power law of inter-obstacle spacing and simulation cell size. The work is performed under the auspices of DOE under contract number W-7405-ENG-48.

**V5.9**
**Numerical Efficiency of Materials Modeling Algorithms.** Stephen Kuchnicki[1], Zisu Zhao[2], Alberto Cuitino[1] and Raul Radovitzky[2]; [1]Mechanical and Aerospace Engineering, Rutgers University, Piscataway, New Jersey; [2]Aeronautics and Astronautics, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Material models using numerical integration are inherently time-step-dependent. To justify application of material models verified against quasi-static tests to large-scale dynamic simulations requiring shorter time steps, the computational analyst must be able to guarantee invariance of his numerical algorithm output with reductions in the time step. A validation paradigm compensating for time-dependence is introduced, introducing the concept of time-convergence of models. The more rigorous condition of time-convergence combined with the small time steps required by large-scale dynamic simulations necessitate a recasting of the numerical integration into an explicit form. The maximal time-step limitation of the explicit integration step is circumvented through use of a subcycling algorithm that subdivides the input time and deformation steps into smaller increments that can be integrated successfully with the explicit algorithm. A generalized algorithm that selects implicit or explicit integration based on the current model state is introduced. This model is successfully applied to large-scale simulations for both face-centered cubic (FCC) and body-centered cubic (BCC) metals. The results of these simulations are compared to experimental observations of stress-strain response and microstructural evolution found in the literature.

**V5.11**
**Strain hardening of nanostructured Cu processed by ECAP, cold drawing and rolling.** Yonghao Zhao[1], Xiaozhou Liao[1], Ruslan Z. Valiev[2] and Yuntain T. Zhu[1]; [1]MST, Los Alamos National Lab., Los Alamos, New Mexico; [2]Institute of Physics of Advanced Materials,

Ufa State Aviation Technical Unversity, Ufa, Russian Federation.

Strain hardening plays an important role to obtain stable plastic flow and delay the onset of localized deformation (necking) under tensile stress. However, most nanostructured metals have been found to exhibit zero strain hardening. In present work, we found nanostructured Cu, processed by ECAP following by cold drawing and rolling, has an evident strain hardening during tensile test. While the ECAP processed nanostructured Cu has no strain hardening. The comparable studies of the microstructural characteristics of the above two nanostructured Cu and the microstrutural evolutions during tensile test will be performed to explore the strain hardening origin of nanostructured Cu.

**V5.12**
**Nanodiamonds are Stable Phase: A Nanothermodynamic Approach.** Cheng-Xin Wang, Qiu-Xiang Liu, Yu-Hua Yang and Guo-Wei Yang; State key laboratory of optoelectronic materials and technologies, School of Physics Science & Engineering, Zhongshan University, Guangzhou 510275, P. R. China, Guangzhou, China.

Since Lewis et al. found nanometer-scaled diamonds from primitive meteorites in 1987, a lot of researchers have also detected nanodiamonds in protoplanetary nebulae, interplanetary dust originating from comets and asteroids, and some circumstellar disks. All extraterrestrial nanodiamonds have similar size distribution, i.e., 3-5 nm, so far. On the Earth, nanodiamonds synthesize by detonation, chemical vapor deposition (CVD), and hydrothermal synthesis and reduction of carbide (HSRC). Pure TNT detonation synthesizes nanodiamonds with diameters about 10 nm. However, Chen et al. synthesized mostly spheroidal diamond nanoparticles with sizes of 3-6 nm by detonating TNT in the inert gases atmosphere. Similarly, nanodiamonds have been produced using CVD techniques. Lee et al. found diamond nanoparticles with diameter of 2-6 nm (small enough to identify the nucleation sites) in CVD diamond films. Furthermore, Gruen et al., by partially or outright replacing the hydrogen with argon in the CVD process, deposited the smooth films of nanoparticles only a few nanometers in size. Recently, the important progress of micron-sized diamond synthesis was developed by HSRC with hydrogen or without hydrogen under conditions of pressure less than 200 MPa and temperature not exceeding 1300 K. Importantly, Gogotsi et al. realized the conversion of silicon carbide to diamonds with diameter of about 5 nm in the noble gas atmosphere under ambient pressure and temperature less than 1300 K conditions. Thus, both extraterrestrial and earth's nanodiamonds are not only stable, not also have the nearly equal size distribution. These important data imply that there would be a common underlying factor in the formation of nanocrystalline diamonds. To our best knowledge, a few studies are involved in the stability of nanodiamonds. However, in all these calculations and experiments mentioned above, we hardly find the reasonable and satisfying explanation for the two fundamental issues: which physical origin causes the nanodiamond stability and why the nearly equal size distribution of nanodiamonds produced in space and on the Earth under very different temperature-pressure conditions is in the range of 3-5 nm. To gain a better understanding of the physical origin of the two fundamental issues mentioned above from the point of view of thermodynamics, in this study, we therefore perform the phase stability analysis of nanodiamonds and calculations of the critical nuclei sizes of diamonds upon CVD and HSRC systems. Interestingly, our theoretical results show that the radiuses of critical nuclei of diamonds upon CVD and HSRC are less than 2.5 and 3.0 nm, respectively. Furthermore, our analysis indicate that at atmospheric pressure and temperature not exceeding 1300 K, nanodiamonds less than 6 nm in diameter are thermodynamic stable phase. Additionally, we provide a reasonable explain for the diamond nucleation upon the hydrogen-free CVD.

**V5.13**
**Mechanical Properties of Fe/Cu Multilayers.**
Nail Chamsoutdinov, Department of Materials Science and Technology, TU Delft, Delft, Netherlands.

Fe/Cu multilayers are interesting target for investigating mechanical behavior of nano-grains since these two metals do not mix at low temperatures and therefore it is possible to control the grain size of both metals. In this work the evolution of stress, texture and morphology in Fe/Cu multilayers (grain sizes from 10 to 60 nm) is investigated by means of X-ray diffraction and wafer curvature technique. The transmission electron microscopy and small angle X-ray scattering were used to check the grain size and morphology. The results are discussed in the framework of constrained dislocation motion in nano-grains and influence of the interface stress.

**V5.14**
**Abstract Withdrawn**

**V5.15**
**Nucleation and Propagation of Deformation Twin in Polysynthetically Twinned TiAl.** Longguang Zhou[1], Luke L. Hsiung[2] and Hanchen Huang[1]; [1]Dept. of Mechanical, Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, New York; [2]Chemistry and Materials Science Directorate, Lawrence Livermore National Laboratory, Livermore, California.

Using molecular dynamics simulations, we study the deformation of polysynthetically twinned (PST) TiAl at room temperature. The simulation cell is pre-strained and thermodynamically relaxed to zero stress, so that no dislocations pre-exist in $\gamma$-$\alpha_2$ interfaces. A uniaxial compression is then applied along one 1/6<112] direction. Our results show that interfacial dislocation pairs nucleate at the $\gamma$-$\alpha_2$ interface under compression. The glide and agglomeration of these dislocations lead to the nucleation of deformation twins from the interface. Based on our studies, twins may nucleate without pre-existing interfacial dislocations. Further we have monitored the propagation of the deformation twin, specifically its interaction with $\gamma$-$\gamma$ and $\gamma$-$\alpha_2$ interfaces. The observations show that $\gamma$-$\alpha_2$ interfaces are stronger obstacles to the twin propagation than $\gamma$-$\gamma$ interfaces are.

**V5.16**
**Second Phase Induced Deformation Mechanisms in Two-Phase Titanium Alloys.** Allan Jaworski Jr. and Sreeramamurthy Ankem; Materials Science and Engineering, University of Maryland, College Park, College Park, Maryland.

Room temperature tensile deformation mechanisms were studied on two different alpha-beta titanium alloys, Ti-6.0Mn and Ti-8.1V. When the single phase alpha of these two alloys are deformed the deformation mechanisms were found to be slip or slip and twinning, depending on the grain size. In the case of the single phase beta alloys, the beta phase of the Ti-V system deforms by slip and twinning, whereas deformation in the beta phase of the Ti-Mn system occurs solely by slip. However, when the two phases are together, i.e. in the two-phase alloys, new deformation mechanisms were found in addition to slip or slip and twinning. For example, in the two-phase Ti-8.1V alloy, the tensile deformation mechanism in the beta phase was found to be stress induced martensite. These new mechanisms appear to be associated with elastic interaction stresses between the two phases and the presence of metastable nanocrystalline omega phase. A detailed account of these deformation mechanisms will be presented. This work is being funded by the National Science Foundation under grant number DMR-0102320.

**V5.17**
**Deformation Mechanisms of Al-Zn-Mg Age-Hardening Alloy.** Nina Alexandrovna Grigorjeva and Tatiana Andreevna Kovalevskaya; Theoretical Mechanics, Tomsk State Univ. of Architecture, Tomsk, Russian Federation.

The plastic behavior of Al-6%Zn-3%Mg alloy was studied. Specimens were deformed at various strains by compression and tension. The mechanical properties, the evolution of dislocation substructure and surface deformation relief were investigated/ The maximum quota of date was obtained for the aged alloy, which yield stress was highest, and for the quenched alloy (supersaturated solid solution). The experimental results seem to be consisting with such suggestion: the shear zone formation leads to the local substructure transformations.

**V5.18**
**Structure Evolution of Plastic Deformed ZnS Crystals.** B.A. Abdikamalov[1], Igor Bdikin[2,3], U.K. Ernazarov[1] and A.L. Kholkin[2]; [1]Berdah Karakalpak State University, Nukus, Republic of Karakalpakistan, Uzbekistan; [2]Department of Ceramic and Glass Engineering, CICECO, University of Aveiro, Aveiro, Portugal; [3]Institute of Solid State Physics, Chernogolovka, Moscow distr., Russian Federation.

Polysynthetic zinc sulfide (ZnS) crystals under monoaxial plastic deformation are transformed to single sphalerite crystals with "S - type" bending of slip plane substructure [1,2]. The mechanism of formation of this substructure was not discussed in literature. In present work, the cross sections of reciprocal lattice nodes of domains from two different possible orientation states obtained by X-ray diffraction (DRON-UM-1) were analyzed. Cross sections of $400_I$ and $331_{II}$ nodes of reciprocal lattice (indexes related to two orientation states) of the virgin crystal are extended along [111] direction and connected to each other. Appreciable changes of the form of diffraction sections of reciprocal lattice nodes are observed already in the initial stages of the plastic deformation ($\epsilon \sim$1-3 %). Under this deformation, the cross sections nodes are extended along [11-2] direction, that is the point where the formation of monodomain crystal substructure begins. The analysis of X-ray rotation patterns of the crystals deformed up to $\epsilon$>8% was made. The system of

microcrystals with the size of 100 Å and misorientation of 5-7° around [110] direction was formed. The obtained results show that the substructure of the uniaxially deformed ZnS crystals is characterized by the presence of non-coherent domain borders with fixed insulated partial dislocations. In places where partial dislocations (slipped on these borders) meet with fixed dislocations, there exist stable dislocation complexes (walls from the same dislocations, agglomerations the same dislocations, etc.). Arisen dislocation walls contribute to domain misorientation of single domain states, and, as a result, the agglomerations from the same dislocations soften their misorientations. On macroscopic scale these dislocation complexes create the "S-type" bending of slip plane around [1-10] direction, normal to directions of applied force and easiest slip. 1. I. M. Shmytko, L. A. Matveeva, S. I. Bredikhin, V. Sh. Shekhtman, S. Z. Shmurak, Phys. Solid State 26 (1984) 1233. 2. B. A. Abdikamalov, M. T. Erezhepov, U. K. Ernazarov, Phys. Solid State 34 (1992) 757.

**V5.19**
**Oscillating Dislocation Structure During Local Melting and Solidification of Ni-based Superalloys.** Oleg M. Barabash, Joe A. Horton, Suresh S. Babu, John M. Vitek, Stan A. David, Judy Pang, Gene E. Ice and Rozaliya I. Barabash; Matals&Ceramics, Oak Ridge National Laborory, Oak Ridge, Tennessee.

We studied how the dislocation structure of Ni-based single crystal superalloy changes during melting and solidification. In our study, we combined polychromatic microbeam synchrotron diffraction measurements together with electron and optical microscopy. We show that the distribution of the thermal gradient is not monotonic. The maximum value of the thermal gradient is observed in the heat affected zone near the fusion line. Periodic dislocation structure is formed during continuous movement of melt zone in thin Ni-based superalloys sheet. Moreover we observe oscillations in the dislocation structure formed under such conditions at both macro and micro scales. Depending on the temperature, the formation of dislocations is accompanied by the partial or complete dissolution of $\gamma$' particles in the matrix. Dislocations form and multiply due to thermal gradients. Their arrangement correlates with the temperature gradient field and with the dissolution and re-precipitation of $\gamma$' particles. The distribution of the dislocation density at the macroscale is due to symmetric temperature gradient perpendicular to the direction of melt zone movement. Within the above macro regions of dislocations oscillations of dislocation density due to grouping at the micro scale were also observed. Typical length scale of dislocation density oscillations is related to the dendrite size and the conditions of local melt and solidification.

**V5.20**
**Fabrication and Characteristics of Al2o3/Al Composite using Porous Alumina Bodies Made by Gel-Casting Method.** Sung Jae Je[1], Jae-Won Kim[1], Yeon Gil Jung[1] and Chang Yong Jo[2]; [1]Department of Ceramic Science and Engineering, Changwon National University, Changwon, Kyungnam, South Korea; [2]High Temperature Materials Group, Korea Institute of Machinery and Materials, Changwon, Kyungnam, South Korea.

Al2O3/Al composites are produced by infiltration of Al alloy into porous alumina bodies. Green bodies are fabricated by a gel casting of slurries dispersed with AKP-30 and PMMA bead as 1:1, 1:2, 1:3 and 1:4 volume ratios respectively. Green bodies are sintered at 1600°C for 2hrs after burning out PMMA bead at 400°C for 2hrs in air. Porosity of porous alumina bodies represents 36%, 51%, 62%, and 70% according to volume ratios of between PMMA bead and AKP-30. Al2O3/Al composites are fabricated by infiltration of Al alloy into the porous bodies. Properties such as density, porosity, shrinkage, and thermal expansion coefficients of the composites are measured. Microstructure of the composites observed by using scanning electron microscope. Mechanical properties of the composites are dependent on the amount of Al alloy infiltrated.

**V5.21**
**Fabrication of Open-Cell Mullite Forms using Gel-Casting Process.** Seong-Hwan Park, Jae-Won Kim, Dong-Baek Kim and Yeon-Gil Jung; Ceramics Science and Engineering, Changwon National University, Changwon, Kyungnam, South Korea.

Open-cell mullite ceramics are fabricated through a gel-casting process, of which processing parameters are investigated. Two kinds of mullite precursor powder are prepared by dissolution of two kinds of aluminium salts (Al(NO3)3−9H2O; type I and Al2(SO4)3−16H2O; type II) in the mixture of colloidal silica sol, respectively. To produce porous mullite ceramics, both mullite precursor powders and PMMA bead ( 5$\mu$m) are co-dispersed by electrosteric stabilized mechanism in aqueous system and then gelcasted. Gel-casted green bodies are sintered at 1600°C for 3h after drying at room temperature. The characteristic of each porous mullite according to a kind of aluminium salt is investigated by XRD, SEM, and mercury porosimeter. The effects of aluminium salt on the synthesizing behavior are investigated.

**V5.22**
**Elaboration by Severe Plastic Deformation, Microstructural Study and Mechanical Characterization by Nanoindentation of New Cu/Ta Nanofilamentary Wires for Use in High Field Magnet.** Vanessa Vidal[1,2], Ludovic Thilly[1] and Florence Lecouturier[2]; [1]Universite de Poitiers, Laboratoire de Metallurgie Physique, Futuroscope, France; [2]INSA, Laboratoire National des Champs Magnetiques Pulses, Toulouse, France.

The elaboration of new reinforced conductors, i.e. with high electrical conductivity and high strength, is essential to provide non-destructive high pulsed magnetic fields over 80 Teslas. Currently, the compromise is obtained with continuous Cu/Nb nanofilamentary wires that combine high strength (2 GPa) with low electrical resistivity (0,6 $\mu$ohm.cm) at 77K [1]. The elaboration process of these nanofilamentary conductors is based on severe plastic deformation applied by repeated drawing and bundling stages. This process leads to a copper matrix containing N=$85^4$ (52.2 $10^6$) continuous and parallel Nb filaments with diameter down to 25 nm. Thus, these nanocomposite wires, with a multi-scale structure, present the remarkable advantage of combining high electrical conductivity and ultra-high strength, very much higher than that predicted by the classical Rule of Mixture. In the first generation of composites, the strength of Nb fibers, that are nanowhiskers, was observed to be inversely proportional to their diameter and approached, for smallest diameters, the theoretical strength for perfect crystals G/2$\pi$. The mechanical properties of the Cu/X nanostructures can therefore be improved by using a reinforced material with a shear modulus G higher than that of Nb ($G_{Nb}$=40GPa): Tantalum was chosen since its shear modulus meets the precedent requirement ($G_{Ta}$=69GPa). Thus, co-deformation tests by cold drawing were performed on the Cu/Ta composite. They revealed the formation of a macroscopic roughness at Cu/Ta interfaces that has been analysed in terms of Grinfeld instabilities [2,3]. This phenomenon, leading to the Ta core fractures, limited the fabrication of Cu/Ta composites, but was finally suppressed by using hot extrusion step prior to the cold drawing process. The results are promising since conductors containing N=$85^3$ and N=$85^4$ Ta filaments were successfully extruded and drawn without failure. Transmission Electron Microscopy observations have been carried out to study the microstructure of the Cu/Ta nanocomposite and are presented for the first time. Mechanical properties information provided by the nanoindentation technique are discussed and allow for the analysis, in the nanometre scale, of the mechanical properties in terms of size effect. These results are then compared to those obtained with the Cu/Nb nanofilamentary wires [4,5]. [1] Advanced Engineering Materials 6(5), 290-297, 2004 ; [2] Acta Materiala 47(3), 853, 1999 ; [3] Acta Materiala 47(9), 2761, 1999 ; [4] Acta Materiala 50(20), 5049-5065, 2002 ; [5] Acta Materialia 51, 195, 2003

**V5.23**
**Particle Size and Processing Effects in Polymer Bonded Sugars.** Clive R. Siviour[2], Chad G. Rumchik[1] and Jennifer L. Jordan[1]; [1]MNME, Air Force Research Laboratory, Eglin AFB, Florida; [2]PCS Group, Cavendish Laboratory, University of Cambridge, Cambridge, United Kingdom.

Polymer bonded explosives (PBXs) are widely used in environmentally demanding military and industrial applications. Understanding the effects of composition and processing on the mechanical behavior of these materials is of utmost importance as optimized mechanical and detonation properties allow a greater operational envelope. Until recently, most research has concentrated on production formulations rather than systematic studies of particle size or processing effects. This study describes results of mechanical tests on polymer bonded sugars, a common mechanical simulant for PBXs, with different particle sizes and loading densities. Materials are tested across a wide range of strain rates from $10^{-2}$ to $10^5$ using conventional Instron and novel split Hopkinson pressure bar techniques.

**V5.24**
**Abstract Withdrawn**

**V5.25**
**Compressive Behavior and Deformation Mechanisms of Cryomilled Nanostructured Al-Mg Alloys.** Guojiang Fan[1], E. J. Lavernia[2], Hahn Choo[1] and P. K. Liaw[1]; [1]Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee; [2]Department of Chemical Engineering and Materials Science, The University of California, Davis, California.

The low ductility that is frequently reported for nanostructured alloys, which are processed via a milling (e.g., attrition, cryomilling, etc), is believed to be due to a lack of dislocation activity. However, nanostructured alloys with bimodal structures show a much improved

ductility. In the present investigation, a cryomilled bimodal Al-Mg alloy was employed to study plastic deformation during compression at different strain rates. The compressive test was completed on samples that were polished to a mirror finish in order to facilitate microstructural analysis. The effect of strain rate and strain level on the morphology of the deformed area as well as stress-strain relationships were analyzed. The deformation mechanisms in terms of the interplay between the dislocation slip and shear banding are discussed, based on the experimental observations. It was found that plastic deformation of the nanostructured bimodal Al-Mg alloy shows a negative strain rate sensitivity, which contributes to the plastic instability. The yield strength as a function of grain sizes for the nanostructured Al-Mg alloy deviates from the Hall-Petch relation, indicating the lack of dislocation pile-ups at grain boundaries during plastic deformation.

**V5.26**
**Electrodeposition of Nanostructured Ni-Co Alloys with Controlled Grain and Twin Sizes.** Bruce Wu and Christopher Schuh; Massachusetts Institute of Technology, Cambridge, Massachusetts.

The importance of nanoscale twins in the plastic deformation of nanocrystalline metals and alloys has begun to be recognized, both through detailed electron microscopy studies and molecular dynamics simulations. In light of these results, it would be very interesting to have experimental control over both grain and twin size in bulk nanostructured alloys. To this end, we have synthesized nanocrystalline Ni-Co using pulsed electrodeposition from a Watts-type bath; owing to the low stacking fault energy of Co, many of the alloy deposits exhibit a large density of nanoscale twins. The various plating parameters have been systematically varied to examine their effect on the composition, quality, and, most importantly, grain size and growth-twin density of the resulting deposits. The unique dual-scale grain and nano-twin structures we have produced lead to difficulties in assigning a single microstructural length scale, and in interpreting trends in microhardness measurements.

**V5.27**
**Plane strain machining as a technique for studying strain-gradient effects in materials.** M. Ravi Shankar, Seongeyl Lee, Jihong Hwang, Srinivasan Chandrasekar and W. Dale Compton; Industrial Engineering, Purdue University, West Lafayette, Indiana.

A study has been made of the large strain deformation field in plane strain machining of metals. The use of a high-speed, Charge-Coupled Device (CCD) imaging system, in conjunction with a sharp sapphire tool, has enabled the deformation field in the primary deformation zone to be resolved with high spatial and temporal resolution. The displacement, velocity and strain distributions have been obtained by applying a Particle Image Velocimetry (PIV) technique to sequences of high-speed images of the deformation zone recorded from a side of the workpiece. The measurements have shown that, in low-velocity machining of metals, typical strains are in the range of 1-5, and average strain gradients are an order of magnitude higher than the corresponding gradients in indentation. A model for estimating the density of geometrically necessary dislocations in the deformation zone is presented. The use of plane strain machining as a controlled experimental technique for studying strain gradient effects in metals is discussed in the context of these observations.

**V5.28**
**Investigation of Precipitation and Deformation Mechanism in Cryomilled Bulk Nanocrystalline Al-Mg Alloys using Transmission Electron Microscope.** Zonghoon Lee[1], Enrique J. Lavernia[2] and Steven R. Nutt[1]; [1]Materials Science, University of Southern California, Los Angeles, California; [2]Department of Chemical Engineering and Materials Science, University of California at Davis, Davis, California.

The precipitation and deformation mechanism in cryomilled bulk nanocrystalline Al-Mg alloys were investigated using analytical and high resolution transmission electron microscope. Al-Mg alloys were prepared by cryomilled nanocrystalline powder, and then consolidated by hot isostatic pressing and extrusion to produce bulk samples, resulting in high strength. The precipitations such as aluminum nitride and oxide were not easily resolvable in microscope because those precipitations were pretty small and not fully crystallized. Thus, the grown crystalline precipitations were investigated using both analytical and high resolution transmission electron microscope in this study. The enhanced strength of Al-Mg alloy might arise from the combination of ultrafine grain size and the existence of precipitations. This process demonstrates an approach to designing and producing bulk nanocrystalline metals that exhibit a high strength.

**V5.29**
**Size Effects in Fe-Mn and Fe-Ni Alloys Nanostructured by Mechanical Alloying.** Leonid Yur'evich Pustov[1], Sergey Dmitrievich Kaloshkin[1], Emmanuil Isaakovich Estrin[2] and Evgenii Vladimirovich Shelekhov[1]; [1]Laboratory of Amorphous and Microstructural Alloys, Moscow Institute of Steel and Alloys, Moscow, Russian Federation; [2]Central Research Inst. of Ferrous Metallurgy, Moscow, Russian Federation.

In Ferro-rich Fe-Mn and Fe-Ni alloys martensitic transformation is the main mechanism of realization of polymorphism in solid state, which causes their unique properties, in particular strain hardening, superplasticity etc. It is known that the martensitic transformation temperature ($M_S$) depends on strength of the initial phase, which is a function of the grain size. High strength of the phase hampers the shear mechanism of the martensitic transformation. Therefore $M_S$ temperature dependence on the grain size of initial phase ($d$) is likewise to Hall-Petch dependence of strength: $M_S = M_0 - Kd^{(-0.5)}$, where $M_0$ - martensitic transformation temperature in the monocrystal, K-constant depending on the preparation method. $Fe_{100-x}Mn_x$ ($6 \leq x \leq 30$) and $Fe_{100-y}Ni_y$ ($12 \leq y \leq 28$) alloys (x and y given as at%) were prepared by mechanical alloying (MA) of elemental powders in a high-energy planetary ball mill. X-ray diffractometry, Mossbauer spectroscopy and SEM were used to determine structure and phase constitution of the samples. Thermo-magnetic measurements were used to determine the phase transformation temperatures. Indentation was used to measure the hardness. MA treatment led to formation of bcc $\alpha$-Fe and fcc $\gamma$-Fe based solid solutions according to equilibrium phase diagram. Structure of MA alloys characterised by high concentration of defects and fine crystallite size ($d$=10-20 nm). As-milled alloys were subjected to the different thermotreatments. A part of the as-milled alloys were compacted using hot pressing. The temperature of $\gamma \rightarrow \alpha$ martensitic transformation in MA alloys were significantly depressed (on 300-350 degrees) in comparison with ordinary alloys due to nanocrystalline structure ($d$=60-90 nm). As a result single $\gamma$-phase structure was observed at room temperature after annealing for $x \geq 10$ in $Fe_{100-x}Mn_x$, and for $y \geq 18$ in $Fe_{100-y}Ni_y$ samples. Bulk samples inherited nanocrystalline structure and phase constitution of the as-milled alloys. Increasing the temperature or duration of the annealing of the samples in the temperature range of -phase stability led to gradual increase of the grain size and the $M_S$ temperature. Austenic alloys possessed the high sensitivity to martensitic transformation under mechanical deformation: $\gamma$-phase partially transformed to $\alpha$-phase that was accompanied by appearance of the ferromagnetic properties and strain hardening. Fraction of the martensitic phase in structure linearly depended on the deformation degree of the compact sample. Indentation hardness of the compacted samples was significantly increased (up to 7 GPa) in comparison with that for ordinary alloys, which was attributed to nanocrystalline structure of the MA alloys.

**V5.30 TRANSFERRED TO V7.1**

SESSION V6: Nanocrystals
Chairs: Diana Farkas and Robin Selinger
Wednesday Morning, December 1, 2004
Room 206 (Hynes)

**8:30 AM *V6.1**
**Large Scale Simulations of Deformation and Fracture Behavior in Nanocrystalline Ni.** Diana Farkas, Materials Science and Engineering, Virginia Tech, Blacksburg, Virginia.

Atomic scale computer simulation using empirical potentials provides a unique opportunity to study mechanical behavior of nanocrystalline materials yielding details of the underlying mechanisms that are not accessible experimentally. Simulation results will be discussed for deformation and fracture behavior of nanocrystalline Ni of various grain sizes in different configurations. The results include fully 3D samples with random grain orientations as well as columnar grain structures, with diameters up to 40 nm. Size effects on tensile deformation and fracture behavior of these materials will be discussed. The basic failure mechanisms are found to be related to the formation of nanovoids along grain boundaries ahead of the main crack.

**9:00 AM V6.2**
**Deformation Mechanism in Nanocrystalline FCC Metals: Bridging Experiments with Simulations.**
Helena Van Swygenhoven, Peter M. Derlet and Anders Froseth; ASQ/NUM, Paul Scherrer Institute, PSI-Villigen, Switzerland.

Atomistic simulations have provided unprecedented insight into the structural and mechanical properties of nanocrystalline materials, highlighting the role of the non-equilibrium grain boundary structure in both inter- and intra-deformation processes. One of the most important results is the capability of the nanosized grain boundary to act as source and sink for dislocations. The dislocation activity suggested by molecular dynamics for three different nc-fcc metals, Al,

Cu, Ni and Au are discussed in terms of the inherent restrictions of the technique, in terms of material properties such as the generalized stacking fault energy curves (Nat. Mat. 3(2004)401), in terms of grain boundary structures and last but not least in terms of experimental observations including efforts to bridge simulations to experiments by means of diffraction pattern calculations.

**9:15 AM V6.3**
**Plastic deformation with a reversible peak broadening in electrodeposited nanocrystalline Ni.** Zeljka Budrovic[1], Helena Van Swygenhoven[1], Peter M. Derlet[1], Steven Van Petegem[1] and Bernd Schmitt[2]; [1]ASQ/NUM, Paul Scherrer Institute, PSI-Villigen, Switzerland; [2]SYN, Paul Scherrer Institute, PSI-Villigen, Switzerland.

Electrodeposited nc-Ni, ultra-fine grained HPT-Ni and coarse grained Ni are deformed under tensile conditions in-situ in the materials beamline of the Swiss Light Source. As expected for coarse grained materials, peak profile analysis demonstrates that plastic deformation is governed by dislocation mediated processes that accumulate a residual dislocation network producing inhomogeneous strains and an irreversible broadening of the Bragg peaks in X-ray diffraction (Science 304 (2004) 273). A similar behaviour is also observed for HPT-Ni. On the contrary, the peak broadening during plastic deformation of nanocrystalline Ni is reversible upon unloading, demonstrating that the deformation process does not build up a residual dislocation network. Moreover, no increase in stacking fault density is measured. The results are discussed in terms of the microstructure and deformation parameters such as strain rate and deformation temperature. It will be shown that this new in-situ technique, based on well known peak profile analysis methods, is an important new approach for addressing the relationship between microstructure and mechanical properties.

**9:30 AM V6.4**
**Direct Evidence of a Deformation Mechanism Crossover in Nanocrystalline Nickel.** Zhiwei Shan[1], Jorg Wiezorek[1], Scott Mao[1], David Follstaedt[2], Jim Knapp[2] and Eric Stach[3]; [1]School of Engineering, Universtiy of Pittsburgh, Pittsburgh, Pennsylvania; [2]Physical and Chemical Science Center, Sandia National Laboratories, Albuquerque, New Mexico; [3]NCEM, Lawrence Berkeley National Laboratory, Berkekey, California.

We report the results of in-situ transmission electron microscope uniaxial tensile tests of nanocrystalline nickel films with an average grain size of about 10 nm. We have found that not only do grain boundary mediated processes become prominent as a deformation mode but a unique defromation mechanism for nanocrystalline metals - grain agglomeration resulting from grain rotation - is observed. Additionally, trapped lattice dislocations are found in individual grains following deformation. A grain size dependent mechanism is proposed to account for the observed change in the prevalent deformation mode. The general implications for interpreting the unusual mechanical behavior of nanocrystalline materials in light of the observed phenomenon will be discussed.

**9:45 AM V6.5**
**HREM Characterization of Plastically Deformed Nanocrystalline Aluminum.** Mingwei Chen, [1]Institute for Materials Research, Tohoku University, Sendai, Japan; [2]Department of Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland.

The extremely high strength and hardness of nanocrystalline materials relative to their coarse-grained counterparts suggest that normal dislocation activity, the dominant plastic deformation mode of ductile coarse-grained materials, may be inhibited by nano-grains. The resultant high stresses may give rise to unique plastic deformation models that are not seen in coarse-grained materials. Various deformation models have been suggested by recent molecular-dynamics simulations. However, definitive experimental observations in support of these mechanisms are limited. Here I report high-resolution transmission electron microscope observations that evidence deformation twins, stacking faults, disclination dipoles and grain boundary evolution in plastically deformed nanocrystalline Al. The presence of these deformation modes is directly related to the nanocrystalline structure because they are rarely observed in coarse-grained Al. A dislocation-based model has been proposed to uncover the preference for the partial dislocation related deformation in nanocrystalline materials. These results underscore a transition from normal slip to partial dislocation controlled deformation mechanisms when a grain size decreases to tens of nanometers.

**10:30 AM *V6.6**
**Atomistic Simulations of the Plasticity Behavior of Polycrystalline Metals.** Michael Baskes and S. G. Srinivasan; LANL, Los Alamos, New Mexico.

Recent advances in computers and atomistic modeling have made the realistic simulation of materials behavior possible. It is now possible to predict the mechanical properties of small volumes of materials containing microstructural features. We examine the effects of deformation on polycrystalline FCC metals. We perform simple shear molecular dynamics simulations using the EAM on nickel samples of $10^4$-$10^6$ atoms to study yield and work hardening. It is found that the deformation is always inhomogeneous when a grain boundary is present. The atomistic simulations reveal that dislocations nucleating at grain boundaries are critical to causing yielding in pristine material as observed in experiment [1, 2]. Detailed investigation shows that the grain boundaries are significantly weaker than the bulk material and yield at a lower stress. Even so, the yield stress of the polycrystalline samples with both low angle and high angle grain boundaries are found to be similar and only slightly lower than the yield stress of single crystals with the same characteristic dimensions. Reduction of geometric constraint in the calculations results in a significant lowering of the yield stress. Complex nanostructures are developed when a shock wave passes through a single crystal. We will present the evolution of these nanostructures and predict the resultant mechanical properties of this material. References [1] J. W. Mitchell, phys. stat. sol. (a) 135, (1993) 455. [2] J. C. M. Li, M. A. Imam, and B. B. Rath, J. Materials Science Letters 11, (1992) 906.

**11:00 AM V6.7**
**Molecular Dynamics Simulation of Deformation and Fracture in Nanocrystalline Ag and Nano-composite AgNi.** Yue Qi and Yang-Tse Cheng; Materials and Processes Lab, General Motors R&D, Warren, Michigan.

The deformation and fracture mechanisms of columnar nano-crystalline Ag and nano-composite AgNi have been studied using molecular dynamics. In addition to dislocation-mediated plasticity at an early stage of deformation, we found grain-rotation induced grain growth and crack formation at larger deformation. The crack nucleation at the grain boundaries and the linkage of such cracks will finally lead to the fracture of the material. However, the ductility of the nanocrystals is largely controlled by the competition between grain growth and crack nucleation. As a result, lower temperature, larger grain size and introduction of a second phase tend to accelerate crack formation and reduce the fracture strain, such decrease the ductility of the nanocrystals.

**11:15 AM V6.8**
**Molecular dynamics simulation of low-temperature plasticity in a model quasi-columnar nc-Si structure.**
Michael John Demkowicz and Ali S. Argon; Mechanical Engineering, MIT, Cambridge, Massachusetts.

Molecular dynamics simulations of low-temperature plasticity in a quasi-columnar atomic configuration representative of nanocrystalline Silicon are presented. The structure was created by filling a high-aspect ratio simulation cell with atoms in a diamond cubic configuration centered on randomly positioned and misoriented crystalline seeds. To ensure full kinetic equilibration, the structure was annealed at temperatures just below the glass transition of bulk amorphous Si for about 15ns. Although it cannot reflect the full range of possible nc-Si microstructures, this atomic configuration nevertheless can be used to gain insight into certain types of plasticity phenomena obtainable in single-component covalently bonded nanocrystalline solids. Deformation under volume conserving plane strain increments to high levels of plastic strain reveals a distinct yield phenomenon linked to the creation within the structure of a thin shear localization zone composed of easily flowing disordered material. Steady state plastic flow is achieved when a plane of material permitting easy shear flow percolates through the entire length of the system. Upon further deformation, new planes of shear localization replace previously formed ones that have rotated away from planes of maximum applied shear stress resulting in periodic rises and drops of overall plastic shear resistance with continued deformation. The effect of total externally applied pressure on the course of plastic deformation is also discussed.

**11:30 AM V6.9**
**Study of Deformation and Failure Mechanisms of Nanograined Thin Films Using *In – situ* TEM and a Novel MEMS Tensile Testing Device.** Khalid Mikhiel Hattar[1], Jong Hee Han[2], Taher A. Saif[2] and Ian M. Robertson[1]; [1]Material Science and Engineering, University of Illinois Urbana-Champaign, Urbana, Illinois; [2]Mechanical and Industrial Engineering, University of Illinois Urbana-Champaign, Urbana, Illinois.

The combination of a novel microelectromechanical system (MEMS) tensile testing device and *in – situ* transmission electron microscopy (TEM) is used to study the deformation and failure mechanisms and simultaneously measure the mechanical properties of nanograined and ultra-fine grained thin gold and aluminium films. The device allows

for uniaxial straining of a free-standing thin film within the confines of the TEM, making it possible to directly relate microstructural evolution to mechanical properties. The use of the TEM allows for detailed characterization of the thin film nanostructure, including dislocation pile-ups, twinning, grain rotation,and intergranular failure mechanisms. This unique combination provides insight into the grain size effect on mechanical properties, most notably in the controversial inverse Hall-Petch relationship.

**11:45 AM V6.10**
**Mechanical Properties and Deformation of Fine-Grained Ni by Dislocation and Grain-Boundary Processes.**
David M. Follstaedt[1], James A. Knapp[1], Zhiwei Shan[2], Scott X. Mao[2], Jorg M.K. Wiezorek[2], Richard C. Hugo[3] and Eric A. Stach[4]; [1]Sandia National Laboratories, Albuquerque, New Mexico; [2]University of Pittsburgh, Pittsburgh, Pennsylvania; [3]Portland State University, Portland, Oregon; [4]Lawrence Berkeley National Laboratories, Berkeley, California.

Thin layers of pulsed-laser deposited (PLD) Ni formed in high vacuum have been found to be fine-grained and equiaxed and with no voids at grain boundaries by plan-view and cross-section TEM. This nanostructure indicates that the material is ideal for exploring the intrinsic mechanical behavior of fine-grained Ni. Here we consider the mechanical properties of PLD Ni in light of deformation processes being found in the material. Nanoindentation (Knapp, J. Mater. Res. 2004) shows that the finest-grained Ni (10 nm) is harder (12 GPa) than fine-grained Ni produced by other methods; moreover, its hardness scaled according to the Hall-Petch relation (H = k/dî/2, d = grain diameter). Yield strengths of 4 GPa are found for this material by detailed finite-element modeling of its response to indentation. A first in situ TEM study of PLD Ni during tensile deformation (Hugo, Acta Mater. 2003) observed intense dislocation activity, and found multiple dislocations in grains as small as 10 nm. The material also appeared very ductile and the layer thinned in advance of a propagating crack during straining. In a second, recent in situ examination during tensile straining (Shan, submitted to Science 2004), clear evidence is found for rotation of numerous adjacent 10 nm grains to produce larger agglomerates with nearly aligned subgrains. Dark-field microscopy and frame-by-frame examination over an interval of <1 sec were required to detect the grain rotation ahead of an advancing crack. This study also finds dislocations trapped within small grains while the tensile strain is still applied. These observations from the same material together with theoretical understandings of the two processes indicate that Ni with 10 nm-size grains is at a cross-over from dislocation-based deformation to grain-boundary mediated deformation, even though any deviation from Hall-Petch scaling at this grain-size appears no more than 20%. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. SXM and JMKW gratefully acknowledge support for this work from the National Science Foundation (NSF-CMS). The work at NCEM was supported by the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

SESSION V7: Rate Effects; Phase Field Models; Amorphous Materials
Chairs: Dennis Dimiduk and Alan Needleman
Wednesday Afternoon, December 1, 2004
Room 206 (Hynes)

**1:30 PM V7.1**
**A Dynamic Finite Temperature Coupled Atomistic/Discrete Dislocation Method.**
Vijay Shastry[1], W. A. Curtin[1] and Ronald E. Miller[2]; [1]Division of Engineering, Brown University, Providence, Rhode Island; [2]Department of Mechanical and Aerospace Engineering, Carleton University, Ottawa, Ontario, Canada.

A method for simultaneously thermostatting an atomistic region and absorbing energetic pulses impinging on the atomistic/continuum interface from the atomistic region is developed to operate within the framework of the Coupled Atomistic/Discrete Dislocation (CADD) method. The approach inserts an additional Langevin damping term into the equations of motion for atoms in a boundary region near the atom/continuum interface, with the damping coefficient ramped linearly over the width of the region. The method is thus nearly identical to the "stadium boundary conditions" devised by Ravelo and Holian. The remaining interior atom dynamics are computed using a standard MD algorithm with no artificial damping or thermostatting. The continuum region deformations are computed using static FEM updated stochastically over time scales comparable to the Debye frequency of the atoms using time-averaged displacements at the atom/continuum interface, thereby providing an evolution of the continuum region that tracks the deformation but does not exchange heat with the atomistics. With an optimal maximum damping coefficient of approximately 1/3 of the Debye frequency, temperature stability is attained at values within 10% of the target temperature, with some system size dependence. For the same damping characteristics and at low temperature, high-energy elastic pulses propagate unimpeded up to the boundary region and then are completely damped out upon approach to the interface, with no measurable reflections. At higher temperatures, thermal fluctuations in the total energy make detailed comparisons of small versus large systems unreliable, although in general elastic pulses are damped out well.

**1:45 PM V7.2**
**Point defect dynamics in irradiated metals: Effect of elastic interactions and alloying.**
Joerg Rottler, Roberto Car and David J. Srolovitz; Princeton Institute for the Science and Technology of Materials (PRISM), Princeton University, Princeton, New Jersey.

The macroscopic mechanical properties of metals are intimately related to their microstructural features and their spatiotemporal evolution. In irradiated metals, the initiation of plastic yield is strongly influenced by the interactions of dislocations with other defects such as self-interstitial cluster. We present a statistical model for the dynamics of point defects in bcc metals that is solved through kinetic Monte Carlo (kMC) and rate equations. Self-interstitial atoms and vacancies can be produced in abundance upon irradiation with energetic particles, but they subsequently anneal due to recombination and absorption at sinks such as dislocations and grain boundaries. The model reveals a sequence of kinetic regimes that lead to a final steady state. We also determine the size distribution of voids that form when vacancies aggregate into cluster. The model is then extended to include long ranged elastic interactions between point defects and dislocations, which have so far mostly been ignored in kMC simulations of microstructural evolution. We also consider metal alloys, where interstitial and vacancy transport must be described through effective diffusivities and correlation effects become important.

**2:00 PM V7.3**
**Deformation Evidence in High Strain Rate Laser-Shocked Nanocrystalline Ni.**
Yinmin Wang[1], E. Bringa[1], M. Victoria[1], A. Hodge[1], J. McNaney[1], A. Caro[1], B. Torralva[1], B. Remington[1], R. Smith[1] and C. Schuh[2]; [1]Chemistry and Materials Science Directorate, Lawrence Livermore National Laboratory, Livermore, California; [2]Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

The current understanding on deformation mechanism of nanocrystalline metals relies heavily on molecular dynamics simulations, which are carried out at strain rates (typically $> 10^7$ $s^{-1}$) that are several orders of magnitude higher than those used in most experiments. Here we present experiments using high intensity laser loading of nc Ni samples that reaches strain rates above $10^6 s^{-1}$, making possible a direct comparison with molecular dynamics simulations. Materials recovered after the shock has been characterized using TEM/HRTEM and mechanical testing, including nanoindentation. In addition, we present simulations of shocks in nc samples, where the high, inhomogeneous stress state leads to nucleation of dislocations even for small grain sizes where grain boundary rotation was the only observed deformation mechanism in previous simulations. This effort would provide results for a unique and first time comparison with simulations spanning the relevant length and time scale covered in the experiments. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract of No.W-7405-Eng-48.

**2:15 PM V7.4**
**Effects of Lattice Incompatibility Induced Hardening on the Fracture Behavior of Ductile Single Crystals.**
Huang Tang[1], Amit Acharya[2] and Sunil Saigal[2]; [1]Mechanical Engineering, MIT, Cambridge, Massachusetts; [2]Civil and Environmental Engineering, Carnegie Mellon University, Pittsburgh, Pennsylvania.

Plane-strain mode-I cracks in a ductile single crystal are studied under conditions of small scale yielding. The specific case of a crack growing in the direction for an FCC crystal is considered. Crack initiation and its subsequent growth are computed by specifying a traction-separation relation in the crack plane ahead of the crack tip. The crystal is characterized by a hardening model that incorporates physically motivated gradient effects. Significant traction elevation ahead of the crack tip is obtained by incorporating gradient effects, allowing a better basis for the prediction of cleavage in the presence of substantial plastic flow. Resistance curves based on parameters characterizing the fracture process and the continuum properties of the crystal are computed. Simulation results indicate that the

length-scale of the lattice incompatibility dominated region has to be comparable or larger than the length of the fracture process zone for gradient effects to have a significant effect on fracture resistance. Both the work of separation and the peak separation stress also play important roles in determining the fracture resistance of the crystal.

**2:30 PM V7.5**
**Material Length Scales in Crystal Plasticity.** Marisol Koslowski, Los Alamos National Laboratory, Los Alamos, New Mexico.

Crystal plasticity involves complicated non-local non-linear mechanisms at different length scales such as the long range dislocation-dislocation interaction, the interaction of dislocations with obstacles, and the formation of microstructures. Crystal plasticity models have been phenomenological, based primarily on empirical laws and averaging over dislocations and their interaction with other crystal defects. Recently, micron-scale experiments have shown that classical models are unable to represent key features on the behavior of deformed crystalline materials as for example size effects. To fully understand the dynamics of crystal plasticity, experiments and simulations have to be able to explore the problem with the resolution necessary to study the key element of plastic flow, the microstructure. This study develops a model to yield a complete characterization of crystal plasticity at all relevant length scales. In particular we use a phase-field model to track individual dislocations and we integrate it into a classical model of single-crystal plasticity. We also make contact with other theories and compute the material length and material strength parameters introduced in non-local models of plasticity.

**3:15 PM V7.6**
**Structural Changes Associated with Homogeneous and Inhomogeneous Flow in Bulk Metallic Glasses.**
Matthew Lambert and Katharine M. Flores; The Department of Materials Science and Engineering, The Ohio State University, Columbus, Ohio.

Bulk metallic glasses (BMGs) represent the limiting case of grain size refinement in metallic materials. Due to their unique, disordered atomic structure, BMGs exhibit strengths on the order of 2 GPa, extraordinary elastic strains on the order of 2%, and fracture toughness values comparable with other metallic alloys. Lacking crystalline defects, BMGs exhibit plastic flow behavior very different from their crystalline counterparts. Flow is thought to be the result of a diffusion-like process involving the "free volume" of the glass. It is well known that at low temperatures and high stresses, plastic deformation is highly localized in shear bands, while at temperatures near the glass transition, deformation becomes homogeneous. The free volume theory for flow indicates that the average free volume increases during flow, and prior studies of free volume distribution suggest that the sizes of free volume sites range from inherent interstitial-like defects to larger defects capable of flow. However, the precise flow mechanism and the evolution of the free volume distribution during flow remain unclear. An examination of structural changes during flow at varying strain rates and temperatures is therefore of interest. In this study, a Gleeble® thermomechanical test system was used to investigate the inhomogeneous-homogeneous flow transition in Zr-based bulk metallic glass. The tensile and compressive behavior was characterized at a variety of strain rates and temperatures. The average free volume before and after deformation was quantified using Differential Scanning Calorimetry. Previous results revealed that 30% inhomogeneous plastic strains achieved by rolling increased the average free volume by 4%, consistent with the free volume theory of flow. More detailed information about the evolution of the free volume distribution was obtained using Positron Annihilation Spectroscopy, where an increase in the concentration of flow defects was observed following deformation. The effect of structural relaxation due to annealing will also be discussed.

**3:30 PM V7.7**
**Ductile Bulk Metallic Glass Foams.** Alan H. Brothers and David C. Dunand; Materials Science, Northwestern University, Evanston, Illinois.

Fully amorphous open-celled foams are processed from the commercial bulk metallic glass-forming alloy Vit106 ($Zr_{57}Nb_5Cu_{15.4}Ni_{12.6}Al_{10}$) using the salt replication method with stable refractory BaF2 salt. Foam structures are highly uniform, with pore sizes on the order of 100-300 $\mu$m and relative densities as low as 15%, and can be cast in diameters as large as 7 mm without crystallization. Despite a lack of significant plasticity in the monolithic alloy, the foamed alloy is ductile in compression due to the unique mechanics of shear banding in thin (<1 mm) amorphous metal struts deformed in bending, opening a wide potential market for these materials in the automotive, aerospace, and biomedical industries. Processing of these novel materials will be discussed, and the effects of pore size and foam density on mechanical properties reviewed.

**3:45 PM V7.8**
**Finite Size Effects on the Structure of Grain Boundaries.**
Emmanuelle Marquis, John Hamilton, Douglas L. Medlin and Francois Leonard; Materials Physics, Sandia National Laboratories, Livermore, California.

As the grain size in a polycrystalline material is reduced, the coupling between the grain boundaries and grain boundary junctions becomes increasingly important. In particular, in nanocrystalline materials, the geometrical incompatibilities that arise at a boundary junction can lead to stress fields that extend over distances that are comparable to the boundary facet itself. Here, we explore the impact of this coupling through a combined experimental and theoretical analysis of grain boundary facets of finite, nanometer-scale dimensions. In particular, we focus on the variation of translation state across Sigma=3 {112} grain boundaries in gold. Our high-resolution electron microscopy (HREM) measurements show a continuous variation in the local lattice translations across such boundaries that depends both on the total length of the boundary as well as on the position with respect to the boundary junction. These measured structural profiles are directly explained using a combination of continuum elasticity theory and first-principles calculations. In particular, we show quantitively how the observed behavior originates from a competition between elastic energy and the energy cost of forming continuous {111} planes across the boundary, a result that provides a method for experimentally estimating the energy barrier of the grain boundary gamma surface. The theoretical analysis also predicts a structural transition between offset-free and non-trivial grain boundary structures at a critical grain boundary size, in agreement with the experimental observations. This work is supported by the U.S. Department of Energy, in part by the Office of Basic Energy Sciences, Division of Materials Sciences, under contract No. DE-AC04-94AL85000.

**4:00 PM *V7.9**
**A Three-Dimensional Multi-Phase Field Model of Dislocation Dynamics and Plasticity in Crystals.** Michael Ortiz and M.P. Ariza; Aeronautics, California Institute of Technology, Pasadena, California.

We present a model of discrete dislocation dynamics based on: a lattice-statics representation of crystal elasticity; and the introduction of eigen-deformations in order to represent the lattice-invariant or plastic deformations of a crystal. The formulation is fully three-dimensional and accounts for the complete complement of slip systems in bcc crystals, including (110) and (112) slip. We show that all plastic deformations can be represented in terms of a number of integer-valued phase fields, one per slip system, measuring the local slip activity. In particular, the value of a phase field at a point on a slip plane counts the number of dislocations in the corresponding slip system which have passed over the point. The present formulation may be regarded as a three-dimensional, multislip generalization of the phase-field theory of dislocation dynamics of Koslowski et al. (2002). However, the present theory differs in the important respect of being based ab initio on a discrete lattice representation of the crystal. This renders dislocation core structures well-defined and obviates the need for postulating a Peierls potential. We show that, within the confines of the theory, discrete dynamics can be given a compelling interpretation in terms of discrete differential operators. Dislocation junctions and the degree of entanglement of the dislocation ensemble can also be understood in terms of homology groups and topological invariants, leading to a succinct formulation of forest hardening. A chief advantage of the theory is that it is analytically tractable. We take advantage of this feature to provide explicit solutions of deformation-induced sub-grain dislocation structures.

**4:30 PM V7.10**
**Enhanced Rate Sensitivity from Nano-sized Twins in Pure Copper.** Lei Lu[1,2], Ruth Schwaiger[3], Ming Dao[1] and Subra Suresh[1]; [1]Dept of Materials Science & Engineering, MIT, Cambridge, Massachusetts; [2]Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China; [3]Forschungszentrum Karlsruhe, Institute for Materials Research II, Karlsruhe, Germany.

Conventional coarse-grained Cu at room temperature is not particularly rate sensitive. However, for the nanocrystalline metals, with grain size typically smaller than 100 nm, an obvious strain rate or load rate sensitivity has been observed in nanocrystalline Cu. It is also known that significant strengthening arises from the formation of nano-scale twins in coarser grained copper. However, it is presently unknown whether such nano-scale twins also promote rate sensitivity. The objective of the present study is to explore the possibility of increased rate sensitivity induced by nano-scale twins. By using the pulsed electro-deposition technique, a pure Cu sheet was synthesized with a unique microstructure: a high density of twins with thickness in the nanometer scale confined in submicron-sized grains. The loading rate sensitivities of the copper samples with different densities

of twin boundary were studied by depth-sensing nanoindentation. Systematic experimental and associated computational simulations reveal for the first time that there is a significantly higher loading rate sensitivity (almost 6 times higher) for the Cu sample with a high density of twin boundaries than that for the coarse-grained counterpart with essentially no twins. With a decrease of twin boundary density, the rate sensitivity of the Cu sample also decreases. Possible mechanisms for the enhanced rate sensitivity of copper due to the introduction of nano-scale twins are also discussed.

**4:45 PM V7.11**
**Computational Modeling of Significantly Increased Strength and Rate Sensitivity due to Nano-sized Twins in Pure Copper.** Ming Dao[1], Lei Lu[1,2] and Subra Suresh[1]; [1]Dept of Materials Science & Engineering, MIT, Cambridge, Massachusetts; [2]Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, Liaoning, China.

A physically-motivated crystal plasticity model of polycrystalline pure Cu with a high concentration of nano-sized twins is developed to study the significantly increased plastic strength and rate sensitivity. To model the microstructure of pure Cu with nanoscale twins, the orientation and size dependent plastic behavior parallel (plastically softer) and perpendicular (plastically harder) to the twin boundaries is specifically modeled and parametric studies are performed. Comparing with recent experimental results on pure Cu with nano-sized twins, the parametric studies show that the proposed model can correctly capture the plastic strength and rate dependence versus nanoscale twin thickness, i.e. both strength and rate sensitivity are significantly increased with higher twin density. A discussion of the possible influences on the ductility due to the presence of many nano-sized twins is also presented.

SESSION V8: Nanoindentation
Chairs: Marc Fivel and Lyle Levine
Thursday Morning, December 2, 2004
Room 206 (Hynes)

**8:30 AM V8.1**
**Strength is Relative: Plastic Deformation as a Function of Nano-, Micro- and Macrostructure in Metals.** Sedina Tsikata, Yoonjoon Choi, Subra Suresh and Krystyn J. Van Vliet; Materials Science & Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

The onset of plasticity in face-centered cubic (FCC) metals can be investigated through localized surface deformation experiments such as nanoindentation. Through nanoindentation experiments, computational simulations and simple material models of FCC metals, we find that this onset is a strong function of the length scale of the deformed material volume, relative to microstructural dimensions such as grain size and macrostructural dimensions such as metal line height and width. Although this size dependence cannot be explained through continuum approaches, these features are predicted by atomistic analysis. We discuss examples of this approach including microscale patterned lines and nanostructured thin films. We find that lines comprising FCC metals are less resistant to plastic deformation than films of the same metal and same microstructural lengthscales, due to dislocation-mediate stress relaxation. Further, we find that bulk and thin film FCC metals are less resistant to plastic deformation for average grain sizes below a critical value of approximately 10 nm, due to a change in deformation mechanism.

**8:45 AM V8.2**
**Study on the indentation size effect in ultrafine-grained metals.** Karsten Durst, Bjoern Backes and Mathias Goeken; Materials Science, University Erlangen, Erlangen, Germany.

Metallic materials show an increasing hardness with decreasing indentation depths, which is referred to as the indentation size effect (ISE). The Gao and Nix model relates the indentation size effect to geometrically necessary dislocations (GND), which density is proportional to the inverse of the indentation depth. The additional hardening due to the GNDs is most pronounced in soft annealed metallic single crystals, whereas the effect is greatly reduced after cold working. This can be correlated to the change in inner length scale of the material due to the increased dislocation density as well as the higher strength. To study the ISE and thus the change of the inner length scale of metals as a function of deformation, nanoindentations were performed in copper and brass in a soft annealed state, after cold-working and in the highly deformed ultrafine-grained state of the materials produced by equal channel angular pressing. We find that for coarse-grained materials, the effect of the GNDs is greatly reduced with some pre-straining and that the hardness is nearly constant at indentation depths of bigger than about 1 $\mu$m. The hardness of ultrafine-grained materials is nearly independent of indentation depth even at a contact depth of 200 nm. In the coarse-grained materials, the dislocation cell structure most probably governs the inner length scale, whereas in the ultrafine-grained materials, the grain boundaries play an important role, reducing the inner length scale well below 0.1 $\mu$m.

**9:00 AM V8.3**
**Mechanical Testing of Nano-Films Using a Spherical Indenter.** Chad Randall Sager[1], Zuhaib Sheikh[1], Thomas J. Mackin[1] and Matthew Begley[2]; [1]Mechanical and Industrial Engineering, University of Illinois Urbana-Champaign, Urbana, Illinois; [2]Civil Engineering, The University of Virginia, Charlottesville, Virginia.

ABSTRACT We present a novel experimental device for indentation testing of a freestanding circular membrane with a microfabricated fixed-fixed beam. Experiments of this type allow us to extract the mechanical properties as well as the residual stress of nano-films. Analytical solutions have been calculated to predict the relationships between load, contact radius, finite indentation strains (and stresses), pre-stretch, and deflection. These closed-form predictions include the important effects of the finite contact area which can lead to important differences in the load-deflection and indentation strain. These effects are excluded in classical point-load models (i.e. classical Schwerin-type solutions). INTRODUCTION Knowledge of the mechanical properties of thin films are important to Integrated Circuits (IC's), thin film optics, MEMS, as well as other areas. Though bulk testing of materials, and therefore bulk mechanical properties, are well established, testing of thin films is not. Many challenges exist in the testing of thin films. There are known effects of differing length scales, native oxide effects, residual/intrinsic stresses due to deposition/growth techniques, not to mention the added complexities due to the layering of thin films. Many investigators have studied the properties of thin films on substrates and have struggled with separating the film/substrate properties. The only way to alleviate this problem is to study freestanding films. Recent efforts have addressed this by utilizing a range of test methods, including: the uniaxial tensile test, bending of a cantilevered beam, and the bulge test. The bulge test is attractive in many respects, but pressurized testing requires nearly defect-free films (i.e. without pinholes or porosity), and therefore is not feasible for many material systems, notably polymers and porous low-k dielectrics. Begley and Mackin recently developed a closed form solution for finite-radius contact indentation testing of free standing films, and have verified the analysis using a host of materials (a paper to appear in The Journal of Mechanics and Physics of Solids). More recently, our group devised a methodology of testing thin circular membranes with microfabricated fixed-fixed beams. This method utilizes a loading beam that is connected to a fixed-fixed beam at its midpoint. The beams are made of Single-Crystal Silicon with known dimensions following standard microfabrication procedures. This new test system allows us to accurately determine the load applied to the circular thin membrane film as well as the deflection at the loading point. The loading beam is positioned over circular thin film membrane with a calibrated piezoelectric stage. Deflection of the beam is read from a cofabricated vernier scale and the difference between the piezoelectric stage movement and the vernier scale, provides both the applied load as well as the membrane's deflection.

**9:15 AM V8.4**
**Effect of Grain-Boundary Relaxation on Mechanical Properties of Nanocrystalline Fe.** D. Jang[1] and M. Atzmon[1,2]; [1]Department of Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan; [2]Department of Nuclear Engineering and Radiological Sciences, University of Michigan, Ann Arbor, Michigan.

In nanocrystalline materials, grain boundaries occupy a significant fraction of the volume. When the grain diameter approaches 10 nm, such materials exhibit plastic behavior that is significantly different from that of large-grained materials. In our previous work,* using nanoindentation of nanocrystalline Fe formed by ball milling, we observed a negative slope of the Hall-Petch plot. The hardness peaked for a grain size in the range of 12-18 nm, reaching a value 5 times that for large-grained Fe. We also observed a strain-rate sensitivity that increased with decreasing grain size. We attributed these observations to grain-boundary sliding being the dominant deformation mechanism at small grain sizes. Our results also suggested that the strain-rate sensitivity of samples of the same grain size depended on their respective thermal history. One possible interpretation of this observation is that the structure of the grain boundary is not unique, and that it can undergo relaxation and densification. Atomistic simulations by Hasnaoui et al.** predict that grain-boundary relaxation, resulting from heat treatment, leads to strengthening. In the present study, we have formed nanocrystalline Fe, with a volume-averaged grain size of 10 nm, by ball milling. The grain size was determined by the Warren-Averbach method of Fourier analysis

of diffraction data. These samples were subjected to low-temperature anneals, which resulted in negligible changes in the grain size, and slight changes in the RMS strain. While the hardness underwent little change, the strain-rate sensitivity, as measured by nanoindentation, increased significantly, and subsequently decreased to below the initial value, as a function of annealing time. The kinetics of this evolution were a strong function of the annealing temperature. These data, together with structural analysis by transmission electron microscopy, will be presented. * D. Jang and M. Atzmon, J. Appl. Phys. 93, 9282 (2003). ** A. Hasnaoui, H. Van Swygenhoven and P.M. Derlet, Acta Mater. 50, 3927 (2002).

**9:30 AM V8.5**
**An Experimental Study of Length Scale Effects in Plasticity for Ni Thin Films.** Jun Lou[1] and Winston O. Soboyejo[2]; [1]Brown University, Providence, Rhode Island; [2]Dept. of Mechanical and Aerospace Engineering, Princeton University, Princeton, New Jersey.

In this study, several experimental techniques were used to investigate plasticity at micron and sub-micron scales for LIGA Ni MEMS materials and single crystal Ni thin films. Micro-bend experiments and a newly-developed cyclic micro-bend technique were used to study the plastic behavior of LIGA Ni thin films under both monotonic and repeated loading. The observed length scale effects were analyzed within the frame work of the phenomenological Fleck-Hutchinson strain gradient plasticity (SGP) theory. Interestingly, the observed length scale effects diminish, as the number of bending cycles increase. Multi-scale indentation experiments were also performed on both LIGA Ni MEMS materials and single crystal Ni thin films. A bi-linear indentation behavior was observed for both materials indented between the micron and nano-scales. The measured indentation size effects were then discussed in light of insights from direct discrete dislocation simulations.

**9:45 AM V8.6**
**Edge Effect During Nanoindentation of Thin Copper Films.** Yakov Michailovich Soifer[1], Armen Verdyan[1], Michael Kazakevich[2] and Eugen Rabkin[2]; [1]Department of Sciences, Holon Academic Institute of Technology, Holon, Israel; [2]Material Engineering, Technion, Haifa, Israel.

The effect of geometrical confinement on mechanical response of the material was investigated. We used nanoindentation as a probe for studying mechanical properties of the polycrystalline copper film deposited on Si substrates and patterned into 4-8 micrometers wide stripes. The dependence of mechanical properties of the strip on a distance from the strip edge was studied. This approach allowed us to vary systematically only one of two size limiting parameters, since the film thickness (300 nm) was constant. The load-displacement P(h) curves and corresponding indent images for different loads and different distances from the strip edge were studied by the nanoindenting atomic force microscopy (AFM) and by Hysitron TriboscopeTM nanoindenter. 1. It was found that starting from some threshold distance (which is determined by the load applied) the indentation compliance decreased with decreasing distance between indent center and the strip edge. For indents centered approximately at the strip edge the indentation compliance was by a factor of 2-3 lower than the one far from the strip edge. 2. Correspondingly, plastic deformation increased as indents approached the strip edge and reached a very high value just at vicinity of the strip edge. 3. Unloading parts of the P(h) curves of the indents in the vicinity of strip edge were different from those measured for indents far from strip edge and showed significant influence of restoring forces. 4. AFM images of the indents revealed characteristic behavior of the material at the strip edge. Necking and fracture of the thin walls separating the indent from the strip edge were observed on the nanometer scale. The critical indentation parameters were determined and the results obtained are discussed in the terms of the microscopic deformation mechanisms describing the interaction of the elastic-plastic boundary beneath the indenter with the edge of the strip.

**10:30 AM V8.7**
**Indentation Size Effects in Magnesium Oxide.** Luc Jean Vandeperre, Finn Giuliani and William John Clegg; Materials Science and Metallurgy, University of Cambridge, Cambridge, United Kingdom.

There are many reports of an increase in hardness when the load is decreased. This increase in hardness has been attributed to a scale effect in the resistance to plastic deformation and is consistent with several theoretical predictions. However, it has also been suggested that the size effect only arises due to measurement errors, and this is consistent with the observation that the size effects are observed both using micro-indenters as well as using nano-indenter, i.e. at load ranges and deformation volumes, which differ by at least an order of magnitude. In this contribution, a systematic range of indentation experiments on single crystal magnesium oxide will be described, which were designed specifically to differentiate between true size effects and other factors, which can give rise to an apparent increase in hardness. The load-displacement data is analysed using a new analytical expression for the loading curve, which allows more information to be extracted from the data collected during indentation. The topography of the indents was characterised using scanning electron microscopy and atomic force microscopy and the deformation processes underneath the indent were determined by transmission electron microscopy. It is shown that even when care is taken to remove measurement errors there is an apparent increase in hardness as the applied load decreases. However, the results also suggest that these arise due to contributions both of the strain-rate sensitivity of the flow stress of MgO, and the blunted tip of the indenter rather than being caused by a pure size effect.

**10:45 AM V8.8**
**An Elastic/Plastic Hardness Stress/Strain Description of an Indentation Size Effect.** Ronald W. Armstrong[1], Louis Ferranti[2] and Naresh N. Thadhani[2]; [1]Mechanical Engineering, University of Maryland, College Park, Maryland; [2]School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Current applied force and displacement sensitivities for measuring the load/penetration behavior in continuous ball-type indentation tests, allows tracking of the elastic-to-plastic transition in indentation behavior [1]. The measurements provide for definition of a hardness stress/strain curve by taking the stress as the mean pressure on the contact surface and the corresponding average strain to be proportional to the ratio of the contact diameter to ball diameter [2], with the contact diameter obtained generally through measurement of indentation depth. Such strain definition easily relates to the residual indentation area being employed for high-load conventional hardness values because the projected contact area of a plastic indentation is essentially unchanged during elastic unloading. For relatively hard materials, the elastic and plastic strains even for conventional sharp-pointed indenters are comparable [3]. Neglect of the elastic contribution to load support at the earliest stages of plastic deformation provides one explanation of a greater hardness being obtained if the value is based only on the residual indentation size. Nanoindentation hardness testing provides an excellent method of following the elastic/plastic transition on the proposed hardness stress/strain basis. In this paper we will present the elastic/plastic hardness stress/strain description for various types of soft and hard, single- and multi-phase materials and discuss the effect of determining the contact area diameter, "d", from the depth of penetration, "h", in comparison to its direct measurement. References 1. L. Ferranti, Jr., R.W. Armstrong, and N.N. Thadhani, Mater. Sci. Eng. A371, 251 (2004). 2. B.L. Hammond and R.W. Armstrong, Philos. Mag., 57, 41 (1988). 3. L. Ferranti, Jr., R.W. Armstrong, and N.N. Thadhani, submitted for the Eighth Intern. Conf. Sci. Hard Mater. (ICSHM8), November 8-12, 2004, San Juan, P.R.

**11:00 AM V8.9**
**Size Effects in Localized Plastic Phenomena: Quasicontinuum Study of Void Growth and Nanoindentation.** Jaime Marian, Jaroslaw Knap and Michael Ortiz; California Institute of Technology, Pasadena, California.

There are many processes in engineering occurring at the nanoscale which are known to have macroscopic implications. The multiscale nature of these processes requires the use of computational tools that are able to connect phenomena across the different temporal and spatial scales involved. In this paper we apply the Quasicontinuum (QC) method to study two important cases in which localized plastic activity affects the macroscopic response of materials. One such case is void growth, which is known to be a critical phenomenon regarding fracture in ductile materials. Voids generated preferentially at mechanically-weak spots of the material grow by dislocation emission and their growth depends acutely on parameters such as void size, shape and loading conditions. Here we apply QC to obtain the operating dislocation emission mechanisms and the corresponding yield maps for different void sizes in Al. Similarly, nanoindentation, where dislocations structures generated beneath the impinging indenter influence mechanical properties such as the material hardness, can be regarded as another case in which atomic-scale defects have a macroscopic effect. In this paper we use QC to assess the influence of the indenter's size on the measured force-displacement curve of Au[001] and provide comparison with experiments.

**11:15 AM V8.10**
**Dislocation Structure Evolution during Nano-Indentation using Multi-scale Discrete Dislocation Plasticity Analysis.** Shafique M.A. Khan and Lyle Levine; National Institute of Standards and Technology, Gaithersburg, Maryland.

With the growing applications of nano-scale components in various technologies like MEMS, thin films, medical diagnostics etc,

nano-indentation testing is finding new expanding opportunities. In addition, it represents a 'non-destructive' testing technique for macro applications since mechanical properties of bulk material can be extracted from nano-indentation testing using appropriate models. Thus accurate models of the nano-indentation process are crucial. Therefore, in the present study, we closely analyze the dislocation structure evolution during nano-indentation using a multi-scale discrete dislocation plasticity simulation code. This code couples two length scales, discrete dislocation dynamics and continuum finite element. Finite element is responsible for applying the model boundary conditions and to simulate the nano-indentation load, whereas discrete dislocation dynamics is responsible for dislocation motion. Results will be presented investigating the effect of nano-indentation on the evolution of the initial random dislocation distribution.

**11:30 AM *V8.11**
**Indentation Size Effect Studied by Discrete Dislocation Dynamics.** Chan S. Shin[1], Marc C. Fivel[1] and Marc Verdier[2]; [1]GPM2, CNRS/INPG, St Martin d Heres, France; [2]LTPCM, CNRS/INPG, St Martin d Heres, France.

Indentation size effect (ISE) corresponds to the increase of hardness when the indentation depth decreases. Although the first observations of this effect were mainly due to artefacts such as a bad surface preparation or a poor shape calibration, the ISE remains measurable when great care is paid to the experiment procedure. The depth at which the ISE is observed is only decreased. Recent explanations invoke strain gradient theory and geometrically necessary dislocations. In this study, discrete dislocation dynamics (DDD) simulations are performed to study the ISE in fcc materials. The work is derived from a first study [Fivel et al 1998]. The code has recently been improved so that the actual shape of the indented surface is computed and updated during the indentation process. This gives access to the real contact area between the indenter and the material which allows deriving the exact hardness of the material. First, DDD results are compared with experiments performed on Au and Cu single crystals. The pile-up and sink-in at the indented surface are correctly reproduced by the DDD code. In a second time, different strain gradient model of ISE are checked using the DDD results. Quantities like statistically and geometrically dislocation densities are computed from the DDD results which give a length scale used in the strain gradient model. Fivel M.C., Robertson C.F., Canova and Boulanger L., Acta Mater., 46(27), pp. 6183-6194, 1998.

SESSION V9: Size Effects in Alloys/Composites
Chairs: Fereshteh Ebrahimi and Krystyn Van Vliet
Thursday Afternoon, December 2, 2004
Room 206 (Hynes)

**1:30 PM V9.1**
**Correlated Transmission of Slip between Lamellae in $\gamma$-TiAl.** M. Sundararaman[2], J. B. Singh[2], A. Couret[3], G. Molenat[3] and Patrick Veyssiere[1]; [1]LEM, CNRS-ONERA, Chatillon, France; [2]Materials Research Division, Bhabba ATomic Reserach Centre, Mumbai, India; [3]CEMES, CNRS, Toulouse, France.

In lamellar TiAl, deformation occurs by propagation of 1/2<110] dislocations and twins across 6 possible orientational variants with $L1_0$. After special annealing conditions, the alloy consists of a majority variant, say O, together with its twin-related variant $O_T$ in lesser proportion though. The mean lamella thickness is of the order of 100nm. In the case of adjacent $O/O_T$ variants, it is shown experimentally that when slip obeys the Schmid law in either O or $O_T$, this is not necessarily the case in the adjacent $O_T$ (resp. O). In the latter variant, slip is always related geometrically to the primary slip mode of the former variant. This relationship not only involves the slip planes (imposed by twin symmetry) but also the slip direction. In the case of 1/2<110] dislocations, the Burgers vectors operating in both variants make the same angle to the interface plane, although slip systems with higher Schmid factor may be available in the second variant. The Schmid law is violated and this is independent of lamellar thickness. The property is interpretd in terms of pilot and driven orientations. Possible implications in the deformation of polycrystals are discussed.

**1:45 PM V9.2**
**The Effect of Grain Size on the Mechanical Response of Pure Aluminum.** Pei-Ling Sun, Ellen Cerreta and George Thompson Gray; MST-8, MS G755, Los Alamos National Laboratory, Los Alamos, New Mexico.

Commercial purity aluminum AA1050, was subjected to equal channel angular extrusion (ECAE), resulting in an ultrafine grained (UFG) microstructure with an as-received grain size of 0.35$\mu$m. This material was then annealed to obtain microstructures with grain sizes ranging from 0.78 to 20$\mu$m. Compression tests were conducted at quasi-static and dynamic strain rates and temperatures of 77 and 298K. The mechanical properties were found to vary significantly with grain size, strain rate, and temperature. At room temperature and the smallest grain sizes, the specimens displayed a negative work hardening rate at low strains. With increasing grain size, the rate of work hardening increased. At 77K, the work hardening behavior was seen to be less influenced by the microstructure. All specimens displayed positive work hardening rates during deformation, however, work hardening rates did increase with increasing grain size. It is believed that the coarse grain size specimens, which possess larger volumes for dislocation accumulation during deformation, result in a higher rate of work hardening than in the UFG specimen.

**2:00 PM V9.3**
**Particle Size Effects on Mechanical Properties of Aluminum/Epoxy Particulate Composites.** Jennifer L. Jordan[1], Clive R. Siviour[2] and D. Wayne Richards[1]; [1]MNME, Air Force Research Laboratory, Eglin AFB, Florida; [2]PCS Group, Cavendish Laboratory, University of Cambridge, Cambridge, United Kingdom.

The Air Force has ongoing efforts in the development of particulate composite materials for aerospace applications. The effect of particle size on the mechanical properties of these particulate composites has not been widely studied across a large range of strain rates. Aluminum/epoxy composites were prepared using several different particle sizes at the same solids loading density. This study describes results of mechanical tests on these aluminum/epoxy particulate composites across a wide range of strain rates from $10^{-2}$ to $10^5$ using conventional Instron and novel split Hopkinson pressure bar techniques.

**2:15 PM V9.4**
**Size Effect on the Plasticity of Cu/Nb and Cu/Ta Nanocomposite Conductors: In-Situ Tem and Nanoindentation Studies.** Ludovic Thilly[1], Vanessa Vidal[1,2] and Florence Lecouturier[2]; [1]Universite de Poitiers, Laboratoire de Metallurgie Physique, Futuroscope, France; [2]INSA, Laboratoire National des Champs magnetiques pulses, Toulouse, France.

Nanofilamentary copper/niobium and copper/tantalum conductors were elaborated by severe plastic deformation (repeated drawing) for the winding of resistive coils producing non-destructive high magnetic fields with long pulse duration. This application requires both very high strength and high electrical conductivity properties. The first generation of Cu/Nb nanocomposites, constituted with $55^4 = 9.10^6$ nanofilaments embedded in the Cu matrix, exhibit an ultimate tensile strength of 2 GPa at 77K, for a conductor with a 0.04 mm$^2$ section, containing Nb nanofibres with a diameter of 40 nm. To improve this result, the effect of heavy deformation as well as the effect of microstructure refinement on mechanical properties was studied thanks to post-mortem electronic and atomic microscopy and in-situ deformation. The later revealed the occurrence of a single dislocation regime in the Cu matrix at nanometre scale, that was modelled to simulate the mechanical properties of the Cu/Nb nanocomposite conductors. The model constitutive laws were also used to define the engineering parameters for mechanical optimisation [1]. Consequently, second generation Cu/Nb conductors were elaborated: they contain $85^4 = 52.2\ 10^6$ Nb nanofilaments with diameter down to 25 nm. For a section of 5 mm$^2$, the ultimate tensile strength at 77K is 1.9 GPa. Concurrently, a "co-cylindrical" structure was designed: the Cu matrix contains $85^4$ Nb nanotubes filled with Cu nanofibres. New Cu/Ta were also elaborated, in order to optimize the whiskers effect, and are currently characterized [2]. To bring further elements to the understanding of the high strength of the Cu-based nanocomposites, the effect of microstructure dimension $\delta$ on their plasticity was studied by nanoindentation. Indeed, these materials are very good candidates for such a study because of their multi-level geometry which allows for the scanning of the entire range of microstructure dimension, from macroscopic to nanometre size [3]. For $\delta > 10\mu$m, no size effect on the composite hardness is observed. In the [1$\mu$m-10$\mu$m] range, a strong increase in hardness indicates a change in plasticity mechanism, attributed to the classical Hall-Petch grain size strengthening. In the nanometre range, the hardness of the nanocomposite Cu/Nb or Cu/Ta regions exceeds that of nanocrystalline bulk counterparts. For the finest structure a reduction of the hardness was observed in the nanocomposite zone: this softening may be attributed to intermixing at the Cu-Nb interfaces as previously evidenced by Tomographic Atomic Probe experiments. The observed size effect on the plasticity of Cu/Nb and Cu/Ta nanostructures added to the dislocation barrier role of interfaces confirm previous analyses based on the occurrence of a single dislocation regime at nanometre scale associated with impenetrable inter-phase interfaces. [1] Phil. Mag. A, 2002, 82, 925; [2] Adv. Eng. Mat., 2004, 6, 290; [3] Acta Mater., 2002, 50, 5049.

**2:30 PM V9.5**
**Effect of Annealing on Strength and Ductility of a Nanocrystalline Ni-Fe Alloy.** Fereshteh Ebrahimi and Hongqi Li; Materials Science and Engineering, University of Florida, Gainesville, Florida.

Nanocrystalline face centered cubic (FCC) Ni-15%Fe alloy with an average grain size of 9nm was fabricated via the electrodeposition technique. Dog-bone shaped tensile specimens with a 5mm gauge length were prepared and annealed for 90 minutes at 250, 400 and 500C in encapsulated quartz tubes. The strength was also evaluated using nano- and mico-indentation techniques. The grain size, lattice parameter and internal stresses were evaluated by x-ray diffraction (XRD). Transmission electron microscopy (TEM) was employed to measure the grain size distribution and fracture surfaces were examined using scanning electron microscopy (SEM). The tensile results were consistent with hardness data in revealing an inverse Hall-Petch relationship. While the as-deposited samples showed impressive tensile elongation, the annealed samples fractured immediately after yielding. However, the ductility, as evaluated by the reduction in area, was increased drastically after annealing at 500C. In this presentation the variations in strength, tensile elongation and ductility upon annealing are discussed in terms of dislocation generation and connectivity of large grains.

**2:45 PM V9.6**
**MD Modeling of Al-Si Nanocrystalline Plasticity.**
Donald K. Ward[1], Yue Qi[2] and W. A. Curtin[1]; [1]Brown University, Providence, Rhode Island; [2]General Motors, Warren, Michigan.

Molecular Dynamics is used to examine the plastic deformation of nanoscale Al-Si crystals under tension until fracture. An MEAM potential that accounts for the directional dependence of Si describes the forces and energies in the model. Crystals are created using a 2D Voronoi cell generator, allowing for a range of geometries including hexagonal grains, with aligned slip systems, as well as random configurations. A columnar grains structure with [011] orientations is used to allow for the greatest number of active slip systems, while 3D periodic boundary conditions accommodate the lattice mismatch. This study focuses on the effect; volume ratios, grain size, grain orientation, grain shapes, overall model size, and other characteristics can have on the overall plasticity of the material. This study will also further examine the mechanisms of dislocation deformation and grain boundary sliding during nanoscale plasticity

**3:30 PM V9.7**
**Mechanical Properties of Ni-W Alloys Above and Below the Hall-Petch Breakdown.** Andrew J. Detor and Christopher A. Schuh; Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Recent advances in electrodeposition have enabled the production of high quality materials with precisely controlled characteristic nanostructural length scales. In the present study, bulk nanostructured Ni-W alloys are produced via pulsed electrodeposition, in order to systematically study the effect of grain size on plasticity in the range of the Hall-Petch breakdown. The nanostructure of these alloys is shown to be controlled by the tungsten alloying addition, and rationalized by a thermodynamic treatment of grain boundary segregation. Mechanical properties including microhardness as well as tensile strength and ductility, are investigated over a range of grain sizes from 50 nm down to the amorphous limit ( 1 nm). This data set offers strong evidence for the breakdown of Hall-Petch scaling, beginning at grain sizes below about 10 nm. Detailed characterization of tensile specimens, through electron microscopy, x-ray diffraction, and chemical analysis, helps elucidate the microstructure, failure mode, and plastic response of these materials.

**3:45 PM V9.8**
**Bauschinger Effect in Thin Metal Films.** Yong Xiang and Joost J. Vlassak; Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

Discrete dislocation simulations of plastic deformation in thin passivated films often show a large Bauschinger effect. Until now, there has been no direct experimental evidence for this behavior. We have developed a new experimental technique that allows us to deform thin films alternating in tension and compression and to measure the corresponding stress-strain curve. In this technique, a micromachined composite membrane consisting of a LPCVD Si3N4 film and the metal film of interest is deformed by applying pressure to the membrane. The Si3N4 film serves a dual purpose: it passivates the surface of the metal film and provides the driving force to deform the metal film in compression when the membrane is unloaded. The technique is applied to thin sputter deposited Cu and Al films. Passivated films show a very strong Bauschinger effect with a yield stress in compression of approximately one third of that in tension. By contrast, the stress-strain curves of unpassivated films show only a slight effect when the films are fully unloaded. A dislocation-based mechanism is proposed to explain the effect in the passivated films. The results of this study provide an unambiguous explanation for observations made using the substrate curvature technique and which suggest a softening of metal films on Si substrates when they are heated until the residual stress becomes compressive.

**4:00 PM V9.9**
**The Effect of Grain Size on the Mechanical Behavior of Hafnium.** Ellen Cerreta and George Thompson Gray; MST-8, Los Alamos National Laboratory, Los Alamos, New Mexico.

The mechanical behavior of hafnium, a hexagonal closed packed metal, is influenced by texture, strain rate, temperature, chemistry, and stress state, however the effects of microstructure are not well understood. As is observed in other group IVa elements, such as titanium and zirconium, deformation at quasi-static and dynamic strain rates occurs through a combination of prismatic and pyramidal slip, which are temperature and rate dependent, and twinning. Additionally, the mechanical response of hafnium is sensitive to the degree of twinning within the microstructure, as twins limit available glide distances for prismatic slip. The mechanical behavior of titanium and zirconium has been previously shown to be highly dependent upon the average grain size of the material. Yield stresses decrease for increasing grain sizes, however work hardening rates are observed to increase. This result is attributed to increased twinning for microstructures with smaller grain sizes due to high pile up stresses incurred with even limited dislocation glide. In this talk, the effects of grain size on the mechanical response of high purity hafnium tested in compression are probed and the flow stress and work hardening behavior is correlated with the substructural evolution during deformation. The influence of strain rate, temperature and texture as a function of the microstructure is also examined and compared to corresponding zirconium and titanium mechanical test data.

**4:15 PM V9.10**
**Scaling Laws for Open-Cell Nanoporous Nanocrystalline Gold.** Andrea Maria Hodge, Juergen Biener, Alex V. Hamza, Luke L. Hsiung and Joe H. Satcher; Chemistry and Materials Science, Lawrence Livermore National Laboratory, Livermore, California.

Nanoporous metals synthesized by electrochemically-driven dealloying of binary alloys have attracted considerable interest due to potential sensor and actuator applications. Despite the progress made in understanding the process of porosity formation during dealloying, very little is known about the mechanical properties of nanoporous metals. Here we present a complete characterization of pore size and grain structure of nanoporous gold foams (70 and 60% porous) processed by Au/Ag dealloying, and their mechanical behavior performed by nanoindentation. The nanoporous Au samples that were investigated have a relative density of 30% and 40% with a sponge-like morphology and pore sizes on a length scale of 100 and 40 nm, respectively. The foam struts for both foams are polycrystalline with a typical grain size $< 50$ nm. The hardness and elastic modulus were determined by nanoindentation and then compared to values calculated from Ashby and Gibson open-cell foam scaling equations. The yield strength of the foams was measured to be 180 MPa for the 30% relative density and 240 MPa for the 40% relative density with a Young's Modulus of 11 GPa for both foams. These results are consistent with expectations drawn from the scaling laws of open-cell foams.

**4:30 PM V9.11**
**Dislocation Junctions with Variable Strength - a Source of Negative Strain Rate Sensitivity in Solid Solutions.** Catalin Picu and Monica A. Soare; MANE, Rensselaer Polytechnic Institute, Troy, New York.

Many solid solutions in which the solute concentration is below the precipitation level (e.g.in Al-Mg, Cu-Mn and Cu-Al alloys) exhibit negative strain rate sensivitity (NSRS) in a certain range of temperatures. A new physically-based constitutive model capturing the negative strain rate sensitivity is presented. The physics behind the effect is the variation of the strength of dislocation junctions due to solute clustering on forest dislocations. The most important junction types are considered: the Lomer Cottrel lock, the glissile junction and junction with collinear Burges vectors, and their strength is evaluated for many geometrical configurations in presence of a cluster of solute on the stationary forest dislocation. The mesoscopic constitutive model is obtained by averaging the strength of the whole population of junctions. Hence, the nanomechanics of junction formation and failure leads to NSRS at the mesoscopic scale. The model predictions are compared with experimental data and it is shown that it reproduces most of the observed features such as the evolution of the transient strain rate sensitivity parameter with deformation and the range of temperatures and strain rates

corresponding to NSRS.

**4:45 PM V9.12**
**Strength measurement of submicron single $\alpha$-alumina particle.** Hisato Ogiso, Mikiko Yoshida, Shizuka Nakano and Jun Akedo; Advanced Manufacturing Research Institute, National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan.

Various properties of particles are known to be different from those of the bulk ones, as the particle size become considerably small, called "size effect". The particles will, therefore, have the size effect on their strength. However no strength has been reported on particles of which diameter is less than several tens micrometer, because it has been difficult to directly measure the strength of such small particles. We have developed a compression test system to measure the strength of a single particle with the diameter of several hundreds nanometer. In this system, a single particle is placed on a diamond substrate, and is then compressed by a diamond compression presser whose top has a small flat. The size of the flat top must be comparable to the particle size, because two potential problems: compression of multiple particles and direct contact, can be arose. Diamond fabrication of a micron-sized flat area was, here, achieved by using focused ion beam technology. $\alpha$-alumina powder whose average diameter is 0.7 $\mu$m was prepared, and each single particle of the powder was tested for the strength using the system. Results from the measurement of ten sample particles showed that the strength of the $\alpha$-alumina particles was 3.2 GPa in their average, and ranged from 1.5 GPa to 7.4 GPa. Both the average strength and the relative deviation of the measured strength are larger than those of $\alpha$-alumina partilces whose average diameter is 2 $\mu$m. The load-displacement curve during the compression test indicated that the particle underwent brittle destruction.