

SYMPOSIUM BB

Mechanical Properties of Nanostructured Materials—Experiments and Modeling

March 29 - April 1, 2005

Chairs

J. G. Swadener

Los Alamos National Laboratory
MST-8, MS G755
Los Alamos, NM 87545
505-667-9952

Erica Lilleoden

Lawrence Berkeley Laboratory
MS 66-200
1 Cyclotron Rd.
Berkeley, CA 94720
510-486-4836

Syed Asif

Hysitron, Inc.
1025 Valley View Rd.
Minneapolis, MN 55344
952-835-6366

David Bahr

School of Mechanical & Materials Engr
Washington State University
Pullman, WA 99164-2920
509-335-8523

Daniel Weygand

Institut für Zuverlässigkeit von Bauteilen und Systemen
Universität Karlsruhe
Kaiserstr. 12
Karlsruhe, 76131 Germany
49-721-608-8508

Symposium Support

†Hysitron, Inc.

†2005 Spring Exhibitor

Proceedings to be published online
(see *ONLINE PUBLICATIONS* at www.mrs.org)
as volume 880E
of the Materials Research Society
Symposium Proceedings Series.

This volume may be published in print format after the meeting.

* Invited paper

SESSION BB1: Microscopic Characterization and Processing

Chairs: Bennett Larson and Helena van Swygenhoven
Tuesday Morning, March 29, 2005
Room 2016 (Moscone West)

8:30 AM *BB1.1

3D X-Ray Structural Microscopy Study of Nanoindentation Induced Deformation in Copper. Bennett Larson, Condensed Matter Sciences, Oak Ridge National Lab., Oak Ridge, Tennessee.

Nanoindentation in ductile materials induces plastic deformation within confined volumes under well-defined boundary conditions and strong strain gradients. Three-dimensional x-ray structural microscopy with submicron point-to-point spatial resolution has been used to make non-destructive measurements of the local deformation microstructure under nano/microindents in single crystal Cu. Measurements of local lattice orientations under spherical indents in Cu (~0.02 degree angular resolution) show a complex distribution of collective lattice rotations with sharply defined features penetrating deeply into the crystal. These local rotation measurements have been used to construct 3D spatially resolved maps of the local lattice curvature in terms of local Nye dislocation tensors. Spatially resolved plots of the three orthogonal rotations and each of the nine components of the dislocation tensor will be presented for selected slices through the deformed volumes. Such measurements provide a direct and absolute connection between experimental measurements of deformation in materials and the results of computer simulations and multi-scale modeling. The outlook for extending x-ray structural microscopy measurements to shorter length scales and polycrystalline materials will be discussed. *Research sponsored by the Department of Energy, Office of Science, Division of Materials Sciences at ORNL managed by UT-Battelle, LLC, under contract DE-AC05-00OR2272. The work was performed on the UNI-CAT beamline at the APS; the operation of the APS is sponsored by the DOE.

9:00 AM BB1.2

Influence of Temperature on in-situ X-ray Peak Profile Behaviour in Nanocrystalline Ni. Stefan Brandstetter¹, Zeljka Budrovic¹, Steven Van Petegem¹, Bernd Schmitt², Antonio Cervellino¹ and Helena Van Swygenhoven¹; ¹ASQ/NUM, Paul Scherrer Institution, Villigen PSI, Switzerland; ²SYN, Paul Scherrer Institute, Villigen PSI, Switzerland.

Electrodeposited nc-Ni and ultra-fine grained HPT-Ni are deformed in-situ under tension at the materials beamline of the Swiss Light Source allowing measurement of peak position and peak width as a function of applied stress and strain, from which information on elastic and plastic properties can be obtained. Continuous, loading/unloading and stress relaxation experiments are carried out at 300K and now also at 200K where diffusional properties are expected to be suppressed. The Young's modulus is calculated from the peak positions as a function of stress during elastic loading, revealing different elastic properties between ED-Ni and HPT-Ni. Peak profile analysis demonstrates that plastic deformation in HPT-Ni 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. On the contrary, the peak broadening during plastic deformation of nanocrystalline Ni is entirely reversible upon unloading, demonstrating that the deformation process does not build up a residual dislocation network (Science 304 (2004) 273). Lowering the deformation temperature for ED-Ni reduces the amount of peak broadening however keeping the reversibility upon unloading. Stress relaxation at room temperature is characterized by a serious drops in peak broadening over the time span of a few hours, whereas at low temperatures, hardly any reduction in peak width is observed. The effect of temperature on the deformation mechanism is discussed in terms of dislocation and grain boundary accommodation mechanisms suggested by atomistic computer simulations.

9:15 AM BB1.3

Characterization of the Microstructure of Nanostructured Electrodeposited Materials by use of Electron Backscattered Diffraction (EBSD). Alice Bastos da Silva and Dierk Raabe; Microstructure Physics and Metal Forming, Max-Planck-Institute for Iron Research, Duesseldorf, Germany.

Detailed experimental studies at the grain scale in nanostructured polycrystalline materials have so far been limited by the presence of impurities, porosity, strain heterogeneity and microstructure heterogeneity. An accurate characterization of the microstructure of nanostructure electrodeposited materials is of great interest in order to improve their properties. The characterization of some microstructural parameters can be routinely done by use of orientation imaging microscopy (OIM). In this study we investigate nanostructured Nickel and Nickel-Cobalt alloys produced by

electrodeposition. The grain sizes range from 40nm to 800nm in diameter. Results mechanical properties were investigated by tensile test performed within a high resolution scanning electron microscope and by nanoindentation. The characterization of the grain shape through the cross-section, population of growth twins, grain boundary character, local texture and local relationship between microstructure and grain orientation were done by use of Electron backscattered diffraction (EBSD).

9:30 AM BB1.4

Process and Properties of Bulk Nanostructured Materials. Ricardo B. Schwarz¹, Tong D. Shen¹, Xing H. Zhang¹ and Ke Han²; ¹MST-8, Los Alamos National Laboratory, Los Alamos, New Mexico; ²National High Mag. Field Lab., Tallahassee, Florida.

During the past two decades, there has been increasing interest into nano-crystalline materials, aiming at exploring their potential for applications. Most of the studies, however, have been in zero-dimensional (nanoparticles), one-dimensional (nanowires) and two-dimensional (thin-film multilayers) nanomaterials. There have been few studies on three-dimensional bulk nanomaterials because good-quality BULK nanocrystalline alloys are difficult to prepare. We have prepared bulk nanocrystalline Ag-Cu alloys that are free of artifacts (porosity, inclusions, cracks, etc). The breakthrough came through the use of a flux-melting technique, which enabled us to purify the molten alloy, removing most heteronucleants. A purified eutectic Ag-Cu melt was thus undercooled by approximately 250 K. At this large undercooling, the rate of crystal nucleation dominates over the rate of crystal growth, resulting in a nanocrystalline microstructure. These alloys are composed of Ag and Cu lamellae, with an average lamellar spacing of 50 nm and an average crystallite size of 40 nm. We have studied the microstructure, physical, and mechanical properties of bulk Ag-Cu nanocrystalline alloys. The alloys have high strength (in the 500 to 1,000 MPa range), reasonable tensile ductility (10% elongation at 500 MPa), very soft elastic moduli (10% of polycrystalline alloys), high conductivity (80% of fully annealed pure Cu), and relatively high thermal stability. Furthermore, the yield strength measured in tension and in compression are approximately the same, indicating the asymmetry between these quantities previously reported for nanocrystalline materials was most likely due to manufacture artifacts. The strength of our nanocrystalline Ag-Cu rods has been further increased by drawing. These alloys have potential for the manufacture of coils for high-energy pulsed magnets.

9:45 AM BB1.5

Mechanical Behavior of a Nanostructured Al Matrix Composite. Jichun Ye, Bing Q. Han, Feng Tang and Julie M. Schoenung; Chemical Engineering and Materials Science, University of California, Davis, Davis, California.

Mechanical milling at cryogenic temperatures (cryomilling) was applied to fabricate a composite powder with 20 wt. % submicron B4C particulates in a nanocrystalline (NC) 5083 Al matrix. A mixture of 50 wt. % cryomilled composite powder and 50 wt. % unmilled coarse-grained (CG) 5083 Al powder was degassed, followed by cold isostatic pressing (CIP) and extrusion to form a nanocomposite with 10 wt. % B4C, 50 wt. % CG 5083 Al and balance NC 5083 Al. The mechanical properties under compressive and tensile loading conditions at various temperatures were examined. At ambient temperature, the composite possessed a yield stress of 1150 MPa and elongation-to-failure of 2.5 % under the compressive load, while under tensile load it fractured in elastic deformation region. A tension-compression symmetry was observed at all elevated temperatures. The composite exhibited a high yield strength of 940 MPa and 340 MPa at temperatures of 373 K (0.43 Tm) and 473 K (0.55 Tm), respectively. Even at a high temperature of 673 K (0.78 Tm), the composite still possessed a yield strength of 100 MPa. A highest tensile ductility of 26% was observed at 473 K. Homogeneous compressive deformation was observed at all the elevated temperatures (373 K). The hybrid microstructure of this composite, including the distribution of each phase, the grain sizes of the Al matrix, the interfaces between these three phases, and the fracture surfaces were characterized using transmission electron microscopy (TEM) and scanning electron microscopy (SEM) techniques. The relationship between the microstructures and mechanical properties was discussed.

10:30 AM BB1.6

Effect of Particle Dispersions on Tensile Properties of Oxide Dispersion Strengthened (ODS) and Nanostructured (NS) Ferritic Alloys. David T. Hoelzer, Mikhail A. Sokolov, James Bentley, Michael K. Miller and Roger E. Stoller; Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Oxide-dispersion strengthening (ODS) of ferritic steels is a well known process for improving their high-temperature strength and creep properties. In recent years, advances in understanding the mechanical alloying (MA) process have resulted in creating a new class of

nanostructured (NS) ferritic alloys that contain a high number density of <10 nm size particles. In this study, different particle dispersions have been developed by ball milling powders of pre-alloyed Fe-14Cr-3W-0.4Ti (nominal wt.%) with 0.3 wt.%Y₂O₃ (17-31 nm initial size) and extruding at 1175°C and 850°C. The microstructural characterization of these materials using energy filtered transmission electron microscopy (EFTEM) and atom probe tomography (APT) showed that a low number density of Y- and Ti-based oxide particles with sizes ranging from ~10 to 50 nm formed at the higher extrusion temperature whereas a high number density of ~2-5 nm size particles enriched in Y, Ti, and O atoms formed at the lower extrusion temperature. The lower extrusion temperature also resulted in the formation of a uniform distribution of ~100-200 nm size grains. The grain boundaries of the nano-grains were observed to be decorated with <10 nm size Y-, Ti-, and O-rich particles. Another parameter that affected the development of the particle dispersion was ball milling conditions. In one experiment, the milling conditions resulted in a bi-modal grain size distribution spanning several microns in which there were regions with 1-4 μm grains and regions with ~100-200 nm grains. The characterization of the large grain regions found that only large Ti-rich oxides formed during processing. Uniaxial tensile tests were conducted on the MA Fe-14Cr-3W-0.4Ti+0.3Y₂O₃ ferritic alloy at temperatures ranging from room temperature to 800°C. In this paper, results of the microstructural characterization and uniaxial tensile tests will be presented and will be used to compare the effect of variations in particle dispersion and grain size on the high-temperature mechanical properties of this ferritic alloy.

10:45 AM BB1.7

Nanoscale Twins in Sputtered 330 Stainless Steel Thin Films: The Influence of Deposition Parameters. Xinghang Zhang¹,

Amit Misra², John G. Swadener², Haiyan Wang², Ana L. Lima², Michael A. Nastasi², Michael F. Hundley² and Richard G. Hoagland²; ¹Mechanical Engineering, Texas A&M University, College Station, Texas; ²Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, New Mexico.

We have synthesized single-phase 330 stainless steel (330 SS) thin films by magnetron sputtering. The as-deposited 330 SS films have extremely high density of twins with nanoscale spacing (a few nm). These twins are of {111} type with Σ3 twin interface aligned parallel to the substrate surface. The as-sputtered films have hardness values exceeding 6 GPa, about an order of magnitude higher than bulk 330 SS. The unusually high strength originated mainly from the strength of twin interface as revealed by molecular dynamics simulations. Analysis based on our thermodynamic model indicates that the nucleation of twins could be related to the deposition rate, or deposition flux. We will present studies on the evolution of microstructures as well as the mechanical and electrical properties of 330 SS with deposition flux and subsequent thermal annealing. These studies will shed some light on the formation mechanisms of nanoscale twins in 330 SS.

11:00 AM BB1.8

New Deformation Mechanism in Nanoscale Fe-C Alloy by Shear $\alpha \rightarrow \gamma$ Transformation. Yulia Ivanisenko¹, Ian MacLaren²,

Ruslan Z. Valiev³ and Hans-J. Fecht^{4,1}; ¹Institut fuer Nanotechnologie, Forschungszentrum Karlsruhe, 76021 Karlsruhe, Germany; ²Department of Physics and Astronomy, University of Glasgow, Glasgow G12 8QQ, United Kingdom; ³Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, 450000 Ufa, Russian Federation; ⁴Division of Materials, Ulm University, 89081 Ulm, Germany.

Molecular dynamics (MD) simulations predict the length-scale relationship between the grain size and deformation mechanism operating in nanocrystalline materials. This length scale is determined by the difficulty for the perfect dislocations to be nucleated when grain size becomes comparable with the length of the dislocation source. Therefore it was MD predicted and experimentally confirmed that in the fcc metals with very small grain size, the deformation propagates via slip of partial dislocations, twinning and grain boundary sliding. Contrary to that, MD simulations of the deformation behaviour of nanocrystalline Fe, a bcc metal, predict the strain induced formation of closely packed hcp and fcc phases. These predictions confirm the conclusion made in early 30th that stress induced phase transformation can play a role of additional deformation mechanism, because if yielding by slip does not intervene, the applied stress will activate the transformation, when the shear-stress component, resolved along the habit plane, reaches the critical value. It is well known that severe plastic deformation (SPD) of pearlitic steel results in formation of nanocrystalline α Fe-C alloy with a mean grain size of 10 nm, dissolved carbides and extremely high hardness of 10-12 GPa. Here we report our HRTEM observations of formation of nano-sized austenite grains as a result of SPD of pearlitic steel using the high pressure torsion (HPT). Orientation relationship between parent ferrite and new austenite are either

Kurdjumov-Sachs or possibly Nishiyama-Wassermann, i.e. the same as those observed for temperature-driven martensitic transformations in Fe and steels. As it can be easily shown that the thermal origin of the austenite formation due to adiabatic heating can be excluded, we pioneer the discovery of the reverse martensitic transformation at the room temperature in α Fe-C alloy. We show that such unusual phase transformation is possible due to two factors: (i) increase of the free energy of ferrite because of the increase of its free volume in NC state, and (ii) since part of the driving force necessary for the transformation is provided by the mechanical work performed by the shear stress at high pressure torsion. The possible stabilising factors preventing the re-transformation of the austenite back to the stable at the room temperature ferrite may be dissolved carbon, residual strain and constraints from neighbouring crystals, small crystallite size, and lack of suitable nuclei for this transformation.

11:15 AM BB1.9

In-situ TEM Study of Interface Sliding and Migration of an Ultrafine Lamellar Structure. Luke L. Hsiung¹ and T. G. Nieh²;

¹Chemistry and Materials Science, Lawrence Livermore National Laboratory, Livermore, California; ²Materials Science and Engineering, The University of Tennessee, Knoxville, Tennessee.

It has been reported that the interface sliding with a cooperative motion of interfacial dislocations plays a crucial role in the creep deformation behavior of ultrafine lamellar TiAl (γ lamellae: 100 ~ 200 nm thick, α_2 lamellae: 10 ~ 50 nm thick). Since the multiplication and motion of lattice dislocations within γ and α_2 lamellae become largely restricted as a result of the ultrafine lamellar spacing, the plasticity is mainly accommodated by the motion of pre-existing interfacial dislocations. It is also possible that the interfaces in lamellar TiAl could migrate directly through the cooperative motion of interfacial dislocations and lead to the coarsening of lamellar spacing. Therefore, shear-induced interface sliding could result in a weakening effect when lamellar TiAl with an ultrafine microstructure is employed for engineering applications. Although it is anticipated that the interface-sliding phenomenon is more prevalent at elevated temperatures, the current investigation provides the direct evidence of the occurrence of interface sliding and migration within ultrafine lamellar TiAl even when it is strained at room temperature. This work was performed under the auspices of the U.S. Department of energy by University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

11:30 AM BB1.10

Effect of Annealing on the Mechanical Properties of Porous Titania Nanoparticle Agglomerate Films.

Oluwatosin A. Ogunsola¹, Gil Lee² and Sheryl Ehrman¹; ¹Chemical Engineering, University of Maryland, College Park, Maryland; ²School of Chemical Engineering, Purdue University, West Lafayette, Indiana.

Titania porous films have a variety of uses in high surface area applications such as sensors, photovoltaic electrodes (solar cells) and semiconductor photocatalysts. In this work, porous films of titania were synthesized via a hybrid process based upon gas-to-particle conversion and particle precipitated chemical vapor deposition. Agglomerates of titania nanoparticles made at high temperature were interconnected at low temperature to form the nanoporous film. Two types of bond exist between the particles making up the porous films: strong chemical bonds that are formed when the particles sinter partially at high temperature and weak physical van der Waals bonds that exist between the particles when they collide at low temperatures. The porous films synthesized are highly porous (98%) but of low mechanical strength due to the presence of the weak van der Waals bonds. Annealing the films increases the interconnectivity of the particles by enhancing old chemical bonds and forming new chemical bonds in replacement of the van der Waals bonds. This will increase the mechanical strength of the film and ensure that it does not collapse in its end use, causing reduction in porosity, which reduces its productivity. The increase in interconnectivity is accompanied by a change in the primary particle and agglomerate sizes, as shown by transmission electron microscope images, BET surface area measurements, and measurements of agglomerate size distributions using dynamic light scattering. The mechanical properties of the films such as elastic moduli and plastic-elastic deformation are measured via nanoindentation. The objective of the work described here is to gain a better understanding of how annealing affects the mechanical properties of these porous titania nanoparticle agglomerate films. This will enable engineering of stronger films without loss of their valuable porosity.

11:45 AM BB1.11

Indentation Studying the Mechanical Properties of Amorphous Al₈₅Ni₁₀La₅ Alloy. Zhihui Zhang, Leonardo

Ajdseltajn, Yizhang Zhou and Enrique J. Lavernia; Dept of Chemical Engineering and Materials Science, University of California, Davis, California.

Nanoindentation and Vickers microhardness indentation are carried out to study the mechanical properties of an amorphous $\text{Al}_{85}\text{Ni}_{10}\text{La}_5$ alloy produced by gas atomization. Formation of deformation zones indicates that the alloy deforms via the shear banding mechanisms. DSC analyses show that this amorphous alloy undergoes a three-stage crystallization process in the temperature range of $250^\circ\text{C} \sim 390^\circ\text{C}$. The glass transition temperature for this alloy is determined to be 259°C at a heating rate of $40^\circ\text{C}/\text{min}$ (and it is of approximately 248°C at a heating rate of $20^\circ\text{C}/\text{min}$). The influence of devitrification on the mechanical properties is studied by annealing the amorphous alloy at temperatures well below or close to glass transition (235°C , 245°C , 250°C), for the first reaction (263°C) and for the second reaction (283°C). In this paper, the microstructure evolution and the nanoscale crystallites are characterized using SEM, TEM and XRD. The effects of devitrification on the indentation size, appearance and shear band formation as well as the corresponding hardness and the elastic modulus will be discussed. The hardening behavior will be described by considering the partially crystallized alloy as a nanocomposite using the rule of mixtures.

SESSION BB2/O3: Joint Session: Mechanical Behavior
of Nanostructured Films

Chairs: Peter Anderson and Erica Lilleodden
Tuesday Afternoon, March 29, 2005
Room 2016 (Moscone West)

1:30 PM *BB2.1/O3.1

Plastic Strength Maps for Metallic Multilayer Thin Films.

Peter M. Anderson, Dept of Materials Science and Engineering, Ohio State University, Columbus, Ohio.

From an engineering perspective, new classes of materials benefit from a knowledgebase from which properties can be viewed as a function of structure. For metallic multilayer thin films, such structural parameters include the multilayer period, volume fraction of phases, interfacial properties between phases, and individual properties of phases such as elastic moduli and stress-free lattice parameter. This presentation discusses the development of plastic strength maps for metallic A/B multilayer thin films. The maps are based on the premise that bulk plastic yield occurs when dislocation loops are able to percolate throughout the multilayer thin film. A simple assumption for percolation is that the film is stressed and deformed in tension sufficiently to eliminate compression in any of the alternating layers. The resulting maps predict contours of constant plastic strength for a given A/B system, as a function of bilayer period and volume fraction of phases. Two implications are that volume fraction may be a relatively potent parameter to increase plastic strength, particularly in the "strength plateau" region at small bilayer period. Second, multilayer thin films are predicted to have a large asymmetry in bulk yield strength in tension versus compression, similar to bulk layered composite materials.

2:00 PM BB2.2/O3.2

Atomic-Scale Analysis of Strain Relaxation Mechanisms in Ultra-Thin Metallic Films. M. Rauf Gungor and Dimitrios Maroudas; Department of Chemical Engineering, University of Massachusetts, Amherst, Amherst, Massachusetts.

Atomic-scale modeling based on molecular-dynamics (MD) simulation provides a powerful means for analyzing atomistic mechanisms of strain relaxation in metallic thin films and enables the development of constitutive equations for continuum modeling of metallic thin-film mechanical behavior. The results of such modeling are particularly important for model-based prediction of materials reliability in today's nanoscale electromechanical devices. In this presentation, we report a comprehensive computational analysis of the atomistic mechanisms of strain relaxation and failure under a wide range of applied biaxial tensile strain in free-standing Cu thin films with the film plane oriented normal to the [111] crystallographic direction. The analysis is based on isothermal-isostrain MD simulations within an embedded-atom-method (EAM) parameterization for Cu and using slab supercells that contain millions of atoms with and without cylindrical voids normal to the film plane and extending throughout the film thickness. Our analysis has revealed various regimes in the film's mechanical response as the applied strain level increases. After an elastic response at low strain ($< 2\%$), plastic deformation occurs accompanied by dislocation emission from the void and film surfaces, void surface morphological transitions, dislocation joggling, vacancy generation by jogged dislocations, vacancy pipe diffusion along dislocation cores, dislocation-vacancy and dislocation-dislocation interactions, as well as formation and propagation of threading dislocation loops. At the lower strain range following the elastic-to-plastic deformation transition ($< 6\%$), void growth is the major strain relaxation mechanism mediated by emission of perfect

screw dislocation dipoles from the void surface and subsequent dislocation propagation; as a result, a plastic zone forms around the void. At higher levels of applied strain ($> 6\%$), a subsequent transition to a new plastic deformation regime gives rise to a practically uniform distribution of dislocations in the metallic thin film. Under such conditions, dislocations are emitted from the free surfaces of the thin film and inhibit void growth as the dislocations emitted from the void surface are pinned by their interaction with the simultaneously generated dislocations from the film's surface. By comparing MD simulation results in identical thin films with and without voids, it is also demonstrated that strain relaxation at high levels of strain is not affected by an existing void in the metallic film.

2:15 PM BB2.3/O3.3

Deformation Mechanisms during Nanoindentation of Ultrafine and Nanocrystalline Metals. Miao Jin¹, Andrew M. Minor², Daibin Ge², Eric A. Stach³ and J. W. Morris, Jr.¹; ¹Department of Materials Science and Engineering, University of California, Berkeley, Berkeley, California; ²National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California; ³School of Materials Engineering, Purdue University, West Lafayette, Indiana.

The plastic deformation mechanisms of materials with grain sizes in the nanoscale regime (1-500nm) are still largely debated. Recent experimental results and computational simulations show that grain boundary sliding and/or grain rotation can become dominant deformation modes when grain sizes shrink to below some critical size (on the order of 10 nm). In this work, we combine conventional ex situ nanoindentation with in situ nanoindentation in a TEM in order to gain insight into the relevant deformation mechanisms for ultrafine-grained and nanocrystalline Al films. We will present real-time videos that show that stress-induced grain growth- resulting from grain boundary migration, grain rotation and grain coalescence- is a common occurrence in these materials as the indentation proceeds. Our results suggest that grain growth and coalescence in these inherently metastable microstructures appear to be important modes of response in the deformation of ultrafine-and nanograined materials.

2:30 PM BB2.4/O3.4

In-Situ Peak Profile Analysis of Submicron Aluminum Thin Films in Tension. D. S. Gianola¹, S. Brandtetter², K. J. Hemker¹, A. Cervellino² and H. Van Swygenhoven²; ¹Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland; ²Paul Scherrer Institute, Villigen, CH-5232, Switzerland.

Mechanical properties of metals are altered when scaling down sample dimensions to the micron scale and/or structural length scales (for instance grain size) to the nanometer scale. In order to study the deformation behavior while concurrently sampling information on the structural changes in a material, a novel in-situ tensile testing technique that uses synchrotron X-rays to track the Bragg peaks during the evolution of straining is utilized. This experiment has been developed at the Swiss Light Source and uses a microstrip detector that covers an angular range of 60° , allowing for the measurement of several diffraction peaks at once during deformation. The specimens are fabricated using MEMS-inspired processes on Si wafers such that the freestanding films span across a rigid Si frame, which alleviates challenges associated with handling of fragile samples. Al is deposited by pulsed DC-magnetron sputtering onto the Si substrate to yield high-purity specimens. Free standing thin films of thicknesses of 200 and 400 nm with mean grain sizes of approximately 40 and 90 nm, respectively, are deformed during tensile load-unload and stress relaxation cycles. The mechanical behavior is represented by means of tensile measurements of yield stress, UTS, total strain and activation volume. The peak broadening in terms of applied stress and a careful TEM characterization of the thin films before and after deformation reveal the underlying deformation mechanisms in submicron thin films. The peak shift in terms of the applied stress allows the determination of Young's modulus. The results will be discussed in terms of both thickness and grain size.

2:45 PM BB2.5/O3.5

Behavior of Individual Grains in Nanocrystalline Ni during Deformation. Zhiwei Shan¹, Eric Stach², James Knapp³, David Follstaedt³, Jorg Wiezorek⁴ and Scott Mao¹; ¹Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; ²National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California; ³The Physical, Chemical and Biomolecular Sciences Center, Sandia National Laboratories, Albuquerque, New Mexico; ⁴Materials Science and Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania.

The dynamic behavior of individual grains in nanocrystalline Ni during deformation has been studied by in situ tensile straining, bright-field transmission electron microscope and micro beam electron

diffraction observations under low local strain rate. It was found that grain boundary mediated processes contributed prominently to plastic deformation, as predicted by theoretical consideration as well as molecular dynamics simulations for ductile metals with a grain size below a critical value. Moreover, it was found that interiors of nano-sized grains experience severe lattice distortions during the deformation. This observation apparently challenges the generally invoked assumption that interiors of nano-sized grains behave as a rigid body during deformation. Based on our experimental TEM observations as well as other recently reported experiments results, a unifying picture for the unusual deformation mechanism for nanocrystalline face-centered-cubic metals is proposed. 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.

3:30 PM BB2.6/O3.6

Grain Agglomeration in Nanocrystalline Ni. Zhiwei Shan¹, David Follstaedt², James Knapp², Eric Stach³, Jorg Wiecek⁴ and Scott Mao⁵; ¹Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; ²Physical and Chemical Science Center, Sandia National Laboratories, Albuquerque, New Mexico; ³School of Materials Engineering, Purdue University, West Lafayette, Indiana; ⁴Materials Science and Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania.

Tensile to fracture of full dense nanocrystalline metals often exhibit a dimple morphology at the fracture surface with characteristic dimple sizes much larger than that of the average grain size. By exploring in situ tensile transmission electron microscopy tests on high quality, free stranding nanocrystalline Ni films with an average grain size of about 10nm, it was found grain agglomerates formed very frequently and rapidly in many locations apparently independently of one another through collective rearrangements of groups of neighboring grains under influence of the applied stress. Guided by the changes documented in the Ni films during the in situ TEM tests, we propose that the dimple structures observed on fracture surfaces could result from the collective motion of those newly formed agglomerates and those pre-existed "nano domains". Implications of the dynamic TEM observations as well as the proposed interpretation on the strength and ductility of nanocrystalline materials are discussed. 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-AC-94AL85000.

3:45 PM BB2.7/O3.7

Characterization and Mechanical Behavior of Cu/Cu Nano-Laminates. Andrea Maria Hodge^{1,2}, Y. Morris Wang^{1,2} and Troy W. Barbee Jr.¹; ¹Chemistry and Materials Science, Lawrence Livermore National Laboratory, Livermore, California; ²Nanoscale Synthesis and Characterization Laboratory, Lawrence Livermore National Laboratory, Livermore, California.

The development of atomic level deposition of layers has allowed the fabrication of free-standing nano-laminates, also known as multilayers. These multilayers incorporate the structural advantages of both composites and nano-crystals, which makes them candidates for a wide range of applications. In this talk, we will concentrate on high purity (99.999%), fully dense, free-standing Cu/Cu nano-laminates, produced by DC magnetron sputtering, and composed of nanometer-sized layers of Cu with thickness ranging from 1.2 nm to 43.6 nm. The macroscopic thickness of the nano-laminate samples was ~25 microns, which, depending on the layer thickness, contained from 520 to 18,000 layers. The nano-laminates were characterized by depth sensing nanoindentation and Vickers microhardness on their top and bottom surfaces (normal to the layering) and parallel to the layers. These tests showed hardness values on the order of 2.5 to 3.0 GPa, a substantial increase in strength over conventional polycrystalline Cu. Our results present a trend towards a Hall-Petch plateau for grain sizes below 10nm. This result differs from previous work on nanocrystalline materials synthesized using other technologies with grain sizes less than 10 nm, which showed a trend towards a reverse Hall-Petch behavior. Extensive plan view and cross-section transmission electron microscopy (TEM) demonstrates the nanocrystallinity of the Cu/Cu multilayers as well as extensive twinning. This allows us to compare the effects on grain size and twinning by individual layer thickness and to relate this to the hardness and yield strength of these Cu/Cu nano-laminates. 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.

4:00 PM BB2.8/O3.8

Mechanical Properties and Deformation under Nanoindentations

in Metal/Nitride Nanoscale Multilayers. Gregory Abadias¹, Yau-Yau Tse¹, Anny Michel¹, Christophe Tromas¹ and Sergey N. Dub²; ¹Laboratoire de Metallurgie Physique, UMR 6630, Université de Poitiers, Futuroscope-Chasseneuil, France; ²Institute of Superhard Materials, Kiev, Ukraine.

Mechanical behavior and stability of thin film structures consisting of alternating nanolayers have been the subject of great research activity over the past decade due to the technological impact of such nanoscale systems, but also to understand the plastic mechanisms operating at small-scales. Enhancement of hardness and tensile strength as compared to the rule of mixture values for their bulk phase counterparts have been reported in a large variety of systems. These include the case of isostructural, miscible metal/metal or nitride/nitride multilayers, but also non-isostructural and immiscible systems, such as Mo/NbN or W/NbN. However, the exact deformation mechanisms operating under nanoindentation tests (dislocation pileups, dislocation motion within individual layers, grain boundary sliding...) have not been clearly established. In particular, the case of nanolayers combining elastically soft metal (Cu, Ag, ...) and hard nitride (TiN) layers remains to be investigated. The present study reports new data on the structure and mechanical properties of such nanocomposite multilayers. Three nanolayered systems, namely TiN/Cu, TiN/Ag and TiN/W, corresponding to different crystal structure and lattice mismatch combinations, have been considered. They were deposited at room temperature using a dual-target ion beam sputtering in Ar-N₂ mixtures, on Si wafers and MgO(001) substrates. The bilayer period Λ was varied between 2.5 and 50 nm and the total film thickness was equal to $\sim 0.2 \mu\text{m}$. Low-angle and high-angle X-ray diffraction experiments as well as TEM observations were used to characterize the microstructure and crystalline orientation, structure of interfaces and type of growth defects. Mechanical properties were studied by nanoindentation tests using a Berkovich tip and coupled with Atomic Force Microscopy surface observations around the indents. Also, a combined FIB-TEM technique was implemented to image the deformed nanolaminates beneath the indenter. In all systems, low-angle XRD patterns exhibited a large number of superlattice reflections, attesting the good reproducibility of the stacking along the growth direction. For the TiN/Cu system, a cube on cube epitaxial growth with semi-coherent interfaces was observed. Detailed analysis of the hardness values vs. contact depth ratio gave access to the intrinsic film hardness. No enhancement of hardness was found for the case of Cu/TiN and Ag/TiN over the rule of mixtures values, in contradiction with predictions of existing theoretical models. A visco-elastic behavior is rather suggested. The different mechanical behavior are discussed in terms of film microstructure, presence of residual stress and influence of interfacial structure.

4:15 PM BB2.9/O3.9

Thermal Plasma Chemical Vapor Deposition of Superhard Nanocrystalline Silicon Carbide Films. Steven L. Girschick¹, Feng Liao¹, William M. Mook², Michael R. Zachariah³ and William W. Gerberich²; ¹Mechanical Engineering, University of Minnesota, Minneapolis, Minnesota; ²Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota; ³Mechanical Engineering, University of Maryland, Minneapolis, Maryland.

Superhard nanocrystalline silicon carbide films were deposited using thermal plasma chemical vapor deposition. Silicon tetrachloride vapor and methane were injected into an argon-hydrogen plasma generated by radio-frequency (RF) inductive coupling. The flow rates were as follows: argon, 42.5-60 slm; hydrogen, 0.5-2.5 slm; silicon tetrachloride, 68-140 sccm; and methane, 80-140 sccm. The plasma issued from the RF torch into a chamber maintained at a pressure of 33.3 kPa. Molybdenum substrates were mounted on-axis and normal to the flow. Substrate temperatures ranged from 750-1250 C. Deposition rates were extremely high, ranging up to approximately 900 microns/h, as measured by micrometer, for silicon tetrachloride flow rates of 140 sccm. Scanning electron microscopy images of film cross sections showed columnar growth, while the film top surfaces were characterized by hemispherical balls that were composed of nanocrystalline grains. The grain size as determined from X-ray diffraction (XRD) using the Warren-Averbach method ranged from 4-27 nm. XRD showed the films to consist of the cubic phase, 3C-SiC, with a pronounced (111) orientation. For substrate temperatures above 1100 C the XRD peak associated with (200) was additionally observed. RBS measurements indicated that the films were in most cases close to stoichiometric SiC. Analysis of the XRD spectra suggested that the films consisted of SiC nanocrystallites in an amorphous SiC matrix, with the degree of crystallinity ranging from 40-100 percent. Neither XRD nor Raman spectroscopy indicated the presence of a diamond phase. Nanoindentation was used to measure the film hardness, modulus and fracture toughness. Two different nanoindenters were used, a Hysitron TriboIndenter and an MTS Nano Indenter XP. Measurements were made both of film cross sections and

of film top surfaces, in both cases first polishing the surfaces. The hardest film measured had average hardness values for indentations into the cross section of 51 GPa and 56 GPa, as measured by the Hysitron and the MTS instruments, respectively, and 48 GPa for top-down indentation (MTS), with modulus values around or slightly above 500 GPa. Several other films tested also had hardness values in the superhard range, above 40 GPa. In general we found a strong correlation between the hardness and the substrate temperature, with the superhard films being deposited at temperatures above 1100 C. The grain size for these films lay in the range 6-20 nm, and the fractional crystallinity was in most cases in the range 80-85%. Fracture toughness was determined for several films using Vickers indentation of the polished top surface. Measured values ranged up to 4.8-4.9 MPa-m^{1/2}. These films are much harder than standard SiC (about 28 GPa). A detailed investigation of the cause of these exceptional mechanical properties is currently in progress.

4:30 PM BB2.10/O3.10

Thermal Plasma Chemical Vapor Deposition of Superhard Nanostructured Si-C-N Coatings. Nicole J. Wagner¹, Megan

Cordill², Lenka Zajickova¹, Joachim V. R. Heberlein¹ and William W. Gerberich²; ¹Mechanical Engineering, University of Minnesota, Minneapolis, Minnesota; ²Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota.

Materials that protect against erosion, wear, and other harmful degradation are of great interest for various industries to coat, for example, automotive engine parts and machining tools. Superhard nanostructured composite coatings offer enhanced mechanical properties that would reduce and possibly eliminate the necessity of costly and hazardous coolants for such applications. While many methods to produce such materials exist, the thermal plasma chemical vapor deposition (TPCVD) process allows the possibility of using versatile multiphase reactants and producing a controllable film structure at high growth rates. In this study, a triple torch plasma reactor was used to synthesize nanostructured Si-C-N composite films via the TPCVD process. The argon-nitrogen plasma provided atomic nitrogen to the multiphase reactants. Carbon- and silicon-based reactants were injected through a central injection probe and ring configuration at flow rates ranging from 0.01-0.20slm. The deposition pressures ranged from 100-200Torr and the substrate temperatures from 900-1400°C. Comparisons are made between variations of the carbon-based reactants (methane and acetylene), silicon-based reactants (silicon tetrachloride and hexamethyldisilazane), and the substrate material (silicon and molybdenum) and pretreatment (roughening and carbonization). The advantages for using each reactant in the gas mixture were investigated. While the depositions made on molybdenum substrates appeared more uniform than those on silicon, the adhesion of the films was reduced. Micro X-ray diffraction was used to verify the presence of silicon nitride, silicon carbide, carbon nitride and carbon in the films. Both α -Si₃N₄ and β -Si₃N₄ diffraction peaks were seen to be dominant for most of the depositions. The bonding of amorphous phases was investigated using Fourier transform infrared spectroscopy. Although many possibilities of bonding existed for this Si-C-N-H system, infrared bands indicated the presence of N-H, Si-H and C≡N or C≡C in various films. Indentation tests were conducted on the polished film cross-sections and surfaces to determine the hardness and elastic modulus of the depositions. Large variations in mechanical properties were observed for minor changes in film composition. In addition, the substrate temperature has a significant effect on mechanical properties. Correlations between indentation results and scanning electron and optical microscope images showed that the mechanical properties greatly depend on the film morphology; the denser, smoother, and more crystalline films tended to display enhanced mechanical properties. Acknowledgements: Funding provided by the Department of Energy Grant No. DE-FG02-85ER13433 Ao15, the National Science Foundation Integrated Graduate Education and Research Traineeship Grant No. DGE-0114372, and the National Science Foundation North Atlantic Treaty Organization Postdoctoral Fellowship Grant No. DGE-0312210.

4:45 PM BB2.11/O3.11

Depth Profiling of Mechanical Properties on the Nanoscale of Single Layer and Stepwise Graded DLC Films by Nanoindentation and AFM. Carlos Ziebert, Sven Ulrich, Michael Stueber and Helmut Holleck; Institut fuer Materialforschung I, Forschungszentrum Karlsruhe, Karlsruhe, Germany.

The stepwise graded layer concept adjusts a graded constitution of the growing film by a stepwise increase of the ion energy, i.e. the bias voltage, during magnetron sputtering [1]. In order to optimise this concept, which has been developed to manage the high internal stresses of thick diamond-like carbon films (DLC), a depth profiling of the mechanical properties particularly in the interface regions between the layers sputtered with different bias voltage is extremely important. The deposition started with a bias voltage of 0 V to

initiate a high adhesion of the growing film. Then the bias voltage was increased stepwise to -150 V and finally to -300 V to produce a hard film surface. To investigate the influence of the thicknesses of the three graded layers on the thickness of the interface regions different layer deposition time characteristics and thus thickness ratios of the three graded layers were adjusted. When the load variation nanoindentation method was used, where the indenter probes deeper and deeper regions of the sample by increasing the applied load, the depth profile becomes smeared because of the combined mechanical behaviour of the film and the hard metal substrate. However, by using the small angle cross-section nanoindentation method (SACS) it was possible to record depth profiles of the hardness, the elastic modulus and the yield stress with nanometer resolution and to investigate the evolution of these mechanical properties with increasing annealing temperature. In this method the area on the differently graded layers is drastically enlarged by preparing a cross-section under a very small angle of about 0.02 to 0.15 and° the distance travelled by the nanoindenter is translated into depth information using a simple geometric formula. It was revealed that the thickness of the interface regions is mainly determined by the thickness ratio of the graded layers. In addition changes in fracture and crack propagation behaviour of single and stepwise graded DLC films have been investigated by nano- and microindentation using a Berkovich indenter and ex-situ AFM-imaging with respect to ion bombardment during sputter deposition and the thickness of the interface regions between the graded layers. Future research will be focused on optimising the interfaces between the graded layers to further enhance the temperature stability, the maximum achievable thickness and the fracture toughness. Therefore additional theoretical and experimental studies will be needed to improve the understanding of the behaviour of mechanical layer properties across interfaces. [1] H. Holleck and M. Stueber, Method of manufacturing a composite material structure, US Patent No. 6, 110, 329 (2000); EU Patent No. EP 0912774B1 (2002).

SESSION BB3/L3: Joint Session: Nanostructured Biomaterials

Chairs: Greg Swadener and Rizhi Wang
Wednesday Morning, March 30, 2005
Room 2016 (Moscow West)

8:30 AM *BB3.1/L3.1

On the Origin of Stiffening in Biopolymers. Teun Koeman, Patrick R. Onck and Erik Van der Giessen; Materials Science Center, University of Groningen, Groningen, Netherlands.

The cytoskeleton of living cells consists of three types of polymer fibers, made from different proteins and with different diameters: actin microfilaments, intermediate filaments and microtubules. Like other biopolymers, the cytoskeletal fibers are semi-flexible polymeric chains which form low-density networks in the presence of appropriate cross-linking proteins. Rheological experiments on in-vitro networks reveal that their stiffness increases as they deform to large strains. It is generally accepted that this stiffening plays a key role in various cell functions. The conventional view on the origin of stiffening is the nonlinear, entropic-like response of individual filaments when subject to stretching. Using the worm-like chain model for filaments, supplemented with an affine deformation assumption for the behavior of the network, several authors have demonstrated that initially isotropic networks of, for instance, actin stiffen when thermal undulations have been stretched out. This paper presents an alternative explanation that is based on the highly non-affine distortions of actin networks and the enormous compliance for bending of individual actin filaments compared to stretching. A finite-element, periodic cell model is presented for a two-dimensional network of filaments that is subjected to shear strains of up to 25%, like in some rheological experiments. It is demonstrated that the overall response of an initially isotropic network is governed by bending of the filaments, and not by stretching. As the overall deformation increases, the network distorts and a fraction of filaments re-orient in the principal stretching direction. After a geometrical transition, the response of the network is governed by the stretching of filaments rather than bending, thus giving a significantly higher overall stiffness. It is demonstrated that the entropic-like behavior of the filaments act solely to delay the transition. It is also shown that this mechanism operates for actin as well as for microtubular networks.

9:00 AM BB3.2/L3.2

Simulation of Protein and Nanotube Interactions. Melik C. Demirel; Engineering Science and Mechanics, The Pennsylvania State University, University Park, Pennsylvania.

Carbon nanotubes are important elements at the intersection of bio-nano technology. Nanotubes can potentially be assembled using protein templates as building blocks for molecular electronic, optical,

and magnetic devices. Carbon nanotubes have been functionalized with proteins with specific and nonspecific binding (e.g. triton and diamide) by several experimentalist groups. We focus on the dynamics and assembly of streptavidin on a carbon nanotube using molecular dynamics methods. We have simulated the monomeric form of the streptavidin which has 135 aminoacids. The van der Waals and hydrophobic forces play an important role in the dynamics of streptavidin on a carbon nanotube.

9:15 AM **BB3.3/L3.3**

Texture and Smart Anisotropy of Biological Nano-Composites. Helmut Klein² and Dierk Raabe¹;

¹Microstructure Physics, Max-Planck-Institut, Duesseldorf, Germany; ²Mineralogisch-Kristallographisches Institut, Georg-August-Universitaet Goettingen, 37077 Goettingen, Germany.

Many biological materials such as encountered in the exoskeleton structures of molusc and crustacea form biological nano-composites with highly directional mechanical and functional properties. Many of the basic structural ingredients in such structures, i.e. the various mineral components, the proteins, glycoproteins, polysaccharides, and lipids, may at least in part occur in crystalline form. These different compounds are not distributed randomly in orientation space but they typically occur in a variety of preferred topological and crystallographic orientations which are optimized with respect to the external mechanical boundary conditions. By using x-ray wide angle analysis (laboratory-scale x-ray Bragg diffraction in conjunction with an areal detector, synchrotron Bragg diffraction) we present corresponding texture investigations on different biological materials.

9:30 AM ***BB3.4/L3.4**

Normal and Lateral Nanomechanics of Cartilage Aggrecan Macromolecules. Christine Ortiz¹, Delphine Dean², Lin Han¹ and Alan Grodzinsky²; ¹Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; ²Electrical Engineering and Computer Science, Massachusetts Institute of Technology, Cambridge, Massachusetts.

The nanomechanical behavior of cartilage extracellular matrix macromolecules has received increasing attention since their degradation causes loss of tissue and joint function with age and arthritis. For example, loss of the brush-like polyelectrolytic aggrecan macromolecules and its constituent highly charged polysaccharide glycosaminoglycan (GAG) chains significantly reduces cartilage's compressive stiffness. In this study, the conformation and compressibility of a chemically end-grafted bovine epiphyseal aggrecan brush as a function of ionic strength and normal compressive load were measured by combining the techniques of micro-contact printing and contact mode atomic force microscopy (AFM). Micro-contact printing enabled the creation of a patterned surface with densely packed, chemically end-grafted aggrecan (2590+/-90 aggrecan/ μm^2) confined to well-defined micrometer-sized surface areas and a hydroxyl-terminated self-assembling monolayer (11-mercaptoundecanol on Au, OH-SAM) confined to the rest of the surface area. AFM imaging over aggrecan-OH-SAM boundaries enabled the measurement of the height, and hence deformation, of the aggrecan as a function of applied compressive load, solution ionic strength, and pH. With ~ 0 nN of applied compressive force, an uncompressed height ~ 350 nm at 0.001M solution ionic strength was observed to decrease nonlinearly with increasing applied compressive force down to ~ 60 nm at ~ 20 nN. Since the contour length, L_{contour} , of a single aggrecan macromolecule is ~ 400 nm, at 0.001M the aggrecan molecules exist in a partially extended state ($\sim 0.88 * L_{\text{contour}}$) due to intra- and intermolecular electrostatic double layer repulsion between the CS-GAG side chains. As the ionic strength of the solution was increased to 1M, salt screening of the CS-GAG electrostatic forces caused the aggrecan layer height to nonlinearly decrease to ~ 100 nm. Lateral force microscopy images showed that for a constant applied lateral displacement rate, the aggrecan layers exhibited a lower lateral force than the neighboring OH-terminated SAM under lower normal load (~ 3 nN), resulting from shearing stiffness of aggrecan layer, and reversed behavior under higher normal load (~ 20 nN) at 0.001M, due to the "stick-slip" mechanism between the OH-SAM tip and the aggrecan molecules on the substrate. Ongoing theoretical modeling of this data is working to simulate how the unique branched macromolecular architecture of aggrecan leads to novel electrostatic double layer repulsion profiles and conformational entropy changes depending on the solution conditions.

10:30 AM ***BB3.5/L3.5**

Flaw Tolerant Nanostructures of Biological Materials.

Huajian Gao, Max Planck Institute for Metals Research, Stuttgart, Germany.

This work aims at developing a mechanistic theory of nanostructured materials in nature. Biological materials such as bone have achieved

superior mechanical properties through hierarchical composite structures of mineral and protein. Gecko and many insects have evolved hierarchical surface structures to achieve extraordinary adhesion capabilities. We show that the unique nanostructure of these biological materials play a key role in their superior properties. We suggest that the principle of flaw tolerance, which can be related to the survivability of a living organism, may have had an overarching influence on the evolution in biology. We demonstrate that the nanoscale sizes allow the mineral nanoparticles in bone to achieve optimum fracture strength and the nanoprotusions in Gecko to achieve optimal adhesion strength. In both systems, strength optimization is achieved by restricting the characteristic dimension of the basic structure components to nanometer scale so that crack-like flaws do not propagate to break the desired structural link. Application of the flaw tolerance principle at different hierarchical levels allows the stiffness and toughness of biological materials to be optimized up to macroscopic length scales.

11:00 AM **BB3.6/L3.6**

Quantitatively Studying Nano-mechanical Properties within the Prism and Organic Sheath of Enamel. Fuzhai Cui, Jun Ge and Xiumei Wang; Department of Materials Science & Engineering, Tsinghua University, Beijing, Beijing, China.

Enamel is made up of enamel prisms separated by thin layer of organic sheaths. The mechanical properties of the prisms and the organic sheaths are obviously different from each other due to different compositions and microstructures. However, quantitative measurements of such differences have been a challenge in the past. The objective of this study is to accurately study the mechanical properties in the isolated domains within single enamel prism. The technique of nanoindentation combined with Atomic Force Microscopy (AFM) was employed to test the enamel specimens from mature human maxillary third molar. It was revealed that the nanohardness and elastic modulus of the sheaths were about 73.6% and 52.7% lower than those of the prisms. AFM topographies of the residual indent impressions also visually confirmed the differences. In addition to nanoindentation tests, the microstructures of enamel were carefully investigated in terms of hierarchical levels of organization to understand the structural reasons of the mechanical differences. We found a close relation between the variations of mechanical properties of enamel and its hierarchical structure. The analysis of the mechanical properties within enamel upon hierarchy is not only helpful to understand its unique property, but may also inspire ideas for the design of novel synthetic materials.

11:15 AM **BB3.7/L3.7**

A Nanoindentation Method for the Determination of the Initial Contact and Adhesion of Soft Materials. Yifang Cao¹,

Dehua Yang² and Wole Soboyejo¹; ¹Princeton Institute of Materials Sciences and Engineering (PRISM); Department of Mechanical and Aerospace Engineering, Princeton University, Princeton, New Jersey; ²Hysitron Inc., Minneapolis, Minnesota.

This paper presents a new method for determining the initial contact position and adhesion of soft polymeric surfaces that are relevant to microelectronics and biomedical systems. Unlike stiff/hard materials, for which depth-sensing indentation (DSI) devices (such as nanoindentation instruments) and scanning probe microscopes (particularly atomic force microscopes (AFM)) are capable of generating force versus displacement data, the deformation of soft materials is usually difficult to determine because it is strongly affected by specimen compliance and adhesion phenomena. These generally make it difficult to determine the initial contact position. In this paper, we propose a method for the determination of the initial contact point during nanoindentation. The method uses a combination of the Maugis-Dugdale adhesion theory and a non-linear least square fitting to obtain zero indentation offset distance, the transition parameter and contact radius at zero load directly. The work of adhesion and the reduced elastic modulus are then determined indirectly. The method is used to analyze the load-displacement characteristics of poly-di-methyl-siloxane (PDMS), which is a material that is being used increasingly in microelectronics and bio medical systems.

11:30 AM ***BB3.8/L3.8**

Strain-Stiffening in Semiflexible Biopolymer Gels.

Paul Albert Janmey¹, Cornelis Storm³, Fred C. MacKintosh² and Tom C. Lubensky¹; ¹University of Pennsylvania, Philadelphia, Pennsylvania; ²Vrije Universiteit, Amsterdam, Netherlands; ³Institut Curie, Paris, France.

Unlike most synthetic materials, biological materials often stiffen as they are deformed. This nonlinear elastic response, critical for the physiological function of some tissues, has been documented since at least the 19th century, but the molecular structure and the design

principles responsible for it are unknown. A structural feature of many biopolymer systems is that filamentous biopolymers tend to be much stiffer than synthetic polymers, with persistence lengths ranging from 50 nm for DNA to >400 nm for intracellular intermediate filaments and extracellular fibrin protofibrils to the much stiffer polymers like F-actin, collagen, and microtubules that have persistence lengths from 10 micron to > mm. Most biopolymers are also quite long, reaching lengths well over a micron both in vivo and in vitro. As a result, the networks formed by these polymers in aqueous solutions can be very dilute and still exhibit large elastic moduli. In many cases found in vivo and reconstituted in vitro, the mesh size of a biopolymer network is of the same order as the persistence length of the polymer, and theories to relate molecular architecture to macroscopic rheology based on those developed for rubberlike systems no longer apply and cannot predict the strain-stiffening seen in experiments. A new model for macroscopic elasticity based on the non-linear force-extension relation of a semi-flexible chain has been developed and compared with experimental measurements of a number of strain-stiffening biological hydrogels. This model accounts for strain stiffening in a wide range of molecularly distinct biopolymer gels formed from purified cytoskeletal and extracellular proteins. The good agreement of theory with experiment shows that systems of semi-flexible chains, such as filamentous proteins arranged in an open crosslinked meshwork, invariably stiffen at low strains without the need for a specific architecture or multiple elements with different intrinsic stiffnesses.

SESSION BB4: Nanoscale Geometries and the Influence of Shape

Chair: Dave Bahr

Wednesday Afternoon, March 30, 2005
Room 2016 (Moscone West)

1:30 PM *BB4.1
W. W. Gerberich

Abstract Not Available

2:00 PM BB4.2

Nanomechanical Size Effects in Silicon. Daibin Ge¹, Andrew M. Minor¹, Eric A. Stach², Sophi Ionova³, Michael J. Jackson³ and J. W. Morris, Jr.³; ¹National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California; ²School of Materials Engineering, Purdue University, West Lafayette, Indiana; ³Department of Materials Science and Engineering, University of California, Berkeley, Berkeley, California.

Lithographically-defined silicon structures make ideal templates for observing the changes in mechanical behavior of nanoscale structures. By fabricating an array of silicon wedges where the only variable among different samples was the size of the plateau at the top of the wedge, we reveal that the proximity of the surface in the smallest structures actually changes the deformation mechanism and leads to softening of the mechanical response. Through a combination of in situ nanoindentation in a TEM and ex situ nanoindentation with post-deformation TEM analysis we show that the dominant deformation mechanism during the indentation of silicon changes from phase transformation in the largest wedges to dislocation plasticity in the smallest wedges. The qualitative results are confirmed with conventional instrumented nanoindentation of the wedges that shows both pop-in displacement excursions and a significant softening trend with decreasing size in the smallest wedges. These results will be discussed in the context of size effects in other small systems, including metallic thin films.

2:15 PM BB4.3

Nanoconfinement Effects on the Mechanical Behaviors of Polymers. Jing Zhou and Kyriakos Komvopoulos; Mechanical Engineering, UC Berkeley, Berkeley, California.

Polymer materials are technologically important for nanomachining and nanostructuring to form advanced nanoelectronic or nano electro-mechanical systems (NEMS), which involves lots of mechanical issues, such as sub-100nm polymer resist for nanoimprinting, ultrathin PMMA for thermomechanical data storage. In addition nanoscale polymer thin films are cast onto the surface biomedical materials, e.g. artificial hip joint replacements, to improve biocompatibility and mechanical performance. However it is still not quite understood that how the polymer chain conformation microscopically affects their mechanical and thermomechanical behavior when the polymeric materials was confined in either 2-dimensional (thin films) or 1-dimensional (nanowires) structures. The continuum theory might fail when the size of polymer materials goes down to nanoscale and approaches the radius of gyration, which is statistically defined as the root mean squared distance between the centroid of the molecule and the each of its monomers. Under this condition, the conformation of

the molecule chain is perturbed by the nanoconfinements, the free surface and interface effects. In our studies, we start with a simplest model system — PMMA — which is completely amorphous in condensed phase and follows classic polymer chain dynamics and statistics. Nanoindentations are performed on 2D nanostuctured PMMA thin films with various thickness and 1D PMMA nanowire structures with various height and width all of which are close to the order of the radius of gyration. Reduced modulus and stiffness are extracted from nanoindentation data to study the correlation between chain conformation, glass transition and nanoscopic mechanical response. Polymer chain statistics theory based model is proposed to interpret the evolution of mechanical behavior with nanoconfinement of polymer molecular chains. This study is fundamentally important for addressing the new physics when polymer materials meets nanoscale and technologically useful for designing and engineering polymer materials for nanostructured mechanical systems.

2:30 PM BB4.4

Ideal Torsional Strength of Carbon Nanotubes. Elif Ertekin and Daryl Chrzan; UC Berkeley, Berkeley, California.

The recent developments towards carbon nanotube based rotational motors (Fennimore, Nature, 2003) indicate that nanoscale materials might eventually be incorporated into devices on a wider scale. Before this can be accomplished, an understanding of the mechanical response of carbon nanotubes to torsional strain is desirable. We use a first-principles density functional approach to explore the ideal torsional strength of carbon nanotubes. We show that the response of all single-walled carbon nanotube remains linear to large torsional strains before becoming unstable at a critical torsional strain. Near the instability, it is possible that Stone-Wales defects can be introduced. The response of the carbon nanotubes in a constant torsional strain rate experiment is described, and an estimate of the critical strain is obtained. Finally, because it is interesting to compare the mechanical behavior of a carbon nanotube to larger scale materials, we devise a technique to compare the torsional response of a pipe composed of embedded carbon nanotubes to pipes of other materials.

2:45 PM BB4.5

Interrupted Tubules in Crystalline Nanotube Ropes: Elastic Analysis. Moneesh Upmanyu¹ and James R. Barber²; ¹Engineering Division, Materials Science Program, Colorado School of Mines, Golden, Colorado; ²Mechanical Engineering Department, University of Michigan, Ann Arbor, Michigan.

We have performed a transversely isotropic, linear elastic analysis for an interrupted tubule (IT) in a cohesive, crystalline filamentous crystal. The axisymmetric elastic solutions for stresses, strains and displacements can be easily derived using the inherent anti-symmetry about the plane of IT-plane. We show that the solutions can be expressed in terms of two related strain potentials evaluated in different transformed coordinate systems. The displacements are long range, implying that these defects accumulate significant strain energy. Our elastic analysis can easily be extended to IT defects in highly anisotropic crystalline nanotube ropes (CNTR), where the coupled coordinate systems are necessarily real. We find that the stresses diverge within a dumbbell-shaped core region, and the core shape is sensitive to the tube radius. Finally, we also discuss registry dependent modifications to the analysis.

3:30 PM *BB4.6

The Nano-Bridge Testing Method for Single Individual Nanotubes or Nanowires. Yong Wang¹, Jian-Rong Li¹, Yafei Zhang² and Tong-Yi Zhang¹; ¹Department of Mechanical Engineering, Hong Kong University of Science and Technology, Hong Kong, Hong Kong; ²Research Institute for Micro/Nanometer Science and Technology, Shanghai Jiao Tong University, Shanghai, China.

In the present work, we report the progress in the mechanical characterization of a single individual nanotube or nanowire by the nanobridge test with an atomic force microscope (AFM). Theoretically, a formula has been developed based on continuum mechanics with the consideration of the substrate deformation, the adhesion between the AFM tip and the nanowire (or nanotube) and the adhesion between the nanowire (or nanotube) and the substrate. Experimentally, we have conducted the nanobridge test on SiC nanowires with diameters ranging from 18 nm to 50 nm on Si templates (substrates). The experimental results indicate that without considering the substrate deformation, the extracted Young's modulus would be about 25 % less than that with considering the substrate deformation. The Young's modulus of the SiC nanowires decreases as the nanowire diameter increases, which is well described by the strain gradient elasticity. Molecular dynamics simulations and high resolution transmission electron microscopy are being conducted to explore the mechanism of the diameter-dependent Young's modulus.

4:00 PM **BB4.7**

Controlled Synthesis and Manipulation of ZnO Nanorings and Nanobows. William L. Hughes and Zhong Lin Wang; Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

An experimental procedure is presented for controlled synthesis of ZnO nanorings and nanobows at high purity and large-yield. Atomic force microscopy manipulation of the nanostructures demonstrates their mechanical toughness and flexibility. Extensive bending of the nanorings and nanobows suggests an extremely high deformation limit with the potential for building ultra-sensitive electromechanical coupled nanoscale sensors, transducers, and resonators. [1] W.L. Hughes and Z.L. Wang "Formation of Piezoelectric Single-Crystal Nanorings and Nanobows", *J. Am. Chem. Soc.*, 126 (2004) 6703-6709. [2] W.L. Hughes and Z.L. Wang "Controlled synthesis and manipulation of ZnO nanorings and nanobows", *Applied Physics Letters*, Pending. [3] For information see www.nanoscience.gatech.edu/zwang

4:15 PM **BB4.8**

Self-Attraction among Aligned Au/ZnO Nanorods under Electron Beam. Xudong Wang, Christopher J. Summers and Zhong L. Wang; Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

As one of the most important direct band gap semiconductors, ZnO has recently attracted great interest in synthesizing various nanostructures and fabricating nanodevices. One of the techniques being investigated for the growth ZnO nanorod arrays is to use gold as catalyst particles. In this technique, Au acts as the nucleation site and is carried on the tip of the ZnO nanorod such that a natural metal-semiconductor junction (M-S) is formed. The vertical aligned Au/ZnO nanorods have an average thickness of ~ 20 nm and length ranging from 300 nm to 1 μ m. An interesting phenomenon of self-attraction among these nanorods under the illumination of an electron beam has been observed in a Scanning Electronic Microscope. The configuration of the contacted nanorods is very stable and can be preserved for months in atmosphere. The Au/ZnO metal-semiconductor junction model is proposed to explain this self-attracting effect, in which gold charges positively and ZnO rod charges negatively because of their different Fermi levels. Each Au/ZnO nanorod can be viewed as a dipole; the total force between two dipoles is attractive only if the nanorods have different lengths. Once the electrostatic attraction overcomes the elastic bending force, bending and contacting are possible. Thus, the self-attracting phenomenon is attributed to the interaction between the e-beam induced charge accumulation near the metal-semiconductor junctions for two nanorods of different length. A model calculation is presented to reveal the relationship between the electric interaction force and the bending force and explain the observed phenomenon. This discovery opens a new way to manipulate nanomaterials and control nanodevices and can be applied in electron beam, electric field or opto-electron related nano-triggers, nano-switches and nano-sensors. [1] For details please visit: www.nanoscience.gatech.edu/zwang

4:30 PM **BB4.9**

Mechanical Properties of CdSe Tetrapods. Liang Fang¹, Miquel Salmeron¹, Yi Cui^{2,1} and Paul Alivisatos^{2,1}; ¹Materials Science Division, Lawrence Berkeley Lab, Berkeley, California; ²Department of Chemistry, University of California-Berkeley, Berkeley, California.

CdSe tetrapods are novel nanoscale crystals with unique electric/optical properties, which makes them promising candidates for making nanocrystal based photovoltaic solar cells. However, their mechanical properties are still not well understood. We used atomic force microscopy to study their mechanical and electrical properties and try to discover the correlation between them. First of all, the AC mode images revealed that each arm of the tetrapods is about 150 nm long except the one that is along the surface normal (the vertical arm). They also showed that the tetrapods deposited on Si surfaces have already been pushed down by the capillary force caused by surface water layer. Additionally, the mechanical properties of CdSe Tetrapods were studied using force-volume technique. We were able to put the AFM tip right on the top of the vertical arm using this technique. We discovered that the tetrapods would undergo elastic deformation if the applied force was less than 52 nN. After we applied force more than 91 nN, the tetrapods would undergo plastic deformation and we started to observe the bending of the vertical arm. Applying a force more than 130 nN on top of the vertical arm would then completely destroy the tetrapods. Current Force Microscopy will be used to study the mechanical and electrical properties of tetrapods at the same time, which will be expected to give us more insight on the relationship between the electrical properties of CdSe tetrapods and their molecular structures.

SESSION BB5: Poster Session
Chairs: Dave Bahr and Greg Swadener
Wednesday Evening, March 30, 2005
8:00 PM
Salons 8-15 (Marriott)

BB5.1

Large-Scale Electronic Structure Calculation and Nanoscale Structure formed in Dynamical Brittle Fracture Simulation of Silicon. Takeo Hoshi^{1,2}, Ryu Takayama^{3,1}, Yusuke Iguchi¹ and Takeo Fujiwara^{1,2}; ¹Department of Applied Physics, University of Tokyo, Hongo, Bunkyo-Ku, Tokyo, Japan; ²CREST, JST, Motomachi, Kawaguchi-shi, Saitama, Japan; ³ACT-JST(JST), Motomachi, Kawaguchi-shi, Saitama, Japan.

Using large-scale electronic structure calculations, atomistic simulations of silicon cleavage are carried out with the sample sizes of more than 10 nm. The investigation is focused on the resultant nanoscale structures; surface reconstruction, step formation and bending in cleavage path [1-2]. For recent years, we have developed theories and program codes for large-scale electronic structure calculations [1-6]. Their common mathematical foundation is to calculate the one-body density matrix, in stead of one-electron eigen states. Test calculations were done with up to ten million atoms [2]. The method is applied to the cleavage process of silicon with a transferable Hamiltonian in the Slater-Koster (tight-binding) form [1,2]. The simulations are carried out under the external loads whose physical origin is the concentrated strain field at the crack tip. The results are analyzed from the quantum mechanical freedoms of electrons. (I) Since experiments reported only the (111) or (110) cleavage plane, the theory should explain why other surfaces do not appear. We focus the unstable cleavage process with (001) plane, due to the smallness of its surface energy. In smaller samples, with the size of less than 10 nm ($L < 10$ nm), the (001) cleavage plane is obtained, in which the asymmetric surface dimers appear. In larger samples ($L > 10$ nm), however, the (001) cleavage plane becomes fairly unstable with many step formations. (II) The (111) cleavage mode, the experimentally observed mode, is investigated. The formation of the pi-bonded (2×1) structure is commonly observed, as expected from experiments. Several step structures are also formed and compared to experiments. Due to the symmetry of (111) plane of the diamond structure, the steps are classified into the two types, known as [-211] and [2-1-1] types. (III) In several larger samples ($L > 10$ nm), the cleavage path is bent from the (001) plane into the (111) or (110) plane, which is consistent to the experimental preference of the (111) and (110) cleavage planes. These results give a theoretical investigation of brittle fracture beyond the traditional approach with surface energy. Among these results, the elementary processes are the drastic change of wavefunctions from the bulk (sp³-bonding) state into surface (non-sp³) ones, which is the origin of the mechanical property beyond the linear elasticity. In general, the nanoscale mechanics of silicon is governed by the energy competition between the bulk (sp³) term and the surface (non-sp³) term. The present results can be understood by the competitive mechanism with the typical length scale of 10 nm. [1] *J. Phys. Soc. Jpn.* 72, 2429 (2003); [2] preprint (cond-mat/0409142); [3] *J. Phys. Soc. Jpn.* 69, 3773 (2000); [4] *Surf. Sci.* 493, 659 (2001); [5] *J. Phys. Soc. Jpn.* 72, 2880 (2003); [6] *J. Phys. Soc. Jpn.* 73, 1519 (2004).

BB5.2

Effect of Viscous Grain-Boundary Sliding on Coble Creep of Polycrystalline Solids. Byung-Nam Kim, Keijiro Hiraga, Koji Morita and Hidehiro Yoshida; Fine-Grained Refractory Materials Group, National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

For steady-state deformation caused by grain-boundary diffusion, the macroscopic creep rate and the effect of viscous grain-boundary sliding in a polycrystal are analyzed by the energy-balance method in two and three dimensions. Furthermore, for a two-dimensional polycrystal of hexagonal microstructures, the stress distribution on grain boundaries and the effect of grain elongation are examined, while for a three-dimensional polycrystal consisting of space-filling polyhedral grains, the upper-bound and lower-bound creep rates caused by grain-size distribution are estimated. The maximum normal stress and the extent of stress concentration on grain boundaries are shown to decrease as the grain-boundary viscosity increases. For infinite viscosity and/or extremely small grain sizes, the distribution of the normal stress becomes uniform. The grain-grain interactions in polycrystals increase the degree of symmetry of diffusional field, resulting in a decrease of the effective diffusion distance. Meanwhile, both the viscous grain-boundary sliding and the grain-size distribution are found to decrease the creep rate. At decreasing grain sizes, the influence of the viscous grain-boundary sliding becomes increasingly important, which explains the recent experimental observations that the creep rates of nano-sized grains are much lower than those predicted by grain-boundary diffusion. The

present analysis reveals that the grain-size exponent is dependent on the grain size and the grain-boundary viscosity: the exponent becomes unity for small grain sizes and/or high viscosity, while it becomes three for large grain sizes and/or low viscosity.

BB5.3

AFM Characterization of the Surface Morphology of the Severe Plastic Deformed Copper. Rynno Lohmus¹, Irina

Hussainova², Lembit Kommel², Kristjan Saal¹, Ants Lohmus¹ and Hele Siimon¹; ¹Institute of Physics Univ of Tartu, Tartu, Estonia; ²Department of Materials Engineering, Tallinn University of Technology, Tallinn, Estonia.

Atomic force microscopy (AFM) techniques are increasingly used for studies of materials surfaces on micro - and nano - scales. AFM readily provides high resolution digitized images of surface features. In situ surface characterization helps to develop a better understanding of microstructure evolution of materials subjected to any kind of mechanical loading as well as to thermal treatment. The main objective of this study is to exploit the capabilities of AFM to accurately perform an analysis of the surface features of nano-structured copper subjected to a plastic deformation and provide the basis necessary for the development of a model describing the evolution of the topography that can be used for explaining the specific mechanical properties of nano-materials. For this purpose, AFM and SEM (scanning electron microscopy) have been used for the investigations of (i) surface morphology of severe plastic deformed pure copper; (ii) formation of slip bands and protrusions in strain controlled hard cycling visco-plastic deformation (HCV) and tension; (iii) influence of heat treatment on mechanical properties and structural characteristics of nanocrystalline copper. Keywords: Atomic Force Microscopy; Copper; Severe Plastic Deformation; Grain Boundary

BB5.4

Cyclic Deformation of Electrodeposited Nanocrystalline Nickel. Benedikt Moser^{1,2}, Timothy Hanlon^{2,3}, Sharvan Kumar⁴ and Subra Suresh²; ¹Materials Science and Technology, EMPA, Thun, Switzerland; ²Materials Science and Engineering, MIT, Cambridge, Massachusetts; ³General Electric Global Research, Niskayuna, New York; ⁴Engineering, Brown University, Providence, Rhode Island.

Nanocrystalline nickel has outstanding hardness, strength and wear properties, relative to pure microcrystalline nickel. Its exceedingly small grain size leads to deformation mechanisms unlike those observed in microcrystalline materials. The investigation of such deformation mechanisms is currently an area of active research. Most of these activities, including in-situ electron microscopy experiments and atomistic simulations, concentrate on monotonic deformation. A more comprehensive understanding of the cyclic deformation of nanocrystalline metals is important for potential structural applications and can also provide valuable insight into deformation mechanisms under monotonic loading conditions. Results of monotonic and cyclic tension tests of free standing electrodeposited nanocrystalline pure nickel foils will be presented. Stress-strain curves are carefully recorded and analyzed. The microstructure before and after deformation has been characterized by transmission electron microscopy (TEM) and the fracture surfaces have been investigated by scanning electron microscopy (SEM). In accordance with earlier studies we find that nanocrystalline nickel exhibits a positive strain rate dependence of the flow stress. A number of mechanisms have been proposed previously to explain such behavior. We present here, however, a purely dislocation based mechanism. A pronounced effect of cyclic frequency on the observed fatigue life in low cycle fatigue is detected. Several possible explanations, including creep fatigue interaction and environmental effects, will be discussed. Additionally, it will be shown that the material exhibits marked cyclic hardening, independent of the cyclic frequency employed. Direct evidence of this hardening in the form a decreasing strain amplitude and hysteresis loop width, as a function of the number of fatigue cycles, will be presented. This particularly important observation will be discussed on the basis of current understanding of the deformation mechanisms in nanocrystalline nickel. A mechanism based on grain boundary dislocation source exhaustion will be described to rationalize the observed behavior.

BB5.5

Measurements of Interface Thickness by Nano-Indentation: Numerically Calibrated Experimental Approach. Chunyu Yang and Ashraf Bastawros; Aerospace Engineering, Iowa State University, Ames, Iowa.

The interfacial fracture toughness and the adhesion strength of two dissimilar materials are governed by the thickness diffusion interface. A new testing methodology is implemented here to estimate the actual interface thickness from a series of nano indentation across the interface, under the same applied load. A numerical relationship is

developed utilizing 2-D finite element simulation to correlate the true interface thickness with the experimentally estimated apparent interface thickness, derived from the transition domain of a series of indents across the interface. A range of material-pairs property combinations are examined for Youngs modulus ratio, yield strength ratio and interface thickness of 0-100 nm. The proposed methodology and the numerically calibrated relationship are in good agreement with the true interface thickness measured by transmission electron microscopy.

BB5.6

The Relation between Sliding and Structure at Grain Boundaries in Nanoscale FCC Bicrystals. Frederic Sansoz¹ and Jean-Francois Molinari²; ¹Department of Mechanical Engineering, University of Vermont, Burlington, Vermont; ²Department of Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland.

Grain boundary (GB) sliding is a key mechanism in controlling the mechanisms of collective grain deformation and crystal plasticity in nanostructured films and coatings. This investigation provides new insights into the influence of GB structural units in the sliding behavior of nanoscale tilt GBs. A series of molecular simulation using the quasicontinuum method will be presented to understand the mechanical response at the nanoscale of bicrystals under simple shear. The energetics and mechanical strength of eighteen $\Sigma <110>$ symmetric tilt GBs and two $\Sigma <110>$ asymmetric tilt GBs were investigated in Cu and Al. Three modes of deformation were found to operate at the nanoscale, depending on the GB equilibrium configuration: GB sliding by uncorrelated atomic shuffling, nucleation of partial dislocations from the interface to the grains, and GB migration. The findings of this investigation can be drawn as follows: (1) the GB energy alone cannot be used as a relevant parameter to predict the sliding of nanoscale high-angle boundaries; (2) the E structural unit, which represents a vacancy-type defect found in the period of some Σ tilt GBs, is shown to be responsible for the onset of sliding by atomic shuffling; (3) GB sliding strength shows slight variations between the different interface configurations, but no apparent correlation with the GB structure.

BB5.7

Nano-Mechanical Analyses of Fe₂O₃+Al Energetic Nanocomposites Using Classical Molecular Dynamics. Vikas Tomar¹ and Min Zhou¹; ¹Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia; ²Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Classical molecular dynamics (MD) simulation is an important technique for structural integrity analyses of custom designed nanostructured materials and nano-sized systems such as nanowires and nanobelts. This research focuses on the classical MD simulation of nanostructured Fe₂O₃+Al energetic nanocomposites for nanomechanical analysis. A generic potential form is used to describe the behavior of Fe+Al+Fe₂O₃+Al₂O₃ system at a range of mechanical loading rates at different temperatures. The potential is able to describe bulk single crystal behavior of Fe, Al, Fe₂O₃, Al₂O₃ as well as interfacial transitions among them. Computational nanocomposites of Fe₂O₃+Al are generated to conform to the experimentally observed attributes of sol-gel and otherwise manufactured Fe₂O₃+Al energetic nanocomposites. Nanomechanical analyses including shock-wave propagation analyses and strength measurement of the nanocomposites are used to reveal the effect of the correlation between nanoscale phase morphology and applied loading on the desired mechanical and energetic attributes. The framework developed offers an important tool for nanoscale manufacturing and design on advanced nanocomposite materials.

BB5.8

Multiscale Modeling of Strengthening and Plastic Deformation Mechanisms in Aluminum-Based Amorphous Nanocomposites. H.T. Liu^{1,2} and Lizhi Sun¹; ¹University of Iowa, Iowa City, Iowa; ²Univ. of California, Los Angeles, California.

Al-based amorphous nanocomposites exhibit exceptional mechanical properties while preserving reasonable ductility. Although it is believed that the enhanced mechanical property is due to the existence of defect-free nanoparticles in the amorphous aluminum matrix, the underlying strengthening mechanism remains to be revealed in order to obtain a fundamental understanding of the material nanostructure-property relationship. Therefore, developing a multiscale constitutive model for nanocomposites is essential to obtain a fundamental understanding of their nanostructure-macro-property relationships. In this work we focus upon theoretical exploration of the mechanical constitutive behavior of amorphous nanocomposites in terms of a multi-scale approach starting from the nanostructure. Local heterogeneous stress field and deformation are calculated based on the concept of eigenstrain and equivalent inclusion method. The

overall elastoplastic constitutive model for amorphous nanocomposites is developed through homogenization averaging procedures. Explicit expressions of the effective elastic stiffness and yield strength of amorphous nanocomposites in terms of the constituent properties and nanostructures are obtained. An interlayer phase between nanoparticles and the amorphous matrix is experimentally observed and incorporated in the proposed model. The interlayer thickness is treated as a characteristic length scale. Thus, the particle size effect on the nanocomposite properties is particularly investigated within continuum nanomechanics framework. It provides direct determination of the intrinsic mechanisms of the nanocomposite structure-property relationship at the nanoscale.

BB5.9

Study of Critical Shell Thickness with Simulation of Strain and Structure in Nanocrystals. Youri Bae and Russel Caffisch; Mathematics, UCLA, Los Angeles, California.

In past experiments, the core/shell semiconductor nanocrystals have been synthesized and characterized. Since high quantum yield (QY) is crucial for use in optoelectronic devices, the synthesis of nanocrystals with high QY has been an important issue in recent nanotechnology, and experiments have shown the size dependence of QY. Thin shell growth on the core preserves its crystalline structure, resulting in lattice mismatch, causing strain on the interface between core and shell. This strain due to lattice mismatch had been suggested to be one of the explanations why the maximum QY is achieved for thin shells with thickness of 1-2 monolayers as well as the general observation that further shell growth reduces the QY. We have developed a powerful formula to model atomistic elasticity, which is suitable for computing this strain effect in epitaxial growth of thin films. Due to the large number of steps near the interface between core and shell, an atomistic strain calculation is needed to model and simulate the strain in thin films. In our research, we examine qualitatively and quantitatively the elastic energy density that arises from the nanocrystal model. We propose that the elastic energy density of nanocrystals is concentrated near interfaces between core/shell and that their maximal value as a function of shell thickness peaks with small shell thickness. We define this shell thickness as critical shell thickness and compare and contrast these results to known QY results from past experiments. Our main results are as follows. We examine the influence of the elastic coefficients and geometric parameters of the model, e.g. the effect of core radius and lattice mismatch of the core and shell, on critical shell thickness. We also show that the critical shell thickness is the same as that of quantum yield from the experimental results. Therefore, our atomistic elastic model has allowed us to investigate the instabilities of nanocrystals which result in its irregular growth with large lattice mismatch.

BB5.10

Computer Simulation of the Localization of Plastic Shear Events in Molecular Glasses. Qing Peng¹ and Marcel Utz^{1,2};

¹Physics Department, University of Connecticut, Storrs, Connecticut;

²Institution of Material Science, University of Connecticut, Storrs, Connecticut.

The mechanism of plastic deformation of molecular glasses, such as amorphous polymers, is not yet fully understood. A particularly vexing problem is the question of the length scale of the elementary processes of plastic relaxation. Using a novel approach based on Delaunay tessellation of the simulation cell, we have studied the localization of atomic strain in discrete relaxation events during plastic deformation of glassy materials, as a function of the chain length of linear molecules. The strain in such relaxation events is highly localized in regions of atomic dimensions. Their size depends on the chain length of the molecules that make up the glass. This may provide a framework to understand recent experimental results on the dependence of the shear activation volume on the entanglement density of glassy polymers (Ho, Govaert, and Utz, *Macromolecules* 36, 7398, (2003)). The implications of our simulation results for a universal theory of plasticity of glassy polymers will be discussed.

BB5.11

Atomistic Modeling of Hardening Mechanism of Nanometer Sized Co Precipitates in Cu Crystals. Jae-Hyeok Shim^{1,2}, Hyon-Jee Lee¹ and Brian D. Wirth¹; ¹Department of Nuclear Engineering, University of California, Berkeley, Berkeley, California; ²Nano-Materials Research Center, Korea Institute of Science and Technology, Seoul, South Korea.

The Cu-Co system is a model precipitation hardened alloy, because nanometer sized coherent Co precipitates form with the cube-on-cube orientation relationship with respect to the Cu matrix during the initial annealing stage. It is well known that Cu-Co alloys exhibit an anomalous temperature dependence of the critical resolved shear stress. In this presentation, we present atomistic molecular dynamics

simulations that investigate this anomalous mechanical behavior by investigating the dislocation bypass mechanism at Co precipitates in Cu. Large-scale molecular dynamic simulations of the interaction between dislocations (edge and screw) and precipitates in Cu-Co alloys have been performed using 3-5 million atoms. Coherent Co precipitates show different resistance to the leading and trailing Shockley partial dislocations. A temperature dependent bypass mechanism of the trailing partial dislocation is observed irrespective of the dislocation type. At low temperatures, the trailing partial bypasses with the Orowan mechanism, whereas it bypasses with the shear mechanism at high temperatures. The combination of a temperature dependent bypass mechanism and the dynamic (inertial) effect of phonon drag explain the anomalous temperature dependence of the critical resolved shear stress in Cu-Co alloys. The change in bypass mechanism with temperature is well described by an analytic dislocation - dispersed obstacle model, developed to describe the new strengthening mechanism. The critical resolved shear stress behavior predicted by this model is compared with the behavior obtained by molecular dynamics simulations and experiments.

BB5.12

A Continuum Investigation of Deformation Mechanisms in Nanocrystalline Metals. D. H. Warner and J. F. Molinari; Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland.

A grain level micromechanical model of nanocrystalline metals was developed and utilized within a finite element framework. By using previous atomistic calculations that examined the mechanical behavior of several tilt grain boundaries, constitutive laws were developed to describe the local grain boundary behavior within the finite element model. In order to account for grain boundary sliding under compression, an application specific contact/adhesion algorithm was developed. Intragranular plasticity was modeled using a size-dependent crystal plasticity methodology. Comparison of the finite element calculations, with previous experimental and molecular dynamics results from literature, yielded useful insights into the deformation mechanisms which operate at these small grain sizes. Although not explicitly included in the model, a correlation between grain boundary sliding and intragranular plasticity was observed. A change in the localization of plastic strain was noted at very small grain sizes and was found to be dependent on the grain size distribution.

BB5.13

AlN-TiN Nano Thin Film Material Characterization through 3-D Non-Linear Finite Element Modeling. Cindy K. Waters¹, Rahul Gupta², Ajit Kelkar Kelkar², Jag Sankar¹ and Dhananjay Kumar¹; ¹Mechanical Engineering, NCA&T State University, Greensboro, North Carolina; ²NCA&T State University, Greensboro, North Carolina.

The purpose of this research is to investigate the hardness and elastic properties of multiple layer aluminum nitride (AlN) and titanium nitride (TiN) thin films of various periodicities, deposited on silicon (111) substrates. These films were grown using the PLD method. To understand the deformation and damage mechanics of these thin films several techniques are being developed and applied. The properties of thin film can be different from those of bulk materials even when the chemical composition of the thin film and the bulk material are identical. In general, indentations with contact depths of less than 10-20% of the film thickness are desired in order to attain the intrinsic film properties and to circumvent the substrate effect. Finite element analysis (FEA) has been used for analyzing the indentation problem. FEA provides insight into the contact area, plasticity, and the effects of a differentiated material substrate on the load displacement behavior of a thin film. FEA requires that the indented body act as a continuum. Thin film thickness is not uniform and varied between 200 and 300nm. 100 mN force was applied through the nanoindenter tip. Multiple cases were studied with mesh edge dimension descending continuously from 0.1 mm to 1.0 micron level. The thin film material was assumed to be initially stress free, which is not the actual case. Both film and substrate are assumed to be homogeneous and isotropic with perfect elastic-plastic behavior. Correlation and convergence was observed with experimental results through elastic-plastic non-linear analysis. It was observed that the mesh size has a significant effect on the prediction of the through the thickness displacements. Initially, only linear elastic analysis was performed, and model over predicted through the thickness displacements. However when the FEA including material and geometric nonlinearity was performed, there was close agreement between the experimental results and the finite element results. Although the film thickness in the present simulations and actual experiments was only 250 nm, the loading on the thin film was continued until the through the thickness displacements exceeded 250 nm. The force-deflection loading-unloading curve generated from non-linear FEM analysis is in good agreement with experimental results when the Elastic Modulus is set to the experimentally

obtained value. Current computational resources limit us to simulations consisting of roughly one million atoms which correspond to the substrate size of approximately 30 nm x 30 nm x 19 nm. A challenge remains when using continuum mechanics dimension models with an atomistic modeling approach based on computation time and file size. Research is ongoing to account for interfacial stress at the boundary of the thin film and the substrate through nonlinear large deformation based LS-DYNA finite element simulation.

BB5.14

Understanding Indenter Geometry and Grain Size Effects in Nanoindentation Experiments. Suman Vadlakonda¹, Reza A. Mirshams¹ and Raja Mahesh Pothapragada²; ¹Materials Science and Engineering, University of North Texas, Denton, Texas; ²Mechanical Engineering, University of North Texas, Denton, Texas.

Nanoindentation has been a widely used technique for determining mechanical responses in materials. The indentation of metallic materials has been a topic involving considerable complexity due to factors like, indenter geometry, tip size, and material strain hardening characteristics[1]. Apart from these characteristics, the effect of grain size and number of dislocation sources play an important role in determining mechanical properties. An attempt is done here to understand relation between geometry of the indenter and grain size of the sample. The effect of pileup during indentation becomes a factor in understanding mechanical response. The relation between pileup height and grain size is also studied in relation to the indenter geometry. This paper relates nanoindentation experiments on polycrystalline Ni, W and Fe films of known texture and thickness.

BB5.15

Mechanical Properties of Nanostructured Al-5083/SiC Composite Prepared by Cryomilling. Feng Tang¹, Masuo

Hagiwara², Jichun Ye¹ and Julie M. Schoenung¹; ¹Chemical Engineering and Materials Science, University of California at Davis, Davis, California; ²National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

A composite with a nanocrystalline Al-5083 matrix reinforced with nano-sized SiC particles was prepared via powder cryomilling and consolidation processes. Tensile and compressive tests were conducted and the results were compared with those from the nanocrystalline Al-5083 alloy without SiC reinforcement. Commercially available gas-atomized Al-5083 powder was mixed with 5 wt.% nano-sized β -SiC powder with an average particle size of 20 nm, and ball milled in liquid nitrogen in order to obtain a powder with a nanocrystalline Al-5083 matrix and nano-sized SiC dispersoids. The cryomilled powder was degassed at 400°C for 20 h and HIPped at 400°C under 172 MPa for 2 h. The composite was then heated to 400°C and rolled into a 12 mm square bar. SEM observation showed that the nano-sized SiC particles were distributed homogeneously in the composite and that the grain size in the matrix was believed to be less than 100 nm after hot rolling (Precise grain size determination is presently under investigation with TEM). Cubic compressive specimens with a side length of 5 mm and tensile specimens with a gauge length of 14 mm and a cross-section of 4 mm x 2 mm rectangle were cut from the rolled bar in a direction parallel to the rolling direction. Thus, the testing loads in the compressive and tensile tests were applied along the rolling direction. The strain rate in both compressive and tensile tests was 1×10^{-3} . The compressive tests showed that the 0.2% proof stress of the composite was 905 MPa. This value is more than 2.5 times higher than the value of 334 MPa [1] obtained for the cryomilled Al-5083 alloy with a grain size of 30 nm but without particulate reinforcement. However, the ductility of the composite was poor and the elongation-to-fracture in the compressive test was only 0.2%. The tensile specimens fractured at 811 MPa in the elastic deformation stage. Our experimental results indicate the feasibility of enhancing the strength of the nanostructured Al-5083 alloy by dispersing the nano-sized SiC particles into it. Further research is in progress to improve the ductility while maintaining the high strength in the nanostructured composite. The Office of Naval Research provided the financial support for this research. [1]. V. L. Tellkamp and E. J. Lavernia, *Nanostructured Materials*, 12 (1999), p. 249.

BB5.16

A Study on the Hydration Behavior of Nanoconcrete Analyzed by NMR. Wei-Ming Hou, Ping-Kun Chang and Jiann-Tsair Chang; Civil Engineering, Vanung University, Chung Li, Taiwan.

This study investigated the hydration behavior of nanoconcrete by means of NMR, LOI (loss of ignition), MIP, and Concrete Resistivity Meter. The results indicated that the cement with nanopowders had already produced the pozzolanic reaction during its early age, which was different from those with fly ash and slag. In the late age, the effect of reaction was more obvious and the content of Ca(OH)₂ within concrete was reduced evidently, which improved the property

of the interface area. Furthermore, because of the microminor particles, the capillary pores and gel pores were filled up efficiently, enhancing the microstructure of concrete to the level that the materials were averagely densified, which had great improvement on the macro-properties of concrete.

BB5.17

Nonuniformity of Deformation in Nanocrystalline Materials. Vitaly Shpeizman, Ioffe Physicotechnical Institute of the Russian Academy of Sciences, St. Petersburg, Russian Federation.

The well-known nonuniformity of deformation in various materials observed both in a multitude of micro- and macroscopic studies is caused by heterogeneity of the material structure and by nonuniformity of the deformation process itself. Modern high-resolution methods of strain and strain rate measurements make it possible to reveal the variations of strain rate on the base of very small strain increments. The interferometric method used in this work allows to record creep curves and to measure creep rates through each 0.15 micron changes in the length of a sample with an error of less than 5 %. In our previous works on polymers we have shown that creep curves consist of repeated sections with relatively high and low velocities and that the period in these strain rate oscillations corresponds to the length of small-scale microfibrils. In the present work we have studied aluminum, copper and other metals with various grain sizes. Special attention was paid to materials with grain sizes from 0.5 to 1 micron that were obtained by multiple equichannel angular pressing (ECAP). Microplastic deformation was observed in all materials at very small stresses which were sometimes less than 10 % yield points, but in metals after ECAP the strain rate was ten and more times lower in compare with large grain metals and with single crystals. Two periods of strain rate oscillations in coarse-grain metals were found. One of them was close to the grain size and remained constant with deformation, and the second period was considerably lower than the grain size and decreased with deformation. By contrast, the only period in ECAP metals was more than or equal to a mean grain size value and changed slightly with deformation. The model of deformation was suggested which connected the period of strain rate oscillations with characteristic sizes determined by initial structure or by conditions of deformation. Usually, the grain size and the distance between any obstacles on the way of dislocations (impurity atoms, crossing of slip planes, etc) are taken into account as a characteristic structure sizes for deformation in crystals. It follows from our results that the free movement of dislocations through a whole grain or grain boundary deformation is an elementary deformation act for ECAP metals, sometimes only the largest grains from grain-size distribution being involved in an initial stage of deformation. By measuring the period of strain rate oscillations we can follow the grain size evolution at different stages of deformation or after any thermal and mechanical treatment of ECAP metals.

BB5.18

Fabrication of High Strength ZrO₂-Spinel Nano-Composite. Koji Morita, Keijiro Hiraga, Byung-Nam Kim, Hidehiro Yoshida and Yoshio Sakka; National Institute for Materials Science, Ibaraki, Japan.

Nanocrystalline ceramics have received considerable attention because the nano-ceramics can exhibit excellent mechanical properties as compared with conventional submicron-grained ceramics with grain sizes of $d > 300$ nm. The nano-ceramics have potential for use in structural applications. The present study was therefore performed to fabricate nanocrystalline ceramics with $d < 100$ nm. In order to fabricate nano-ceramics, high-energy ball-milling and spark-plasma-sintering (SPS) techniques were employed. Tetragonal ZrO₂ powder mixed with 30vol% spinel powder was ball-milled for 50-400 h to obtain much finer powders. The milled powders were sintered with a SPS machine. The powder mixtures were placed into a graphite die and pressed at 70 MPa. By applying the loading, the powders were rapidly heated up to 1573 K and then held at the temperature for 5 min. Using ball-milling process, nanocrystalline ZrO₂-spinel powders can be fabricated from the sub-micrometer sized powders of ≈ 300 nm. After 400 h ball-milling, an amorphous-like phase was observed among nanocrystalline ZrO₂ and spinel particles of $d < 10$ nm. From the nanocrystalline powder, a fully dense nanocrystalline ZrO₂-based composite with grain sizes of < 90 nm were successfully developed using the SPS technique. The nano-composite exhibits excellent mechanical properties, including Young modulus, hardness and fracture strength. In particular, the fracture strength increased with decreasing grain size: the maximum strength is about 2.5 times higher than that of submicron-grained materials.

BB5.19

Fabrication Method of Metallic Closed Cellular Materials Containing Organic Materials. Satoshi Kishimoto and Norio Shinya; Materials Research Laboratory, National Institute for Materials Science, Tsukuba, Ibaraki, Japan.

A metallic closed cellular material containing organic materials for damping systems has been developed. Powder particles of polymer coated with a Ni-P alloy layer using electro-less plating or Al alloy using physical vapor deposition were pressed into green pellets and sintered at high temperatures in vacuum or a spark plasma sintering method. A metallic closed cellular material containing organic material that cell size was sub-micrometer or micrometer was then fabricated. The density of this material is smaller than that of other structural metals. The compressive tests and internal friction measurements were carried out to measure the mechanical and damping properties of this material, respectively. The results of the compressive tests showed that this material had the different stress-strain curves among the specimens that had different thickness of the cell walls and the sintering temperatures of the specimens affect the compressive strength of each specimen. The stress-strain curve of each specimen had a long plateau region and it seems that the energy absorbing capacity of this material was very high. The damping tests showed that the internal friction of this material was very high. These results indicate that this metallic closed cellular material containing organic materials can be utilized as energy absorbing material and passive damping material.

BB5.20

Calculation of X-Ray Spectra for Nanocrystalline Materials.

Peter M. Derlet, Stefan Brandstetter, Antonio Cervellino and Helena Van Swygenhoven; ASQ/NUM, Paul Scherrer Institution, Villigen PSI, Switzerland.

The use of x-ray diffraction (XRD) techniques has played a central role in the microstructural characterisation of nanocrystalline materials allowing the determination of mean grain size, grain size distribution and root-mean-square strain through peak profile analysis. There is however no detailed information on how structural parameters other than grain size influence the peak profile in nc-GB networks. More recently, XRD has been used to study the plastic deformation properties of nc-electrodeposited Ni via in-situ deformation in the Swiss synchrotron Light Source revealing a reversible peak broadening upon unloading and therefore no permanent accumulating dislocation network. Additionally, this method allows the measurement of Young's modulus simply from the peak position as function of the applied stress without the necessity to measure strain. Motivated by these experimental results we present a method to calculate the two-theta X-ray diffraction profiles derived from multi-million atom computer generated nanocrystalline Ni systems as a function of both elastic and plastic loading conditions, and this for a variety of grain boundary network structures with different amounts of intrinsic and extrinsic grain boundary dislocations. In addition to mean grain size and root-mean-square inhomogeneous strain, peak position as a function of applied stress is also investigated to obtain information about elastic properties. These quantities are then compared with experimental results.

BB5.21

Fracture Toughness Properties of Nanostructured Ferritic Alloy. Mikhail A. Sokolov, David T. Hoelzer, Michael K. Miller and Roger E. Stoller; Metals and Ceramics, ORNL, Oak Ridge, Tennessee.

The ferritic-martensitic (FM) steels are the primary candidates for a fusion power plant first wall and blanket structure. However, their utilization is limited to temperatures of approximately 600°C due to inferior tensile and creep strength at higher temperatures. Oxide-dispersion strengthening (ODS) is a well known process to improve the strength and creep properties of ferritic steels. Moreover, recent advances in manufacturing and understanding of the strengthening mechanisms have resulted in the creation of a new type of materials, namely, nanostructured (NS) ferritic steels. Significant improvement in strength and creep properties have been achieved of these NS steels compared to the conventional ODS steels. Elevated temperature strength in these steels is obtained by a high number density of ultra-fine, nanometer-scale complex titanium-yttrium-oxygen particles dispersed in a ferritic matrix. The NS steel investigated in this study was prepared by mechanically alloying a pre-alloyed Fe-12.3%Cr-3%W-0.39%Ti (wt.%) powder with ~20-nm size 0.25wt.%Y₂O₃ powder. The ultrafine scale microstructure of the NS ferritic alloy (12YWT) has been characterized by atom probe tomography (APT). APT revealed the presence of a high number density, 1.4x10²⁴/m³, of 2-4 nm diameter Y-, Ti-, and O-enriched particles. Tensile tests were performed at different temperatures from room temperature to 800°C. This 12YWT alloy exhibited a high room temperature yield strength of ~1300 MPa and ultimate strength of ~1400 MPa. Yield strength decreased as the test temperature increased, but remained higher relative to conventional FM steels. Fracture toughness characterization of the 12YWT alloy was performed with 3-point slow bend testing. Specimens were machined in the L-T orientation. The specimens were fatigue precracked before testing to a ratio of the crack length to specimen width of about 0.5. Tests were conducted at -50°C, 23°C,

50°C, 100°C, 500°C, and 600°C. The temperatures were maintained within ±2°C during the tests. The unloading compliance method was used for measuring the J-integral. All specimens tested up to 50°C failed by brittle instability. Fracture toughness, K_{Jc}, varied from 41 to 84 MPa√m. The ductile-to-brittle transition fracture toughness temperature, T_o, of 12YWT alloy was determined to be 102°C. This is a high absolute value of T_o compared to a T_o value of about -100°C for conventional FM steels such as F82H. Research supported by the Office of Nuclear Energy, Science and Technology (I-NERI 2001-007-F), and at the SHaRE User Center by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

BB5.22

Characterization of Ion Beam Synthesized Germanium Nanocrystals Embedded in a Sapphire Matrix. Qing Xu^{1,2}, Ian D. Sharp^{1,2}, Diana O. Yi^{3,2}, Christopher Y. Liao^{1,2}, Jeffrey W. Beeman², Kin Man Yu², Joel W. Ager², Daryl C. Chrzan^{1,2} and Eugene E. Haller^{1,2}; ¹Department of Materials Science and Engineering, University of California, Berkeley, Berkeley, California; ²Materials Sciences Division, Lawrence Berkeley National Laboratory, Berkeley, California; ³Applied Science and Technology Graduate Group, University of California, Berkeley, Berkeley, California.

Isotopically pure ⁷⁴Ge nanocrystals, between 5 nm and 10 nm in diameter, have been fabricated in a sapphire matrix via ion implantation followed by thermal annealing at temperatures above 980 °C. Properties of these nanocrystals are compared directly to those of nanocrystals that have been formed by germanium implantation into amorphous silica. Evaluation of transmission electron micrographs and Raman spectra from nanocrystals embedded in crystalline and amorphous matrices provides significant information about the matrix/nanocrystal interactions in both systems. Transmission electron microscopy reveals that nanocrystals embedded in sapphire are faceted, whereas those embedded in amorphous silica are spherical and often twinned. Our previous work has shown that nanocrystals embedded in silica can exhibit significant matrix-induced compressive stresses of up to 1.8 GPa. [1] The stress can be relieved via post-growth thermal annealing and a diffusive stress-relaxation mechanism has been identified. Ge nanocrystals formed in sapphire are under even higher stresses, which can approach 4 GPa, relative to bulk. Moreover, post-growth annealing at temperatures up to 1200 °C does not relax the stress, which we attribute to the lower self-diffusivities of the matrix atoms in sapphire as compared to silica. Possible mechanisms for stress generation during growth in sapphire, which include solidification of liquid germanium clusters upon cooling from the growth temperature and direct aggregation pressure arising from growth rates exceeding the rate of cluster accommodation by the matrix, will be discussed. [1] D. O. Yi, et al., Mat. Res. Soc. Symp. Proc. Vol. 818, P8.16.1 (2004).

BB5.23

Matrix Modification of Carbon-Carbon Composites with Carbon Nanofibers. Uday K. Vaidya¹, Rahul Jain² and Anwarul Haque³; ¹Materials Science & Engineering, University of Alabama at Birmingham, Birmingham, Alabama; ²Materials Science & Engineering, University of Alabama at Birmingham, Birmingham, Alabama; ³Aerospace and Engineering Mechanics, University of Alabama, Tuscaloosa, Alabama.

Carbon-carbon composites (CCCs) are known for their extremely high thermal stability and high thermal shock resistance. Typical usages of CCCs are in aircraft disk brakes and nozzle cones of re-entry space vehicles. The processing of CCCs can be divided into three stages: Curing, carbonization, and densification. Carbonization is concomitant with volatile emanation, void formation, microcracks formation, and sometimes delaminations. Voids and cracks are formed because of volatile egression and differential contraction between fiber and matrix, which debilitate the composite. In this study, we studied the influence of carbon nanofibers (CNFs) when mixed with phenolic matrix precursor, on the microstructure and interlaminar shear strength (ILSS) of the uncarbonized (as cured) and carbonized composite samples. Different loadings (0 %, 2 %, and 5 %) of CNFs, in conjunction with various percentages of surfactant (0 %, 12.5 %, and 50 %) treated carbon fabric reinforcement, were used for this study. The rationale for treating the C-fabric with surfactant was to vary interfacial conditions of fiber-CNF-matrix bonding. This paper emphasizes the work done in processing of carbon-carbon CNF composites, and the influence of surfactant and various percentages of CNFs on the resulting microstructure and ILSS. The use of CNFs provided a bridging mechanism for matrix microcracking and reduced matrix shrinkage that occurs during first carbonization. At the as-cured and carbonized stages, 2 % CNFs proved to be the optimal loading yielding highest ILSS values of ~ 40 MPa and 6 MPa respectively.

BB5.24

Dislocation - Radiation Obstacle Interactions in Structural Metals under Irradiation. Hyon-Jee Lee¹, Jae-Hyeok Shim^{1,2},

Jamie Marian³ and Wirth D. Brian¹; ¹Department of Nuclear Engineering, UC Berkeley, Berkeley, California; ²Korea Institute of Science and Technology, Seoul, South Korea; ³Lawrence Livermore National Laboratory, Livermore, California.

The interaction between moving dislocations and nanometer-sized radiation defect clusters controls the yield strength, ductility and work localization behavior of structural materials under irradiation. Using large-scale molecular dynamics (MD) simulations, enabled by increasing powerful and inexpensive high performance parallel computers with semi-empirical inter-atomic potential, we can directly study atomistic processes and determine the sequence-of-events controlling dislocations and obstacle interactions. Such information is crucial in understanding the relationship between the mechanical and structural properties and should be incorporated in developing physical models to better predict the mechanical properties under various operating conditions. Further, it can be used to design new structural materials with superior properties to retard degradation process under irradiation. To this end, we investigate the atomic-scale interactions of dislocations with the commonly observed radiation obstacles using MD simulation methods. From TEM experiments, a high number of stacking fault tetrahedra (SFT) are observed in face centered cubic metals (FCC), while dislocation loops or voids are observed in body centered cubic (BCC) metals. To consider different types of radiation obstacles, FCC Cu and BCC Mo and Fe are chosen for this study. For FCC Cu, we examine factors such as dislocation type, temperature, SFT size, dislocation velocity and the interaction geometry to qualitatively and quantitatively understand the dislocation and obstacle interaction mechanism. For BCC Mo and Fe, considering the unique non-planar core structures of BCC screw dislocation, we first report the behavior of screw dislocation motion as a function of temperature and applied shear stress. Then, we introduce defects into the system and observe their interaction behavior with screw dislocations in both dynamic and static conditions.

SESSION BB6/EE6: Joint Session: Linking Length Scales in the Mechanical Behavior of Materials
Chairs: John Balk and Daniel Weygand
Thursday Morning, March 31, 2005
Room 2016 (Moscone West)

8:30 AM *BB6.1/EE6.1

Interplay between High-temperature Deformation and Grain Growth in Nanocrystalline Materials by Hierarchical Multiscale Simulation. Dieter Wolf¹, Andrew Haslam², Vesselin Yamakov³, Dorel Moldovan⁴, Rong Ding¹ and Simon Phillpot⁵;

¹Materials Science Div., Argonne Natl Lab, Argonne, Illinois; ²Dept. of Chem. Engrg., Imperial College, London, United Kingdom; ³National Institute of Aerospace, Hampton, Virginia; ⁴Dept. of Mechanical Engrg., Louisiana State University, Baton Rouge, Louisiana; ⁵Dept. of Materials Science and Engrg., University of Florida, Gainesville, Florida.

Molecular-dynamics simulations have been used to elucidate the intricate, highly non-linear coupling between grain-boundary diffusion creep and grain growth in nanocrystalline metals. We demonstrate how the materials-physics based insights into the underlying deformation and grain-growth mechanisms extracted from these simulations can be rigorously incorporated into a mesoscopic simulation model, thus overcoming the length and time-scale limitations inherent to the MD approach. The objects evolving in space and time in the mesoscale simulations are the discretized grain boundaries and grain junctions rather than the atoms themselves, involving characteristic length and time scales governed by grain-boundary processes and parameters rather than atom vibrations. This then enables analysis of the intricate interplay between grain growth and grain-boundary diffusion creep for a system containing a large number of grains with arbitrary sizes. Work supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under Contract W-31-109-Eng-38.

9:00 AM BB6.2/EE6.2

Atomistic Modelling of Interactions between Lattice Dislocations and Grain Boundaries in Body-centered Cubic Transition Metals. Matous Mrovec¹, Christian Elsaesser¹ and Gumbsch Peter^{1,2}; ¹Fraunhofer-Institut fuer Werkstoffmechanik, Freiburg, Germany; ²Institut fuer Zuverlaessigkeit von Bauteilen und Systemen, Universitaet Karlsruhe, Karlsruhe, Germany.

With grain sizes decreasing towards the nanometer scale, the plastic deformability of polycrystalline metals is increasingly controlled by

interactions between lattice dislocations and grain boundaries. In order to elucidate atomic-level mechanisms and processes of such interactions, computer simulations were carried out for atomistic models of dislocations interacting with boundaries in body-centered cubic (bcc) transition metals. The interatomic interactions were described by real-space bond-order potentials (BOP) derived from tight-binding electronic-structure theory [1]. These recently developed BOP have been applied already with success to simulate atomistic core structures of individual screw dislocations [2] and twin boundaries [3] in molybdenum, tungsten and other bcc metals. In this contribution, BOP simulation results will be presented for atomistic structures and mechanical responses of tungsten bicrystal models containing a twin boundary and a screw dislocation. [1] M. Mrovec, PhD Thesis, University of Pennsylvania, Philadelphia (2002). [2] M. Mrovec, D. Nguyen-Manh, V. Vitek, D. G. Pettifor, Phys. Rev. B 69, 095115 (2004). [3] T. Ochs, C. Elsaesser, M. Mrovec, V. Vitek, J. Belak, J. A. Moriarty, Phil. Mag. A 80, 2405 (2000).

9:15 AM BB6.3/EE6.3

The Nature of Slip in Deformation Simulations of Metallic Nanocrystalline Materials. Helena Van Swygenhoven, Anders Froseth and Peter M. Derlet; ASQ/NUM, Paul Scherrer Institution, Villigen PSI, Switzerland.

Slip in molecular dynamics simulations of fcc nanocrystalline materials is mediated by partial dislocations nucleated and absorbed at grain boundary regions. The ultimate nature of this slip, whether it be extended partials, full dislocations or twin faulting has recently been shown to depend on the general planar fault energy curves of the corresponding metal. In order to fully understand the detailed process of full dislocation activity as observed in MD simulations of nc-metals a more elaborate theory based on the dislocation nucleation process, the curved character of the dislocation, the GB structure of the nucleation site and the interactions of the dislocations with the GB has to be developed. In the present work we show that the splitting distance of the dissociated full dislocation cannot account for the correct nucleation physics of the leading and trailing partial dislocations. Even once both partial dislocations are nucleated, the local stress distribution plays an important role in the separation of the partials. Furthermore, by means of a detailed temporal output of energy, crystallinity and local averaged stress, we present a detailed analysis of the stress distribution and evolution in the GBs during emission of the dislocations allowing to pin down the important parameters of the nucleation and absorption processes of dislocations from GBs. We request that this submission be considered in the joint session between BB and EE

9:30 AM BB6.4/EE6.4

Developing Experimentally-Tuned Grain Boundary Networks for use in Molecular Dynamics Simulations. Anders Froseth, Peter M. Derlet and Helena Van Swygenhoven; ASQ/NUM, Paul Scherrer Institution, Villigen PSI, Switzerland.

It is well known from experimental observations that not only the grain size but also the type of grain boundary (GB) network greatly influences the mechanical response of nanocrystalline (nc) metals. Detailed information on an optimal GB network is however lacking. Until now, Molecular Dynamics (MD) deformation simulations of 3D-nanocrystalline metals have mainly been performed for a relatively small number of grains using the Voronoi construction and random grain orientations. We have developed a method using a combined Voronoi/Delaunay technique to geometrically construct samples with a pre-selected number of special GBs. Motivated by TEM observations of nc materials we employ this method to fine-tune simulated GB networks to more realistically represent known experimental structures. We present results for a series of nc-Ni samples containing 200 grains with a pre-selected number of either special symmetric low angle boundaries, or carefully selected Coincidence Site Lattice (CSL) boundaries. We demonstrate how special GB networks respond differently to plastic deformation compared to GB networks containing random grain orientations, give details on the activated deformation mechanism and draw conclusions concerning experimental observations. We request that this submission be considered for the joint session between EE and BB

9:45 AM BB6.5/EE6.5

The Impact of Length and Time Scale Limitations on Solute Drag Theory During Experiment and Modelling of Recrystallization in Aluminum Alloys. Mitra L. Taheri¹, Eric Stach², Velimir Radmilovic³, Hasso Weiland⁴ and Anthony D. Rollett¹; ¹Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, Pennsylvania; ²Materials Science and Engineering, Purdue University, West Lafayette, Indiana; ³National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California; ⁴Alcoa Technical Center, Alcoa Center, Pennsylvania.

The effect of solutes on the migration of boundaries has long been a topic of investigation due to its tremendous impact on its applications in materials science, both industrial and academic. The lack of measurement of the solute drag effect can be attributed in part to the limitations of current imaging capabilities of electron microscopes. Thus, there exists a limited amount of experimental investigation with respect to solute drag, all of which has been performed using scanning and transmission electron microscopy, regardless of their respective inadequacies. Evidence of both solute drag as well as differences in growth mechanisms of certain boundary types has been noted in both current literature and the results presented herein for an Aluminum-Zirconium alloy system. Recently, there have been indications of the limitations of scanning electron microscopy on the length and time scale of these experiments. New theoretical developments suggest that boundary motion should be jerky. This suggestion emerged from extensive computer simulation, together with new theory, and is supported by results from in-situ annealing experiments within a scanning electron microscope. In response to these findings, in-situ annealing experiments within a transmission electron microscope have shown a stark contrast between solute segregation at small scales and large scales; these experiments were coupled with Z contrast imaging and energy dispersive spectroscopy to locate solute segregation. Specifically, Zr was found to segregate to Al boundaries at grain sizes of approximately 2 microns and not at boundaries for grains of diameters of 20 microns or larger, suggesting a boundary breakaway phenomenon directly related to length and time scales during annealing. In addition, Zr was found to inhibit specific boundary types, promoting mobile 111 tilt boundaries due to strong segregation to twist boundaries. This as well as segregation with respect to boundary morphology and character during recrystallization are presented in this paper.

10:30 AM *BB6.6/EE6.6

Modelling Hardening Behaviour and Dislocation Grain Boundary Interaction in Thin Films. Peter Gumbsch^{1,2}, Daniel Weygand^{1,2} and Zhaohui Jin¹; ¹University of Karlsruhe, Karlsruhe, Germany; ²Fraunhofer IWM, Freiburg, Germany.

The plastic deformation of polycrystalline fcc metal thin films is investigated by simulating the dynamics of discrete dislocations in a representative columnar grain. The simulations are based on the assumption that dislocation sources or multiplication sites are rare and that sources have to operate several times to generate appreciable plastic deformation. The work hardening of such thin films, the influence of boundary conditions and the evolution of dislocation densities are studied. In particular, the interaction of a dislocation with forest dislocations leads to different types of sessile or glissile lock. While the former have already attracted much attention, the behavior of the glissile lock, which can change the character of a dislocation in its glide plane, has not yet been investigated in detail by simulation. The discrete dislocation dynamics simulations results show, that the glissile lock is an effective dislocation source, which allows populating a new slip system that has not been activated before. As this multiplication mechanism is completely athermal, it is a possible stress relaxation mechanism for thin films at low temperatures where thermally activated cross-slip is becoming unlikely. Another important stress relaxation mechanism in thin films and nanostructured materials is dislocation passage through grain boundaries or (re)nucleation of dislocations from grain boundaries. Atomistic simulations are applied to study these processes. In particular, the strength of heavily twinned materials is analyzed in terms of dislocation-twin boundary interaction.

11:00 AM *BB6.7/EE6.7

Quantitative Modeling at the Nanoscale. Lyle Levine, F. Tavazza, L. Ma, R. Wagner and A. M. Chaka; NIST, Gaithersburg, Maryland.

Device design at the nanoscale is inherently multiscale and is further complicated by a lack of modeling tools with quantifiable uncertainties. For example, in determining the operational elastic limits of a proposed nanoscale device, the macroscopic stresses on the device must be propagated down to the individual atoms where the first dislocation or crack initiates. Making such simulations quantitative is extremely challenging by itself, but setting up methodologies that can be used routinely by device designers adds even more constraints. To bridge the relevant length scales, we are using finite element modeling (FEM) to model the macroscopic geometry of nanomechanics experiments, with an initial emphasis on nanoindentation. The FEM mesh refines down to individual atom positions, allowing elastic displacements to be directly propagated to localized atomistic simulations that use classical potentials. In the vicinity of the first inelastic event, bond distortions become large and classical potentials cannot be used for quantitative predictions. For these atoms, we have developed a hybrid quantum-classical algorithm for embedding a quantitative, all-electron, quantum mechanics-based

simulation within the larger classical potential region. Validation testing requires quantitative topographic measurements of the indenter tip and the sample surface before contact and quantitative measurements of sub-nano-Newton forces. Specific validated simulations will be developed as Standard Reference Simulations that device designers can use to calibrate their own modeling predictions.

11:30 AM BB6.8/EE6.8

Direct Calculation of Stiffness and Mobility of Flat Grain Boundaries - Atomistic Simulations. Moneesh Upmanyu¹ and Zachary T. Trautt²; ¹Engineering Division, Materials Science

Program, Colorado School of Mines, Golden, Colorado; ²Engineering Division, Colorado School of Mines, Golden, Colorado.

Dislocation-grain boundary interactions play an important role in recrystallization and deformation of polycrystalline materials. In most multi-scale approaches, the induced motion of grain boundaries is assumed to be proportional to the dislocation density gradients, and the grain boundary stiffness is approximated to be equal to the grain boundary free energy. Here, we present molecular dynamics simulations at extracting grain boundary stiffness during motion induced explicitly by dislocation density gradients. The capillarity forces acting on grain boundaries depend on their stiffness. In this study, we perform molecular dynamics (MD) simulations of capillarity induced fluctuations in grain boundaries with inclination dependent grain boundary energies aimed at extracting their stiffness. The simulations are performed in triangular lattice Lennard-Jones model system at $T=0.3T_m$, for two symmetric tilt grain boundaries with identical inclinations and misorientations. Both boundaries exhibit a well-defined fluctuation spectrum. Analysis of the fluctuation spectrum of the high symmetry boundary reveals that its spectral content is consistent with capillarity wave theory, and the calculated grain boundary stiffness is significantly higher than the free energy of a corresponding asymmetric tilt grain boundary. On the other hand, the fluctuation spectrum of a low symmetry boundary exhibits peaks at certain modes, and the boundary is not rough. Plot of the equilibrium grain boundary profile reveals that it assumes stable inclinations dependent on the peaks in the fluctuation spectrum. Therefore, the inclination dependence low symmetry boundary has cusps in the vicinity of the symmetric tilt inclination, and the grain boundary stiffness cannot be extracted based on the original form of capillarity wave theory.

11:45 AM BB6.9/EE6.9

The Effect of Initial Microstructural Characteristics on Abnormal Grain Growth in Single-Phase Materials: A Mesoscopic Simulation Study. Dorel Moldovan¹ and Rakesh Kumar Behera^{1,2}; ¹Mechanical Engineering, Louisiana State University, Baton Rouge, Louisiana; ²Materials Science and Engineering, University of Florida, Gainesville, Florida.

A mesoscopic simulation methodology has been used to investigate the effect of the initial grain size distribution and grain boundary (GB) energies and mobilities on the development of abnormal grain growth in a single-phase polycrystalline material. The simulation approach is based on the Needleman-Rice (1980) variational formalism for dissipative processes. Our studies show that regardless of the initial grain size distribution, in the absence of anisotropy in GB properties, the system evolves by normal grain growth characterized by a uniform self-similar grain-size distribution, i.e. the presence of a few large grains in the initial microstructure does not promote abnormal grain growth. On the contrary, the presence of a limited set of grains having GBs with higher mobilities and lower energies may lead to abnormal grain growth provided the biased GB mobility/energy values are larger/lower than certain threshold values. Kinetic and topological aspects of normal to abnormal grain growth transition are investigated.

SESSION BB7: Indentation as an Approach to Nanomechanical Studies

Chairs: Erica Lilleodden and Asif Syed
Thursday Afternoon, March 31, 2005
Room 2016 (Moscone West)

1:30 PM *BB7.1

Mechanics of Nano-Imprinting. John Pethica, Graham Cross and Barry O'Connell; Trinity College Dublin, Dublin, Ireland.

The long established methods of forming a pattern in a soft material by imprinting with a harder master stamp are now being extended into the nanoscale. With resolution down to 1-2nm, and cheap mass production potential, it has attractions for device manufacture. Since the primary interest is in the fidelity of the final imprint shape, relatively little is known about the events during the load and unload process leading to that shape. We have recently used a nano-indentation system to observe the displacements of a flat

circular punch with nanoscale features, as it is loaded into, and then separated from a thin polymer film at room temperature. This allows us to infer the stresses, strains, and geometries present at the various stages of imprint, and therefore gives insight into possible improvements to the process. This talk will give an overview of the less expected observations and their significance for fidelity of imprint. A residual layer is left underneath the flat punch which in turn affects the mask ratio. Surprisingly, its thickness is independent of magnitude of the applied load (once beyond the plastic flow pressure), or the initial thickness of the polymer film. We show this is partly due to elastic relaxation of the stamping punch, particularly the hinterland behind the immediate nanoscale features. Aspect ratios of features in imprint need to be designed to allow for the effect. Mesas may be formed by punches with a cavity milled into the flat face. Residual stresses and strain rates significantly affect the mesa replication. Two types of flow occur, depending on whether creep relaxation is possible or not. In some polymer systems, although very high strains occur, there is not always corresponding strain hardening, which affects the understanding of formation of larger area mesas. Finally the potential for multi-layer imprinting will be discussed.

1:30 PM *BB7.2

Characterization of the dynamic properties of polymers using instrumented indentation and a flat punch. Warren C. Oliver, Erik G. Herbert, and Pierre J. Morel; MTS Nano Instruments Innovation Center, Oak Ridge, Tennessee.

Using instrumented indentation techniques, the complex modulus of an extremely compliant synthetic tissue ($E' \sim 40$ kPa) has been investigated over the frequency range of 0.05 to 243 Hz. Unlike most indentation experiments, accurately isolating the materials response in this case requires a much more rigorous characterization of the measurement system. In this work, we review the dynamic model used to characterize the measurement system and clearly identify its inherent assumptions and limitations with respect to testing extremely low modulus materials. The implications of this review will be discussed for polymer thin films.

2:30 PM BB7.3

Quantitative Electromechanical and Elastic Measurements by SPM. Sergei V. Kalinin¹, Stephen Jesse¹, Junsoo Shin^{2,1}, Arthur P. Baddor¹, Mark Kachanov³ and Edgar Karapetian⁴; ¹Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; ²Department of Physics and Astronomy, The University of Tennessee, Knoxville, Tennessee; ³Department of Mechanical Engineering, Tufts University, Medford, Massachusetts; ⁴Department of Mathematics and Computer Science, Suffolk University, Boston, Massachusetts.

In the last decade, a number of nanoindentation and Scanning Probe Microscopy (SPM) based techniques for local characterization of elastic and electromechanical properties on the nanoscale have been suggested. The image formation mechanism in these techniques, including Piezoresponse Force Microscopy (PFM), Atomic Force Acoustic Microscopy (AFAM) and Ultrasonic Force Microscopy (UFM), as well as in nanoindentation, is ultimately based on the bias-dependent contact mechanics of the tip-surface junction. We demonstrate local electromechanical and elastic imaging and spectroscopy for a broad range of materials systems from ferroelectric to biological systems. To quantify these data, the nanoelectromechanics of the tip-surface junction, including the structure of coupled electroelastic fields and stiffness relations, is analyzed for flat, spherical, and conical indenter geometries. Exact solutions in elementary functions for electroelastic fields inside the material are obtained using the recently established correspondence principle between the elastic and the piezoelectric problems. The stiffness relations fully describe the indentation process and relate indentation depth, force and bias to the relevant material properties, and indenter parameters. This extends the results of Hertzian mechanics to piezoelectric materials. The structure of the electroelastic field yields a quantitative measure of the signal generation volume in electromechanical SPMs and also provides a quantitative basis for the analysis of tip-induced polarization switching and local hysteresis loop measurements. An approach for combined imaging of elastic and electromechanical properties of materials is presented. It is shown that for a transversally isotropic material this provides the most comprehensive materials properties achievable. It is also shown that the indenter shape, generally unknown in SPM experiments, can be reliably calibrated, thus providing a pathway for quantitative electromechanical and elastic studies of piezo- and ferroelectrics at the nanoscale. Research was sponsored by the U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the National Science Foundation grant DMR-0072998. Research performed as a Eugene P. Wigner Fellow (SVK).

2:45 PM BB7.4

Determination of the Mechanical Properties of Spin-on Zeolite Low-k Thin Films. Mark Johnson¹, Zijian Li², Yushan Yan² and Junlan Wang¹; ¹Mechanical Engineering, University of California, Riverside, California; ²Chemical and Environmental Engineering, University of California, Riverside, California.

With the semi-conductor technologies continuously pushing the miniaturization limits to sub-0.09 microns, there is strong interest in low dielectric constant (low-k) materials that can replace traditional SiO₂ based insulators as intermetal dielectrics in order to speed up the signal transmission and decrease interlayer crosstalk and interline leakage. In order to survive the necessary technological integration processes and provide reliable material and structure template for the desired IC functions, the new low-k materials have to be mechanically strong and stable. Thus the material selection and mechanical characterization are vital in the successful development of next generation low-k dielectrics. In this talk, a new class of low-k dielectric materials-nanoporous zeolite thin films prepared from IC compatible spin coating technique is characterized using depth sensing nanoindentation technique. The different trends observed in the hardness and modulus as a function of indentation depth using two different indenters- Berkovich and cube corner- will be discussed. Cube corner indenter appears to be more appropriate for the characterization of these submicron low-k films. The property-synthesis relation will also be addressed.

3:30 PM *BB7.5

Nanoindentation of Semiconductors. James S. Williams and Jodie E. Bradby; Electronic Materials Engineering, Australian National University, Canberra, Australian Capital Territory, Australia.

This presentation will review our recent work on nanoindentation of semiconductors using a range of nanoindentation apparatus and analysis methods, including micro-Raman, transmission electron microscopy, atomic force microscopy and in-situ electrical measurements. Firstly, nanoindentation of silicon (Si) has generated much interest in recent years as a result of pressure-induced phase transformations that can occur under the indenter. For example, diamond-cubic (Si) can transform into a metallic phase at a pressure of 11.5 GPa and, on pressure release, to a number of other possible phases. We have found that it is possible to control the end phases by controlling the indentation conditions, to cycle between crystalline (Si III and Si XII) and amorphous Si phases by controlling the unloading rate for room temperature indentation. Noting that the different end phases exhibit very different properties, particularly electrical conductivity, nanoindentation opens up prospects for selective 'processing' of silicon down to the less than 100nm scale. In the case of germanium (Ge), there has been some conjecture over whether it is possible to induce similar phase transformations at room temperature by indentation. We have used a range of indentation conditions, as well as thin film Ge and Ge-Si structures on Si substrates, to explore this issue. We have found Ge either deforms via extended defect propagation within the Ge-I phase or phase transforms, depending on the loading conditions and/or the thin film thickness/composition. For compound semiconductors (GaAs, InP, GaN and ZnO), these materials deform via defect propagation at room temperature rather than phase transformation. The nature of the deformation processes (and hardness) differs between these materials. Details are presented and discussed, particularly the onset and origin of so-called "pop-in" processes under loading. Implications for device fabrication and MEMS/NEMS structures in these materials are also discussed.

4:00 PM BB7.6

Deformation Behavior of Nanocrystalline BCC Ta. Yinmin Wang¹, Andrea M. Hodge¹, Juergen Biener¹, Alex V. Hamza¹ and T. G. Nieh²; ¹Chemistry and Materials Science Directorate, Lawrence Livermore National Laboratory, Livermore, California; ²Materials Science & Engineering, The University of Tennessee, Knoxville, Tennessee.

Our knowledge of the deformation mechanism of nanocrystalline materials is currently limited to FCC metals and their alloys. Although nanocrystalline BCC materials promise superior strength, their deformation mechanisms are poorly understood. In this talk, we will present experimental results on the deformation behavior of nanocrystalline BCC Ta with grain sizes of 10-30 nm, using nanoindentation and high-resolution transmission electron microscopy. Our results reveal that the plastic deformation of nanocrystalline BCC Ta proceeds predominantly via a partial dislocation mechanism, in deviation from previous theoretical and experimental work on other BCC nanocrystalline materials. We will discuss the implications of this surprising behavior in nanocrystalline BCC materials. 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.

4:15 PM BB7.7**Nanoporous Au - A Brittle yet Ductile Material.**

Juergen Biener, Andrea M. Hodge, Alex V. Hamza and Luke M. Hsiung; Nanoscale Synthesis and Characterization Laboratory, Lawrence Livermore National Laboratory, Livermore, California.

Nanoporous metals have recently attracted considerable interest fueled by potential sensor and actuator applications. From a material science point of view, one of the key issues in this context is the synthesis of a nanoporous metal with both high tensile and compressive strength. Nanoporous Au has been suggested as a candidate material for this application due to its monolithic character. The material can be synthesized by electrochemically-driven dealloying of Ag-Au alloys, and exhibits an open sponge-like structure of interconnecting ligaments with a typical pore size distribution on the nanometer length scale. Besides the observation of a ductile-brittle transition in nanoporous Au very little is known about its mechanical behavior. However, the question still remains whether the brittle nature of nanoporous Au is caused by an intrinsic brittleness of Au ligaments on the nanometer length scale, or is a consequence of the macroscopic structure. Here we present our results regarding the mechanical properties and the failure mechanism of nanoporous Au. Depth-sensing nanoindentation reveals that nanoporous Au with a relative density of ~40% exhibits a yield strength of ~145 MPa and a Young's modulus of ~11 GPa. Thus the yield strength of this material is almost one order of magnitude higher than predicted by foam scaling laws. The failure mechanism of nanoporous Au under tensile stress was evaluated by microscopic examination of fracture surfaces using scanning electron microscopy (SEM). On a macroscopic level, nanoporous Au is very brittle. However, SEM micrographs reveal the ductile nature of the material on a nanometer length scale. Localized yielding events lead to failure of individual ligaments as demonstrated by the observation of characteristic necking and fine point features. Thus microscopically the material is very ductile and high strain values can be accommodated by individual ligaments. Two-dimensional defects caused by inhomogeneities of the original Au-Ag alloy composition facilitate crack formation. 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.

4:30 PM BB7.8

Direct Visualization of Pile-up Formation During Indentation in Nanocrystalline and Amorphous Metals. Benedikt Moser¹, Jean-Marc Breguet² and Johann Michler¹; ¹Materials Science and Technology, EMPA, Thun, Switzerland; ²Laboratory for Robotic Systems, EPFL, Lausanne, Switzerland.

Extensive research has been devoted to the formation of pile-up during instrumented indentation and its influence on the measurement of mechanical properties such as hardness and Young's modulus. In most of these studies the topography of the residual indent is measured by methods such as scanning electron microscopy (SEM), atomic force microscopy (AFM), or optical methods. These methods can yield some information on the true contact area between the indenter and the tip and the size (volume) of the pile-up. Additionally, information on the amount of strain hardening can also be extracted from these measurements. However, measured after the fact, they do not enable conclusions on the formation of the pile-up during the process of indentation. This is particularly interesting when material properties vary with indentation depth (graded materials, film/substrate systems) or when the pile-up shows features that specifically pertain to a material class and its deformation mechanism (e.g., shear bands in bulk metallic glasses). We present here first results from micro-indentation experiment using a recently designed instrumented in-situ indentation system that allows the direct high resolution observation of the immediate vicinity of the indenter tip during the indentation process inside the scanning electron microscope. Experiments have been done in nanocrystalline pure metals and alloys as well as in bulk amorphous metals. We will first present the instrument with its capabilities. We will then present video sequences from micro-indentation experiments on nanocrystalline metals and compare with experiments on their microcrystalline counterparts. Marked differences in the size of the pile-up and its surface structure developing during the indentation process have been found between nanocrystalline and microcrystalline materials. These differences will be linked to the deformation mechanism and grain size in the respective material. The use of automated image analysis for measuring the true contact area in-situ is explored. In a second part we will show results from experiments in bulk metallic glasses where the shear band formation around the indent can be observed and directly linked to displacement bursts in the load-displacement curve. The growth characteristics of these shear bands will be discussed in more detail on the basis of results from automated video analysis.

4:45 PM BB7.9

Effect of Pileup in Nanoindentation of Micro and Nano Crystalline Ni using FEM. Raja Mahesh Pothapragada¹, Reza

Mirshams¹ and Suman Vadlakonda¹; ¹Mechanical Engineering, University of North Texas, Denton, Texas; ²Material Science, University of North Texas, Denton, Texas.

A much better understanding of the contact mechanics is obtained mostly through finite element modeling. Indentation into materials like microcrystalline Ni and nanocrystalline Ni results in pile-up of material around the indentation. Indentation experiments were conducted and pileup is calculated by directly measuring the dimensions of the residual impression by AFM techniques. The experiment were modeled using the software package Nano SP1 which is based on FE code COSMOS/M. The experimentally determined tip shape function was included to reproduce the same contact area for a given contact depth as in the experiment. The fundamental material properties affecting pile-up are the ratio of the effective modulus to yield stress E_{eff}/σ and the work hardening behavior. Two separate cases of work hardening were considered one with no work hardening that is an elastic-perfectly plastic material and other with a linear work hardening. Specifically it is observed pile up is large only when h_f/h_{max} is close to one and degree of work hardening is small. It should also be noted that when $h_f/h_{max} < 0.7$ very little pile-up is observed no matter what the work-hardening behavior of the material. The graphs plotted between Pile-up vs. E/Y and Pile-up vs. Yield strength describes material behavior. These graphs give an easier way of identifying properties. When pile-up occurs the contact area is greater than that predicted by the method and both the hardness, modulus are overestimated. In this paper we will also discuss the amount by which these properties are overestimated.

SESSION BB8: Fatigue, Fracture and Strain Rate Effects

Chairs: Eric Guyer and Ruth Schwaiger
Friday Morning, April 1, 2005
Room 2016 (Moscow West)

8:30 AM *BB8.1

Nanomechanics at the Organic-Inorganic Interface.

Adrian B. Mann^{1,2} and Michelle E. Dickinson^{1,2}; ¹Ceramics & Materials Eng., Rutgers University, Piscataway, New Jersey; ²Biomedical Engineering, Rutgers University, Piscataway, New Jersey.

The mechanical behavior of the interface between organic molecules and inorganic materials is vital in a huge number of applications. Examples range from the lubrication of engines and the biodegradation of art objects to the biofouling of sensors and the osseointegration of bone implants. Unfortunately quantifying the mechanical behavior of organic-inorganic interfaces is extremely difficult. It is complicated by the need to perform nanoscale measurements on systems where the organic component is usually viscoelastic, sometimes a liquid, frequently adhesive and often environmentally sensitive. In contrast, the inorganic component typically exhibits none of these attributes. Combining recent developments in point-probe instrumentation and carefully thought out experimental methodologies we have been able to obtain the first truly quantitative measurements of nanomechanical properties at the organic-inorganic interface. Two quite different examples of organic-inorganic interfaces will be described. The first is an organic liquid (long chain alcohol) on an inorganic surface (GaAs) and the second is a viscous, proteinaceous biofilm (salivary pellicle) on a naturally occurring bioceramic (dental enamel). Our results show that a thin layer of organic liquid can substantially increase the elastic modulus and hardness of a surface. It is important to realize that this is not a true change in mechanical properties. Rather it is due to the ability of an organic liquid in a highly confined geometry to sustain elastic strains. This modifies the nanoscale geometry of contacting surfaces and changes the effective elastic properties of the contacting surfaces. In the second example the organic film (salivary pellicle) is relatively thick (around 1 micron), but its viscoelastic properties and structure are very sensitive to its chemical environment. We have found that poly-phenolic compounds, such as those found in tea and wine, can dramatically increase both the storage and loss modulus of the pellicle. The morphology and color of the pellicle layer are also affected by the poly-phenolic compounds. The color changes are actually one of the main causes of extrinsic staining on dental enamel. The pellicle's increased mechanical properties due to staining explain why teeth stains are so hard to remove by abrasive cleaning with a toothpaste and brush.

9:00 AM *BB8.2

Bauschinger Effect in Thin Metal Films. Joost J. Vlassak and Yong Xiang; DEAS, 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 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 an 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 films. Passivated Cu films show a 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 Cu 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.

9:30 AM **BB8.3**

Deformation Mechanisms of Nanostructured Materials. Yuntian T. Zhu, Materials Science & Technology Division, Los Alamos National Lab, Los Alamos, New Mexico.

Nanostructured materials deform via mechanisms not accessible to their coarse-grained counterparts. Partial dislocation emission from grain boundaries, stacking faults and deformation twinning may occur in metals such as Al, which does not deform by twinning in its coarse-grained state. In this presentation I shall discuss several deformation mechanisms in nanomaterials as well as their formation conditions. Specifically, I shall first give a brief overview on the deformation mechanisms, observed by both molecular dynamic simulations and experiments. I shall then present a dislocation-based model to describe the nucleation and growth of deformation twins in fcc metals. I shall also discuss other nanocrystalline-related deformation features such as wide stacking faults and five-fold twins.

9:45 AM **BB8.4**

Contact Fatigue of Nanocrystalline Metals Using Instrumented Indentation. Ruth Schwaiger¹, Joerg Knyrim¹ and Subra Suresh²; ¹Institute for Materials Research II, Forschungszentrum Karlsruhe, Karlsruhe, Germany; ²Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Nanostructured materials show high potential for high performance applications due to their enhanced strength, hardness, wear, and corrosion resistance. Investigating the contact damage resistance of nanocrystalline and ultra-fine crystalline metals is critical to an overall assessment of their usefulness as structural components and coatings. Recent work revealed that differences in the quasi-static deformation characteristics of nanocrystalline and ultra-fine crystalline Ni exist, but results on the contact fatigue behavior in the literature are rare. We studied the influence of grain size on the time- and cycle dependence of contact deformation using a nanoindenter. Contact fatigue behavior was investigated through a systematic series of experiments on model materials, i.e. electrodeposited fully-dense Ni with different grain sizes down to the nanocrystalline grain size regime. Cyclic contact loading was found to significantly enhance grain growth in fine-grained metals. An increase in grain size by as much as a factor of 20 has been observed. This microstructural instability is most apparent in materials with a very fine initial grain size and represents a threat to the use of fine-grained materials in many applications. Damage evolution and mechanical response of nanocrystalline metals were studied as a function of cycle number under normal- and sliding- contact loading.

10:30 AM **BB8.5**

Contact and Contact Fatigue Behavior of Nanocrystalline Materials. Simon C. Bellemare¹, Timothy Hanlon² and Subra Suresh¹; ¹Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; ²Global Research and Development, General Electric Company, Niskayuna, New York.

The present work attempts to probe the mechanical properties of nanocrystalline metals under conditions of highly confined, and large plastic strains. The effects of microstructural and experimental length scales on the above deformation behavior are also investigated. Contact and contact fatigue behavior was studied by repeatedly scratching, under well controlled conditions, the surface of nickel and copper specimens with dissimilar grain sizes and twin densities. Friction forces were monitored in-situ during each experiment. Finite element modeling was used to predict the stress and strain fields associated with the various loading conditions employed. These

predictions indicate that the local plastic strain increases substantially with the ratio of the penetration depth to the tip radius. Scratching is shown to induce much larger plastic strains than those obtained through indentation and uniaxial tensile testing, thereby complementing these testing techniques in addition to providing a basis for understanding and predicting the tribological response. Under a low penetration depth to tip radius ratio, and at Newton level normal loads, the friction coefficient of pure nickel was found to steadily increase with the number of scratch passes, prior to reaching a steady state. This steady state friction coefficient, as well as the extent of accumulated damage, was found to increase with increasing grain size. Considering that the macroscopic yield strength is also a function of grain size, however, it was necessary to decouple the effects of these two properties on the observed tribological response. We report here results of critical experiments in which the yield strength of ultrafine crystalline copper was varied, without altering the grain size. In addition, results are presented for experiments with grain size and penetration depth both maintained in the nanometer range.

10:45 AM **BB8.6**

Multiscale Modeling of Nanobeams Structures for MEMS/NEMS Applications Subjected to Cyclic Loading. Behrouz Shari¹, Ronald E. Miller² and Dennis D. Klug¹; ¹Stacie Institute for Molecular Sciences, National Research Council of Canada, Ottawa, Ontario, Canada; ²Department of Mechanical and Aerospace Engineering, Carleton University, Ottawa, Ontario, Canada.

Knowledge of mechanical properties is essential for the successful modeling and design of Micro/nanoelectromechanical Systems (MEMS/NEMS) whose dimensions are only about several nanometers. Unfortunately, most of our knowledge about materials behavior under elastic and plastic loading conditions fails to describe cases in which a characteristic dimension is of a few nanometers. Each MEMS/NEMS device that is considered seems to exhibit differences in mechanical behavior and introduces new fundamental questions. This is a consequence of the strong effect that size introduces on fracture, plasticity, and fatigue. Cyclic loading, for instance, may ultimately limit product lifetime in certain applications. Cyclic loading is of particular interest in devices such as RF switches or fixed-fixed nanobeam structures used in MEMS/NEMS which operate under repeated loading at different frequencies and operational temperatures. We have studied the cyclic behavior of single-crystal copper and aluminum nanobeams by using multiscale modeling. A new modified coupled atomistic/continuum dislocation (CADD) method at finite temperature has been chosen to carry out this study. The Dynamic CADD model simultaneously captures the atomistic mechanism and the long range mobility of dislocations and their interactions without the computational cost of full atomistic simulations. The method also permits the simulation of system sizes that are approaching experimentally accessible systems. Repeated loads are applied by a displacement-controlled nanoindenter in the centre of the span as a harmonic function. Nanobeams are fixed in both ends, and then the maximum moments (or critical stress) occur under the indenter (or load position) and close to the fixed ends. These critical sections of nanobeams are modeled as atomistic regions and the remaining are modeled with finite elements. The load-displacement (load/unload) curves for different temperatures are compared for different load frequencies. The initial nucleation of dislocations in critical zones which have been modeled as atomistic regions, are investigated. Correlation between the observed features in the load-displacement curves with the motion, cross-slip and pile-up of dislocations are reported. Results of critical stresses from both atomistic and finite element point of views are presented and compared. Finally, under repeated load/unload cycles, a comprehensive study has been performed to demonstrate the generation and motion of dislocations as a function of temperature and load frequency. The accumulation of plastic deformation or damage evolution under cyclic loading is presented.

11:00 AM **BB8.7**

A Fracture-Mechanics and Kinetics Analysis of the Persistent-Slip Band Oxidation Mechanism for Fatigue-Crack Initiation in Ductile MEMS. Brad L. Boyce and Thomas E. Buchheit; Materials and Process Sciences Center, Sandia National Laboratories, Albuquerque, New Mexico.

The fatigue behavior of an electrodeposited Ni MEMS structure formed by the LIGA process has been evaluated using a micro-cantilever beam experiment under fully-reversed loading ($R = -1$). Prior to crack-initiation, thick (~400 nm) oxides have been shown to form on the surface of the persistent slip bands. These thick oxides are thought to accumulate due to enhanced diffusion created by PSB-induced cyclic film rupture of the passive oxide. Once the oxide is sufficiently thick, the imbedded flaws are large enough to exceed the material's fatigue threshold, thereby propagating into the matrix. The driving force for propagation out of the surface oxide is assessed using

a fracture mechanics framework. From this analysis, the critical oxide thickness for propagation is estimated to be in the vicinity of $1 \mu\text{m}$, consistent with experimental observations. The PSB-induced oxide mechanism also presents a basis for assessing the kinetics of oxide thickening, and therefore the rate of crack-initiation. The rupture events can occur no more often than once per cycle (once per 50 msec) and in lab air at room temperature nickel is expected to repassivate well before the subsequent cycle. Therefore, under these conditions oxide growth and hence crack initiation is rate-limited by the frequency of the mechanical rupture events and not by the classical thermally-driven oxidation kinetics that scale with the square root of time. This mechanistic analysis may provide insight into the incubation time for fatigue cracking, an otherwise nebulous quantity that dominates the fatigue life under high-cycle fatigue conditions. Discussion will center around a comparison of micro- and nano-grained behavior ($\sim 5 \mu\text{m}$ and 50 nm) and the existence of a length scale threshold below which this initiation mechanism is apparently eliminated and resulting fatigue lives are notably improved. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

11:15 AM BB8.8

Accelerated Crack Growth in Nanoporous Organosilicate Thin-Film Glasses. Eric P. Guyer and Reinhold H. Dauskardt; Materials Science & Engineering, Stanford University, Stanford, California.

Integrating nanoporous low-k thin-films into interconnect structures has proven to be a significant technological challenge due to their high propensity for mechanical failure and susceptibility to stress corrosion cracking. Nearly all levels of processing and subsequent device packaging involve subjecting these fragile device structures to mechanical loads in the presence of chemically active aqueous environments. The combination of harsh processing conditions and the fragile nature of low-k films has severely limited device yields and long-term reliability. Here we demonstrate the significant effect salient aqueous solution chemistries have on accelerating the rate of crack growth in nanoporous methylsilsesquioxane (MSSQ) thin-films. We further demonstrate how systematically increasing the porosity of MSSQ films, such that the dielectric constant was reduced from 2.9 to 1.9, reduces the cohesive fracture energy of the films by $\sim 1 \text{ J/m}^2$. This seemingly small decrease corresponded to an increase in crack growth rates of over 4 orders of magnitude. The first principles model we have developed to describe this behavior is shown to be consistent with the experimental data obtained for four different MSSQ films. It is also demonstrated that this model can be extended to successfully describe crack growth in carbon doped oxide films and even bulk silica glass. Implications for the integration of nanoporous thin-films in advanced interconnect devices are considered.

11:30 AM BB8.9

Unusual Fracture Behavior of Nanoporous Polymeric Thin-Films. Andrew Kearney and Reinhold H. Dauskardt; Materials Science and Engineering, Stanford University, Stanford, California.

Nanoporous polymeric thin-films are being considered for a range of applications in thin-film devices, device packages, sensor and actuator technologies. The integration of these materials has in many cases presented significant processing and reliability challenges as polymer thin films have inherently lower elastic modulus, strength and hardness compared to other material classes. However, polymer layers often exhibit both adhesive and cohesive fracture properties that are higher than those reported for glass, ceramic, and sometimes even metal layers. Interfacial chemistry, polymer deformation properties and polymer layer thickness are the prime determinants of fracture properties. Of particular concern for emerging technologies is the need for nanoporous versions of thin films. In nearly all cases, the introduction of nanoscale porosity has a deleterious effect on mechanical properties, producing materials and layers that are structurally weaker than the materials they replace. In the present study involving poly(arylene) ether (PAE) films, we present surprising evidence that the fracture resistance of porous forms of the PAE films exhibit increasing fracture resistance with porosity. Such behavior is in stark contrast to a wide range of reported behavior, which indicates that the fracture toughness of porous solids typically decreases markedly with porosity. A fracture mechanics based model is presented to rationalize the increase in fracture toughness of the voided polymer film and explain the behavior in terms of the nanoporosity volume fraction and pore size. It is shown that a certain dependence of pore size and volume fraction is required to increase rather than decrease the fracture resistance. Implications for the optimum void size and volume fraction are considered.

11:45 AM BB8.10

Mechanics of Nanostructured Polymers and Polymer Nanocomposites at High Strain Rates. Adam D. Mulliken and

Mary C. Boyce; Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Polymeric materials are known to exhibit strong time-dependent mechanical behavior, as evidenced by rate-dependent elastic moduli, yield strength, and post-yield behavior. The nature of the rate sensitivity is found to change between different temperature regimes as various primary and secondary molecular mobility mechanisms are accessed. The ability to tailor these molecular-level mechanisms through the incorporation of nanoscale particles offers new opportunities to design polymer-based material systems with different behaviors (elastic, yield, post-yield) in different frequency regimes. In this study, a combined experimental and analytical protocol has been carried out in order to examine the effects of specific nanoscale structural changes on the macroscopic rate-dependent mechanical behavior of amorphous polymers. A number of polymer nanocomposites and their homopolymer counterparts have been evaluated at strain rates ranging from 10^{-4} to 10^4 s^{-1} . This range in strain rates was achieved in uniaxial tension and compression tests using a dynamic mechanical analyzer (DMA), a servo-hydraulic testing machine, and a split-Hopkinson pressure bar. DMA tension tests were used to characterize the viscoelastic behavior of these materials, with focus on the rate-dependent shift of material transition temperatures. Uniaxial compression tests on the servo-hydraulic machine (10^{-4} to 1 s^{-1}) and the split-Hopkinson pressure bar (10^3 to 10^4 s^{-1}) were used to characterize the rate-dependent yield and post-yield behavior. The data reveals fundamental changes in the nature of the rate dependence of the mechanical properties between different frequency regimes. Based on the experimental data, the nanoscale alterations were evaluated in terms of their effect upon the rate-dependent mechanical properties through interactions with primary (α) and secondary (β) intermolecular motions. The development of a constitutive model that captures and predicts the observed behavior over the wide range in strain rate is presented. Particular attention is focused on the behavior of these polymer systems under the strain and strain rate conditions encountered in blast, projectile impact, and other impact loading scenarios.

12:00 PM BB8.11

Deformation Behavior and Strain Rate Sensitivity of Nanostructured Materials under Moderate Temperatures. Cecilie Duhamel, Sandrine Guerin-Mailly and Yannick Champion; CECM, CNRS, Vitry/Seine, France.

Nanocrystalline and ultra-fine grained materials are of growing interest because of their mechanical behavior and unusual shaping abilities. Except for very small grain sizes where radically new stress relaxation effects occur, the yield strength of nanostructured metals, in accordance with the Hall-Petch relation, is much higher than the yield strength of their coarse-grained counterparts. However, major drawback is the apparent low ductility (at usual quasistatic strain rates) due to the absence of strain hardening [1]. A better understanding of the mechanisms controlling plastic deformation in nanocrystalline materials is thus necessary in order to design new nano-architectures with an enhanced ductility. The apparition of inhomogeneous deformation can be predicted by instability criterions. As FCC metals become strain-rate sensitive with decreasing grain size, Hart's criterion ($1/\sigma)(d\sigma/d\epsilon) - 1 + m \leq 0$ can be applied to determine the apparition of plastic instability [2]. Accordingly, the strain-rate sensitivity m helps avoid localized deformation and maintain uniform tensile elongation. Increasing m should enable to delay the apparition of strain instability and, consequently, the material failure. Previous work conducted on nanostructured copper prepared by powders metallurgy (grain sizes 90 nm) has revealed the nearly perfect elasto-plastic behavior in tension of this material [3]. Failure occurs after 12% deformation and no apparent necking is detected. In addition, compression tests with strain-rate jumps have revealed a maximal strain-rate sensitivity m of 0.045 for $\epsilon = 10^{-5} \text{ s}^{-1}$ at room temperature. Strain-rate jumps tests in compression were carried out on nanostructured copper under moderate temperatures ($353\text{K} - 393\text{K}$). Strain-rate sensitivity was determined as a function of temperature (T) and strain rate ($\dot{\epsilon}$). Increasing temperature from 298K to 393K enables to obtain m value as high as 0.17. These results of enhanced m are encouraging in terms of gain in ductility. They are also characteristic of the existence of a thermally activated process different from the normal rate-controlling mechanism observed for microcrystalline FCC metals. [1] H.S. Kim and Y. Estrin, Appl. Phys. Lett., 79, 4115, (2001) [2] Y.M. wang and E. Ma, Acta Mat., 52(6), 1699, (2004) [3] Y. Champion and al., Science, 300 (2003)