SYMPOSIUM W

The Limits of Strength in Theory and Practice

November 28 – 30, 2000

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* Invited paper

SESSION W1:
Chair: J. W. Morris, Jr.
Tuesday Morning, November 28, 2000
Fairfax B (Sheraton)

8:30 AM *W1.1
LATTICE STABILITY AND LIMITS OF STRENGTH IN THEORY AND ATOMISTIC SIMULATIONS. Ju Li, Sidney Yip, Massachusetts Institute of Technology, Dept. of Nuclear Engineering, Cambridge, MA.

We examine the theoretical basis and simulation techniques for understanding the response of crystalline lattice to critical stress loadings at finite temperatures. Various derivations now exist giving the stability criteria for homogeneous material in terms of load-dependent elastic stiffness coefficients. These criteria provide upper bounds on the material's strength, approachable sometimes in indentation hardness experiments. The extension of homogeneous deformation to finite wavelength is the analysis of phonon soft mode for a stressed lattice, which is theoretically rigorous at zero temperature. We will present lattice dynamical results obtained using model potentials for a variety of solids (Ar, Cu, Mo, Si, SiC, SiO_2, and ZrC) under hydrostatic, shear, and uniaxial loadings. We find that simple lattices often fail by elastic instabilities (k=0 modes), whereas the binary lattices mostly fail by soft modes at Brillouin zone boundaries. The latter is often interpreted as the breaking of heteropolar bonds driven by the local competition for chemical affinity. To study the lower limits of strength, one must deal with defect mobility. However an acute problem for atomistic simulations is the lack of a sound measure of how the defect is driven, since at the available small system size, linear elasticity, not to mention the concept of stress field itself, may not be applicable. We propose a local measure of the thermodynamic driving force for the evolution of an isolated defect which focuses on energy considerations in much the same spirit as the J-integral in fracture mechanics. This concept is applied to the study of strength and deformation of Cu in various idealized microstructures by molecular dynamics simulation.

9:00 AM W1.2
ATOMISTIC SIMULATIONS OF CRACK PROPAGATION IN SiO^2 UNDER EXTREME CONDITIONS OF PRESSURE AND TEMPERATURE. M.J. Caturla, A. Kubota, T. Lenosky, V. Bulatov, T. Diaz de la Rubia, M. Feit, Lawrence Livermore National Laboratory, Livermore, CA.

The mechanical strength of silica glass is of great importance for industrial and scientific applications, including semiconductor processing and laser optics damage issues. In particular, silica optics used to focus UV laser light can undergo damage that grows with subsequent laser pulses. Mitigation of the damage growth is of extreme importance for the National Ignition Facility under development at Lawrence Livermore National Laboratory. For this application it is important to understand the time-resolved atomic-scale mechanism of crack growth in silica under extreme conditions of pressure and temperature. We have modified our parallel molecular dynamics code MDCASK by including fast multipole expansion algorithms to describe long-range coulombic interactions. We compare several empirical interatomic potentials with ab initio (GGA) calculations under different strain levels in order to select that empirical potential that is most suitable for this study. We show interesting trends in SiO^2 fracture properties of pre-existing cracks as a function of varying conditions, including large temperature and pressure gradients induced by high localized absorption of UV laser light.
This work was carried out under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

9:15 AM W1.3
DIRECTIONAL ANISOTROPY IN THE CLEAVAGE FRACTURE OF SILICON. Rubén Pérez and Peter Gumbsch, Universidad Autonoma de Madrid, Departamento de Fisica Teorica de la Materia Condensada, Madrid, SPAIN; Max Planck Institut für Metallforschung, Stuttgart, GERMANY.

Total-energy pseudopotential calculations are used to study the cleavage fracture anisotropy in silicon. This full quantum mechanical analysis provides an accurate description of the nonlinear forces acting on the crack tip atoms. A fixed boundary approach is used to enforce the elastic boundary conditions representing the crack field. The influence of the fixed boundary is determined through the scaling of the results with system size and with the changes with the relative position of the crack elastic field singularity and the atomic position of the crack tip. The simulations show that the bond breaking at the crack tip proceeds differently for the different crack orientations. Crack tip bonds smoothly lengthen with increasing load and cracks propagate easily on {111} and {110} planes provided crack propagation proceeds in the $\langle\bar{1}10\rangle$ direction. This continuous breaking process mimics what one would expect from continuum theory and therefore results only in a small lattice trapping. In contrast, if the crack is driven in a $\langle 001\rangle$ direction on a {110} plane the bond breaking process is discontinuous: the crack tip bond length remains almost unchanged until the external load reaches a critical value at which the bond abruptly breaks. This bond breaking is associated with pronounced relaxations of a small number of atoms surrounding the crack tip. The origin of the discontinuous behaviour can be partly traced back to some load sharing between the crack tip bond and the neighbouring bonds, which effectively shields the crack tip bond from the applied load and results in a large lattice trapping. The different lattice trapping for different crack propagation directions can explain the experimentally observed cleavage anisotropy in silicon single crystals.

9:30 AM *W1.4
INFLUENCE OF CRYSTALLINE SYMMETRY ON ELASTIC STABILITY AND BIFURCATION PHENOMENA AT FINITE STRAIN. Frederick Milstein, Univ of California, Santa Barbara, CA.

The theoretical response of metals to various modes of loading at finite strain is examined. Particular attention is given to the topic of elastic instabilities at points of bifurcation, as well as to post bifurcation phenomena leading to phase change or material failure, as predicted by elastic stability theory. The mechanical response, including theoretical strengths, is strongly influenced by crystalline symmetries and associated bifurcation behavior. Prominently featured are computational results based on an embedded-atom method that is formulated to reproduce, identically, empirical values of the three second order elastic moduli (c11, c12, and c44) and the six third order elastic moduli (c111, c112, c123, c144, c166, and c456) of 12 cubic metals (Al, Cu, Mo, Na, Li, K, Rb, Nb, Fe, Ni, Au, and Ag). This formulation ensures that both the linear (harmonic) and non-linear (anharmonic) response of the metals is represented in the computations. Examples of both lattice static and molecular-dynamic results are presented.

10:30 AM *W1.5
COMPUTATION OF THE IDEAL STRENGTH. David Roundy, Marvin L. Cohen, Dept. of Physics, Univ. of California Berkeley, Lawrence Berkeley National Laboratory, CA.

The ideal strength is the upper limit of the strength of a material. Advances in computing have made possible increasingly accurate calculations of the ideal strength, but there are challenges in imposing a desired stress tensor. We will discuss the methods used in calculations of the ideal and discuss the results of recent *ab initio* calculations of the ideal tensile strength as well as the ideal shear strength of various materials.

11:00 AM W1.6
CORRELATION OF TOPOLOGY OF CHARGE DENSITY WITH ENERGETICS IN SHEAR AND CLEAVAGE DEFORMATIONS IN FCC METALS. Nicholas Kioussis, M. Herbranson and E. Collins, California State Univ. Northridge, Dept of Physics, Northridge, CA; Mark Eberhart, Chemistry and Geochemistry, Colorado School of Mines, Golden, CO.

We have employed *ab initio* total-energy electronic structure calculations, based on the full potential linear muffin tin orbital method, to evaluate the energetics and topology of the charge density in shear and cleavage deformations in Al, Ag, and Ir, exhibiting different mechanical properties. We will present a correlation between planar fault and cleavage energetics with the shape of the charge density in the neighborhood of its critical points, i.e. the anisotropy of the principal curvatures of the charge density parallel and perpendicular to the bond path. This anisotropy factor is directly related to the bond stiffness and the bond stiffness gradient for both breaking and forming bonds. We show that one or both principal curvatures perpendicular to the bond path vanish at the unstable stacking fault configuration.
Supported by U.S. Army Research Office.

11:15 AM *W1.7
THEORETICAL TENSILE STRENGTH IN METALS AND INTERMETALLICS. M. Sob, L.G. Wang, M. Friak, Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, CZECH REPUBLIC; V. Vitek, Department of MS&E, University of Pennsylvania, Philadelphia, PA.

Fully self-consistent ab initio electronic structure calculations of the theoretical tensile strength in metals and intermetallics loaded uniaxially along several crystallographic directions are performed using the full-potential LAPW method. It turns out that the theoretical tensile strength and elastic anisotropy at higher strains are closely connected with the presence or absence of higher-symmetry structures along corresponding deformation paths. Total energy

calculations show that all higher-energy cubic structures studied are locally unstable with respect to tetragonal and/or trigonal deformation modes. In intermetallics, there may or may not be symmetry-dictated energy extrema corresponding to cubic lattices depending on the atomic ordering. However, other energy extrema along the deformation paths besides those required by symmetry occur. Configurations corresponding to energy minima on the deformation paths may represent metastable structures that can play an important role in interfaces and other extended defects. As specific examples, tensile strengths of single-crystalline tungsten, NiAl and $MoSi_2$ loaded uniaxially along principal crystallographic directions will be discussed.

11:45 AM W1.8
STRAIN-RATE DEPENDENT COOPERATIVE DISLOCATION GENERATION IN CRYSTALS AT FINITE TEMPERATURES: APPLICATIONS TO THE YIELDING OF WHISKERS AND THE BRITTLE-DUCTILE TRANSITION. M. Khantha, V. Vitek, R. Folk and D.P. Pope, Department of MS&E, University of Pennsylvania, Philadelphia, PA.

A new strain-rate dependent mechanism of dislocation generation that can become active suddenly above a critical temperature is described. This mechanism is a thermally driven, stress assisted cooperative instability of many dislocation loops that leads to an outburst of dislocation activity above the strain-rate dependent critical temperature. In contrast to the large temperature-independent energy barrier associated with the thermal activation of individual dislocation loops, this process is marked by an activation barrier that decreases dramatically with temperature becoming zero at the critical temperature. The strain-rate dependence originates from the glide of pre-existing and thermally nucleated dislocations below the critical temperature. We demonstrate by theory and experiments that the onset of yielding in a crack-free crystal with a very low dislocation content (Si in our study) is remarkably similar to the brittle-to-ductile transition in a pre-cracked crystal of the same material.
This research was supported by U.S. Air Force Office of Scientific Research grant F49620-98-1-0245.

SESSION W2:
Chair: Peter Gumbsch
Tuesday Afternoon, November 28, 2000
Fairfax B (Sheraton)

1:30 PM *W2.1
PREDICTABILITY OF INTRINSIC STRENGTH. T. Suzuki* and H.O.K. Kirchner**. *Institute of Industrial Science, University of Tokyo, Tokyo, JAPAN. **Institut de Sciences des Materiaux, Université Paris-Sud, Orsay, FRANCE.

So far dislocation theory has served in the interpretation of nanoscopic and microscopic observations in crystal plasticity, the aim being an understanding of the evolution of the microstructure. For a long time the approach has remained descriptive rather than predictive. Only recently a wealth of macroscopic plasticity data on bcc metals [1] and semiconductors [2, 3] have been consolidated into universal relationships describing the temperature dependence of the flow stress. This is possible as far as the deformation mechanism is an intrinsic lattice one (the Peierls process). With such universal scaling laws one can predict the strength of a crystal for which the lattice and elastic constants are known, but for which, for various reasons, plasticity data are not available, for example plutonium (e-Pu) or boron-nitride (c-BN).
[1] T. Suzuki and H.O.K. Kirchner, Phil. Mag. A, 79 (1999) 1629.
[2] T. Suzuki, I. Yonenaga and H.O.K. Kichner, Phys. Rev. Lett., 75 (1995) 3470.
[3] H.O.K. Kirchner and T. Suzuki, Acta mater., 46 (1998) 305.

2:00 PM W2.2
LOWER LIMITS OF STRENGTH IN BCC METALS AT LOW TEMPERATURES. R. Gibala, University of Michigan, Ann Arbor, MI.

The flow stress of high purity body centered cubic metals at low homologous temperatures, e.g. below about 0.15 of the absolute melting temperature, or equivalently high strain rates is govern by the relatively low intrinsic mobility of screw dislocations in the bcc crystal structure. By contrast, the mobility of edge dislocations is much higher and affords plastic deformation at correspondingly lower flow stresses. The large difference in critical flow stress for edge and screw dislocations can be demonstrated in many ways. In this paper, we examine several such methods, especially experiments in which surface films may be used to afford preferential nucleation of dislocations at the film-substrate interface and generation into the bcc substrate under applied stress. Experiments on high purity Group V and VI metals and selected solid solution alloys are described.

2:15 PM W2.3
STATIC AND DYNAMIC ELASTIC INTERACTION BETWEEN DISLOCATIONS AND POINT DEFECT CLUSTERS IN FCC METALS. Lizhi Sun, University of Iowa, Dept of Civil and Environmental Engineering, Iowa City, IA; Jianming Huang, Nasr M. Ghoniem, University of California at Los Angeles, Dept of Mechanical and Aerospace Engineering, Los Angeles, CA.

Under extreme environmental loading conditions, such as those achieved in metals irradiated by neutrons, electrons, particles or laser light, and in metals quenched from high temperatures or subjected to high-speed deformation by shock loading or high strain rates, copious concentrations of point defects and defect clusters are produced. The interaction of dislocations with point defects and their clusters results in significant modification of the materials strength. In this work, we analyze the interaction energy and configurational forces arising between dislocations and such defect clusters. A new analytical solution to the interaction energy between glissile dislocations and vacancy clusters in the form of perfect Stacking Fault Tetrahedra (SFT's) in FCC metals is first presented. This is complemented by numerical solutions for the interaction energy of truncated static SFT's. Damped Dislocation Dynamics (DD) simulations are then used to determine the necessary critical flow stress, accounting for dislocation deformation during its interaction with SFT's. The mechanisms of dislocation interaction with interstitial defect clusters are also analyzed by DD computer simulations. It is shown that substantial dislocation deformation occurs during such close-range interactions, and that the critical flow stress is achieved when dislocations reach a symmetry-breaking configuration, allowing for line tension effects to aid in dislocation break-away from interstitial defect clusters.

2:30 PM W2.4
PEIERLS STRESS AND LATTICE RESISTANCE IN FCC METALS. David L. Olmsted, Kedar Y. Hardikar, Rob Phillips, Division of Engineering, Brown University, Providence, RI.

There are only a few results available of atomistic computations of Peierls stress for fcc metals. By comparison, there are many more atomistic computations for bcc metals, as well as mixed discrete-continuum computations of the Peierls-Nabarro type for fcc metals. One of the reasons for this is the low Peierls stresses in fcc metals. Because atomistic computations of the Peierls stress take place in finite simulation cells, image forces caused by boundaries must either be relaxed or corrected for if valid results are to be obtained. We present computations of the line energies, Peierls stress and lattice resistance curves for screw, edge and mixed character dislocations in aluminum, based on an embedded-atom potential. As far as we are aware, these are the first atomistic computations of the Peierls stress of edge or mixed dislocations for an fcc metal.

2:45 PM W2.5
ATOMISTIC VIEW OF THEORETICAL STRENGTH OF BCC METALS: CORE STRUCTURE AND MOBILITY OF 1/2 <111> SCREW DISLOCATION IN Ta. Guofeng Wang, Alejandro Strachan, Tahir Cagin, William A. Goddard, III, California Institute of Technology, Materials and Process Simulation Center, Pasadena, CA.

Using a new, first principles based, Embedded-Atom-Model (EAM) potential for tantalum (Ta), we have carried out atomistic simulations to investigate the core structure, core energy and Peierls energy barrier and stress for infinite and straight 1/2 a<111> screw dislocation. Equilibrated core structures were obtained by relaxation of dislocation quadrupoles with periodic boundaries. We found that the equilibrium dislocation core has three-fold symmetry and spreads out in three <112> directions on {110} planes. Core energy per Burgers vector b was determined to be 1.36 eV/b. We studied dislocation motion and annihilation via Molecular Dynamics simulations of a periodic dislocation dipole cell, with <112> or <110> dipole orientation. In both cases the dislocations move in zig-zag on primary {110} planes. Atoms forming the dislocation cores are distinguished based on their atomic energy. In this way we can accurately define the core energy and its position not only for equilibrium configurations but also during dislocation movement. Peierls energy barrier was computed to be ~0.08 eV/b with a Peierls stress of ~0.03 u, where u is the bulk shear modulus of perfect crystal. The preferred slipping system at low temperature is <112> directions and {110} planes. Dislocation core defects, flip and kink, have been constructed and energetically relaxed. Preliminary computation results showed that formation energy of a flip is ~0.57 eV and formation energy of kink is ~0.62 eV. It is also found that flip

is a rather stable defect while kink can move freely along dislocation line. We attributed the lower experimental flow stress of dislocation to the high mobility of kink.

3:30 PM *W2.6
DEPRESSING BRITTLE FRACTURE OF IRON BY ULTRA GRAIN REFINING. Kotobu Nagai, National Research Institute for Metals, Ibaraki, JAPAN.

Low temperature brittleness is thought as an inevitable phenomenon for bcc-iron unless some alloying elements like Ni are added. However, an empirical equation predicts very low ductile-to-brittle-transition-temperatures (DBTT) for low carbon ferrite-pearlite steels that should have ultrafine grain sizes smaller than 1 micron. Industrial steels with the grain sizes of 10 micron order show the DBTT higher than 200 K. The author's group has successfully fabricated thick samples containing the ultrafine grains large enough to take full-size impact test pieces for a low carbon steel. The DBTT of such the ultrafine grain steels was lower than 77 K, which is very low comparable to that for the 9% Ni steel. The strength of the steel was almost doubled compared with that of the starting material with a 20 micron ferrite and pearlite structure.

4:00 PM W2.7
THE LIMIT OF STRENGTH AND DUCTILITY. Zhen Guo and J.W. Morris, Jr., Berkeley, CA.

Computations of the limits of strength in tension suggest that bcc metals necessarily cleave on {100} at a tensile stress of about 0.08E(<100>) while fcc metals cleave (if at all) on {110} at a tensile stress of 0.05-0.07E(<110>). Finite-element anlyses of the normal stress in the stess concentration region ahead of a sharp crack suggest that the peak tensile stress may reach four times the yield strength or more, depending on work hardening behavior. Taking these results together, we can estimate the highest values the yield strengths of typical metals and alloys could reach without forcing brittle fracture. We shall report results for a number of materials, concentrating on steel and aluminum. Interestingly, preliminary calculations suggest that the "ductile fracture limit" is exceeded by a number of existing alloy steels, but is not approached in structural aluminum alloys.

4:15 PM W2.8
ULTRAFINE GRAIN REFINEMENT BY HEAVY DEFORMATION OF SUPERCOOLED AUSTENITE AT JUST ABOVE Ar3. Chang Sun Lee, Jae Sang Lee, Jong Kyo Choi, Wung Yong Choo, POSCO, Plate, Rod & Welding Research Team, Pohang, KOREA.

Grain refinement is a promising way to enhance both the strength and the toughness of structural steels. Recently, it is known that ultrafine ferrite of around 1μm can be obtained by heavy deformation at just above Ar3 temperature during cooling of austenite. But the mechanism of ultrafine grain refinement is not clear till now because the amount of deformation is far beyond the usual one. In this paper, flow curve during deformation, dilatation curve after deformation, and microstructure were investigated in order to understand the mechanism of the grain refinement. And it was found out there is a strain induced dynamic transform during deformation of supercooled austenite to ultrafine ferrite by the analysis of dilatation curve during heating just after deformation. The grain growth rate of ferrite transformed during deformation was slower than that of ferrite transformed during cooling after deformation. And the effect of deformation temperature, amount of deformation and austenite grain size on the grain refinement and the mechanical properties was investigated in a single heavy reduction and repeated light reductions.

4:30 PM W2.9
LOW-TEMPERATURE, LARGE-STRAIN DEFORMATION BEHAVIOR OF NANO-CRYSTALLINE PALLADIUM BY MOLECULAR-DYNAMICS SIMULATION. V.I. Yamakov, D. Wolf, S.R. Phillpot, Argonne National Laboratory, Argonne, IL; H. Gleiter, Forschungszentrum Karlsruhe, Karlsruhe, GERMANY.

Molecular-dynamics simulations are used to elucidate the low-temperature tensile-deformation behavior of nanocrystalline fcc Pd up to about 90% strain with no sign of crack nucleation. The simulated model system contains 16 randomly oriented grains of uniform size and shape arranged in a periodically repeated, initially cubic box. The response of the system to a constant, uniaxial applied stress at a temperature of about 20% of the melting point reveals two stages in the deformation process. First, up to about 10% strain the system deforms uniformly, with a constant strain rate and a grain-boundary accommodated deformation process. On further straining, the accumulated stress in the system gradually relaxes, as evidenced by a rapidly increasing strain rate, until a total strain of about 70% is reached at which the strain rate decreases again. Responsible for this inhomogeneous plastic-flow behavior is a process of grain refinement associated with the nucleation of new grains from the triple junctions; the underlying mechanism involves stress-induced amorphization of the triple junctions followed by recrystallization and the formation of new grains. As evidenced by the reduction in the strain rate, the newly formed grain microstructure, containing more spherically shaped but smaller grains, apparently accommodates the highly elongated system shape better than the original microstructure with highly elongated grains. This mechanism of grain refinement during plastic flow might provide an explanation for the appearance of spherical grains even after rather substantial plastic elongation under uniaxial stress.
*Work supported by the U.S. Department of Energy, Office of Science, under Contract W-31-109-Eng-38.

4:45 PM W2.10
PHASE FIELD MICROELASTICITY APPROACH TO DYNAMICS OF DISLOCATION SYSTEM. Yu U. Wang, Rutgers Univ, Dept of Mechanical and Aerospace Engineering, Piscataway, NJ; Yongmei M. Jin, Rutgers Univ, Dept of Ceramics and Materials Engineering, Piscataway, NJ; Alberto M. Cuitiño, Rutgers Univ, Dept of Mechanical and Aerospace Engineering, Piscataway, NJ; Armen G. Khachaturyan, Rutgers Univ, Dept of Ceramics and Materials Engineering, Piscataway, NJ.

The elastic strain and the strain energy generated by the dislocation system of arbitrary geometry are formulated in terms of the Phase Field Microelasticity (PFM) theory previously proposed by one of the authors. The exact solution for the strain energy is presented as a functional of the phase fields describing any spatial configuration of dislocations. Incorporating the PFM solution in 3-D Phase Field kinetic model results in the kinetic equations governing an evolution of an arbitrary dislocation pattern under applied stress. The proposed model automatically takes into account the "short-range interactions", such as multiplication and annihilation of dislocations as well as a formation of various metastable microstructures involving dislocations and defects. This model, as any Phase Field model, does not impose *a priori* constraints on dislocation patterns formed during the evolution. The Phase Field Dislocation model is used to simulate the mechanical behavior during plastic deformation, such as strain hardening and plastic flow. Nucleation and critical flow stresses for dislocations are studied. Examples of simulation of the 3-D fcc system are discussed.

SESSION W3:
Chair: Nasr M. Ghoniem
Wednesday Morning, November 29, 2000
Fairfax B (Sheraton)

8:30 AM *W3.1
TECHNICAL AND REGULATORY DEVELOPMENTS NEEDED TO ENABLE APPLICATION OF MASTER CURVE TECHNOLOGY TO THE FRACTURE INTEGRITY ASSESSMENT OF COMMERCIAL NUCLEAR POWER REACTORS. Mark Kirk, U.S. Nuclear Regulatory Commission, Rockville, MD.

The Master Curve, as introduced by Wallin and co-workers in 1984, has evolved into a mature technology for characterizing the fracture toughness transition of ferritic steels. Considerable empirical evidence provides testament to the robustness of the Master Curve procedure. However, in 1997, the NRC staff detailed several technical issues requiring resolution prior to staff acceptance of applications of Master Curve technology to the fracture integrity assessment of nuclear RPVs. Current and recently completed research programs sponsored by both the NRC and EPRI focus on closure of these issues. This paper reviews the issues detailed in 1997, comments on their continued relevance in light of recent research results, and details areas where either additional research or a change or research focus is warranted.

9:00 AM W3.2
DISCRETE DISLOCATION SIMULATION OF THIN FILM PLASTICITY INCLUDING THE EFFECT OF DISLOCATION SOURCES. Burghard von Blanckenhagen, Peter Gumbsch, Eduard Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

The flow stress of thin metal films is much higher than the flow stress of the corresponding bulk material and scales approximately with the inverse film thickness. This was shown in many experiments but the mechanisms are not yet fully understood. A discrete dislocation dynamics simulation is used here to investigate dislocation motion in thin metal films. A thin polycrystalline film with columnar grains on a substrate was simulated. As a first step, grain boundaries are introduced as impenetrable obstacles for the dislocations. In analogy to the Hall–Petch model it is assumed that flow in a neighbouring grain is achieved if the force on the grain boundary exceeds a certain critical value. Dislocation sources are included explicitly. Work hardening is contained in the back stress of the dislocations on the

source. The calculations show that the film thickness and grain size have to be treated equivalently, the smaller of the two controlling the flow stress. It is found that the optimum dislocation source size is approximately one fourth of the film thickness (or grain size) if the source is of Frank–Read type and if a source is required to operate several times. In the regime of grain and source sizes treated here, the Hall-Petch relation has to be modified to include source effects. For small grains the source term dominates and one finds a flow stress dependence on the inverse film thickness (or grain size). The results indicate that the effect of dislocation sources has to be included into a theory of thin film plasticity.

9:15 AM W3.3
QUANTITATIVE DETERMINATION OF THE EFFECT OF ELASTIC ANISOTROPY ON GRAIN-BOUNDARY DISLOCATION NUCLEATION AND DISLOCATION GLIDE IN Al (011) THIN FILMS ON Si (100). E.A. Stach, U. Dahmen, National Ctr.r for Electron Microscopy, Lawrence Berkeley Natl. Lab, Berkeley, CA; W.D. Nix, Dept. of MS&E, Stanford Univ., Palo Alto, CA.

Quantitative experimental observations of dislocation nucleation and glide in the model Al (011) / Si (100) thin film system are presented. Using a simple physical vapor deposition technique, a thin film composed solely of two symmetry-related grain orientations of (011) aluminum on Si (100) substrates can be created. This provides an ideal system within which to determine the specific behavior of dislocations during thermal stress relaxation. In prior work, it was shown that in-situ transmission electron microscopy allows real time observation of dislocation climb, cross-slip, grain boundary nucleation and glide. In particular, it was found that upon cooling from an anneal at 450°C, dislocation nucleation occurs preferentially at the cusps in the grain boundaries, beginning at a temperature of 275°C, followed by slip along the inclined {111} glide planes. Subsequent nucleation and glide on these same slip systems occurs sporadically upon further cooling, until the sample reaches a temperature of 75°C. At this point, dislocation nucleation and glide occurs instantaneously along all four a/2 <110> {111} slip planes. Herein, we show that the observed behavior is a direct result of the anisotropy of stress in an (011) oriented film. Anisotropic elasticity theory shows that the magnitude of the resolved shear stress on particular Burgers vectors of dislocations that lie on inclined {111} slip planes is substantially higher than on the perpendicular {111} planes at this temperature. The calculations indicate that the stress on these dislocations at 275°C is on the order of 350 MPa. At a temperature of 75°C, the same analysis indicates a nearly identical level of stress on another set of dislocations that reside on both the inclined and the perpendicular slip planes, thereby directly explaining the experimental observations. These results indicate that grain boundary nucleation of dislocations may play a critical role in the observed high strengths of metallic thin films on rigid substrates.

9:30 AM W3.4
INELASTIC DEFORMATION MECHANISMS IN POLYCRYSTALLINE FCC METALLIC THIN FILMS. Mauro J. Kobrinsky, Carl V. Thompson, MIT, Dept. of MS&E, Cambridge, MA; Mihal E. Gross, Bell Labs, Lucent Technologies, Murray Hill, NJ.

While the fine microstructure of polycrystalline metallic thin films is believed to be the origin of their high strength, a comprehensive understanding of the connection between strength and microstructure is still lacking. To explore the relations between strength and characteristic length scales, we performed experiments on submicron-thick Ag and Cu films on Si substrates, as well as submicron-size Cu interconnects (lines) on substrates. In particular, we will report on the effects that grain size, film thickness or line width, characteristic length of the dislocation entanglement, and temperature have on the flow stress of films and lines. We have found that the mechanical properties of the films and the lines are determined by two main inelastic mechanisms: diffusional creep and dislocation-mediated plasticity controlled by the thermally-activated motion of dislocations through obstacles, which presumably are forest dislocations. As expected, diffusional creep is dominant at high temperatures and small grain sizes, while dislocation-mediated plasticity is dominant at low temperatures and large grain sizes, the transition temperature being a function of film thickness and average grain size. For diffusional creep, the relevant length scale is determined by the film thickness and average grain size, while in dislocation plasticity, the relevant length is the average distance between the pinning points, which was found to be on the order of 50-100 nm. This value is substantially smaller than the average grain size, suggesting that dislocation-dislocation cutting is the process that determines the low-temperature strength of the films. Finally, we have also found that the strength of the films first increases with decreasing film thickness and grain size, but then reaches a maximum value to finally decrease with decreasing thickness and grain size. This behavior is the result of the interplay between dislocation-mediated plasticity and diffusional creep.

10:15 AM W3.5
STRESS DEPENDENT STRUCTURE OF 90° PARTIAL DISLOCATION IN Si. Karin Lin, D.C. Chrzan, Department of Physics, University of California, Berkeley, CA and Center for Advanced Materials; Ernest Orlando Lawrence Berkeley National Laboratory, Berkeley, CA; Department of MS&E, University of California, Berkeley, CA and Center for Advanced Materials, Ernest Orlando Lawrence Berkeley National Laboratory, Berkeley, CA.

The ability to predict the stable core structure of dislocations is an essential step toward predicting their mobility. Dislocation mobilities, in turn, influence the lower limit of strength. Ideally, core structure predictions should be constructed using *ab initio* electronic structure total energy techniques. This prediction is complicated, however, by the need to either study artificial arrangements of dislocations (*i.e.* periodic supercells), or by studying small, cylindrical systems while capping the dangling surface bonds. In the periodic supercell technique, the boundary conditions one imposes may exert a significant influence on the core structure of the dislocation. The use of periodic supercells for the prediction of the relative stability of the two proposed core structures for the 90° partial dislocation in Si (the so-called single-period and double-period core reconstructions) is explored. The total energies of a number of different cell configurations are computed using Tersoff potentials. It is demonstrated that: 1) The periodic supercell technique produces results equivalent to those obtained by imposing cylindrical boundary conditions. 2) Tersoff potentials predict that the relative stability of the competing single period and double period core structures depends systematically on the applied shear stress. And 3) Tersoff potentials predict that pressure induces a transition in the stable core structure, with high pressure favoring the single-period core reconstruction.
The support of the Director, Office of Energy Research, Office of Basic Energy Sciences, U.S. Department of Energy under contract No. DE-AC03-76SF00098 is gratefully acknowledged.

10:30 AM W3.6
ATOMISTIC SIMULATION OF KINK-PAIR ENERGETICS OF SCREW DISLOCATIONS IN BCC IRON. Alfonso H.W. Ngan, Department of Mechanical Engineering, The University of Hong Kong, Hong Kong, PR CHINA; M. Wen, Advanced Materials Section, Production Fundamentals Division, Chugoku National Industrial Research Institute, AIST, MITI, Hiroshima, JAPAN.

In bcc iron, there are two degenerate states A and B of the screw dislocation core. Motion of the screw dislocation core would hence proceed by jumps from A to B, A to A, etc. In this work, the nudged elastic band method is used to calculate the activation paths connecting such jumps. Kink-pairs are found to involve in all such jumps. These kink-pairs are denoted according to the Duesbury scheme as BnApB, AnBpA, AnApA and so on, where n and p denote the sense of the kinks, the first and third capital letters denote the state before jumping, and the middle capital letter denotes the state after jumping. Based on symmetry arguments, the set of possible kink-pairs is shown to reduce to only two non-equivalent heteromorphous kink-pairs, BnApB and AnBpA, and one homomorphous kink-pair, AnApA. The simulation results also show that the heteromorphous kink-pair BnApB has significantly lower activation energy than the other heteromorphous kink-pair AnBpA as well as the homomorphous kink-pair AnApA. Hence, under a "negative" applied stress, the B to A jump is easy, but the A to B jump is difficult. The situation under a "positive" applied stress is reversed. The difficult jump on the primary{101} plane can however be by-passed by jumping onto an intersecting cross-slip plane, the morphology of which is equivalent to the easy jump on the primary plane. The motion of the screw core would therefore be zig-zag and the average slip plane would tend towards the mrss plane. We believe this is the proper explanation of the observed pencil glide at elevated temperatures. The activation energy of the rate-controlling BnApB kink-pair drops rapidly with increasing applied stress. The predicted flow stress at each temperature, however, is always about three times of the experimental value.

10:45 AM *W3.7
THE ULTIMATE STRENGTH OF MULTILAYER THIN FILMS. Peter M. Anderson and Zhiyong Li, Ohio State University, Dept of MSE, Columbus, OH.

Many multilayer thin films consisting of alternating layers of metallic, intermetallic, or ceramic phases display a peak strength at a critical bilayer thickness. In systems for which crystal slip intervenes as the limiting strength process, a critical event appears to be the transmission of dislocations across interfaces. At larger layer thickness, deformation occurs by confined layer slip within individual layers, followed by slip across interfaces. In this regime, strength monotonically increases with decreasing layer thickness. At smaller layer thickness, the stress for confined layer slip becomes so large that

dislocation transmission intervenes first. In this smaller thickness regime, the dislocation must propagate across multiple layers before it is able to expand parallel to interfaces. A consequence is that the strength of the multilayer thin film is dictated by interfacial strength to transmission. A continuum-level, Peierls model is employed to study the critical stress for dislocation transmission across interfaces. The results suggests that interfaces can be made stronger to slip transmission by making the interface weaker to in-plane slip. In such cases, the dislocation core can become trapped in the interface and a significant stress is required to extract the core from the interface. For multilayer films for which cracking intervenes as the limiting strength process, a similar analogy to the dislocation process holds. In particular, there is a critical layer thickness below which the stress to propagate a confined layer crack becomes so large that crack transmission across interfaces intervenes first. By analogy, the limiting strength is dictated by interfacial resistance to crack transmission.

11:15 AM W3.8
THE TEMPERATURE DEPENDENCE OF MICROCRACK ARREST IN FERRITE AND ITS RELATIONSHIP TO A UNIVERSAL MASTER TOUGHNESS CURVE SHAPE. M. Hribernik, G.R. Odette and M.Y. He, University of California, Santa Barbara, Dept of Mechanical and Environmental Engineering, Santa Barbara, CA.

Considerable empirical evidence suggests that the cleavage initiation toughness curve for structural steels in the brittle to ductile transition has a approximately universal KJc(T-Tr) shape on a relative temperature scale (T - Tr). Microstructural and strength differences in steels, and even other bcc alloys, primarily result in variations in the reference temperature (Tr) but have relatively little impact on the master curve (MC)-type shape. A new micromechanical model combining analytical representations of finite element analysis (FEA) simulations of crack tip stress fields with a local critical stress-critical stressed area (s*-A*) fracture criterion is consistent with a universal MC shape if the s* increases modestly with temperature in the regime where the yield stress approaches its athermal limit. The s* is the stress needed to propagate a dynamic microcrack from a broken trigger particle, and is usually given by a Griffith-type criteria related to the size of the trigger particle and the micro-arrest toughness of the metal matrix, Kmicro. Thus the most general and fundamental explanation of a universal MC shape, is a corresponding increase in Kmicro with temperature. Various experimental approaches to evaluating Kmicro(T) are described, including measurement of crack arrest toughness in oriented iron single crystals. This method involves compression loading of composite diffusion bonded specimens composed of a brittle phase region for dynamic crack initiation and an oriented single crystal crack arrest region embedded in polycrystalline iron compression loading bar. The implications of the observed Kmicro(T) behavior to the shape of the MC are described along with a dislocation dynamics model.

11:30 AM *W3.9
LOCAL AND GLOBAL FAILURE MODES IN POLYCRYSTALLINE AGGREGATES. W.M. Ashmawi and M.A. Zikry, North Carolina State University, Department of Mechanical and Aerospace Engineering, Raleigh, NC.

A multiple-slip dislocation-density based constitutive formulation and specialized computational schemes have been developed to characterize material failure on the appropriate physical scales needed for the accurate prediction of physical mechanisms that control failure initiation, growth, and coalescence. Dislocation-density transmission and blockage interfacial conditions and local stress fields have been obtained for grain-boundary distributions associated with random and tilt orientations. These evolving local stress fields are used as failure criteria to track the initiation and evolution of intergranular and transgranular fracture. The interrelated effects of grain boundary orientation, dislocation density pile-ups and evolution, geometrical and thermal softening, void distribution and geometry, and hydrostatic stresses on failure paths in cubic crystalline materials have been studied. Based on the present analysis and on comparison with experimental observations, it is shown transgranular and intergranular failure can be characterized in terms of the competition between the strengthening and the softening mechanisms of the crystalline structure.

SESSION W4/B5: JOINT SESSION
Chairs: Daryl C. Chrzan and Richard G. Hoagland
Wednesday Afternoon, November 29, 2000
Room 310 (Hynes)

1:30 PM *W4.1/B5.1
THE INVERSE HALL-PETCH EFFECT - FACT OR ARTIFACT? Carl Koch, North Carolina State Univ., Dept. of MS&E, Raleigh, NC.

The first observations of Chokshi et al (1) of softening with decreasing grain size for nanocrystalline Cu and Pd stimulated interest in this apparent inverse Hall-Petch behavior for decreasing grain size in the nanoscale regime. However, it was subsequently pointed out that for most of the experiments where inverse Hall-Petch behavior was observed, the grain size was changed by annealing the initially finest grain size specimens to grow the grains. It was suggested that thermally treating nanocrystalline samples may cause changes in the structure (eg. decreases in porosity, changes in grain boundary structure, etc.) along with the grain growth, and these changes may be responsible for the inverse Hall-Petch behavior. In spite of the uncertainty in the experimental evidence for the inverse Hall-Petch effect, a number of mechanisms have been proposed for its occurrence. Molecular dynamics simulations have also predicted the inverse Hall-Petch effect. Since conventional dislocation-induced deformation mechanisms are unlikely at the finest nanoscale grain sizes, changes in the grain size dependence of hardness and strength are not surprising. This paper will critically review the available experimental evidence for the inverse Hall-Petch effect and discuss it in the context of the simulation studies and the various proposed mechanistic models.
1. A.H. Chokshi, A. Rosen, J. Karch, H. Gleiter, Scripta Metall. 23, 1679 (1989).
Support from NSF under grant no. DMR-9871980.

2:00 PM W4.2/B5.2
LARGE TENSILE DUCTILITIES AND ULTRAHIGH YIELD STRENGTHS AT ROOM TEMPERATURE IN NANOCRYSTALLINE FeCo ALLOYS. Chang He Shang, D. Van Heerden, R.C. Cammarata, The Johns Hopkins University, Department of MS&E, MD; C.L. Chien, The Johns Hopkins University, Department of Physics and Astronomy, Baltimore, MD; T.P. Weihs, The Johns Hopkins University, Department of MS&E, Baltimore, MD.

Researchers have pursued nanocrystalline materials for structural applications for more than a decade because materials with grain sizes below 1 micrometer have been predicted to display and, in some case, have shown significant improvements in mechanical properties compared to coarse-grained materials. High yield strengths and superplasticity have been demonstrated, and enhanced ductility has been predicted for nanocrystalline intermetallics. However, while nanocrystalline Cu has shown enhanced ductility at room temperature, nanocrystalline intermetallics typically fail in the elastic regime with less than 1% strain-to-failure. The extreme brittleness in nanocrystalline intermetallics is generally attributed to flaws or porosity that are produced during fabrication. In this study, we have investigated the mechanical properties and microstructures of fully dense, single-phase nanocrystalline FeCo alloys that were fabricated using bulk processing techniques. The nanostructured alloys possess very attractive mechanical properties at room temperature with tensile yield strengths as high as 2.2 GPa and tensile ductilities ranging from 3% to 18%. Both tensile strengths and hardnesses follow Hall-Petch relationships, and the fracture surfaces show ductile features. Results from TEM, SEM, and X-ray diffraction investigations will be used to describe the microstructure of these flaw-free, nanocrystalline materials.

2:15 PM W4.3/B5.3
DEFORMATION BEHAVIOR AND FAILURE MODE OF CONSOLIDATED FULL-DENSITY NANOPHASE Fe AT QUASI-STATIC AND DYNAMIC STRAIN RATES. D. Jia[1], Y.-M. Wang[2], K.T. Ramesh[1,2] and E. Ma[1]. [1]Dept. of Mechanical Engr. [2]Dept. of MS&E, Johns Hopkins Univ., Baltimore, MD.

Full density Fe with grain sizes in the nanophase to submicron range has been consolidated from mechanically milled powders. The deformation behavior of such materials, as a function of grain size, strain rate, and temperature, have been studied using quasi-static and high strain rate (Kolsky bar) tests. With ultrafine grain sizes, Fe exhibits high strength, little work hardening, and plastic strains localized in shear bands. Shear banding appears to be the dominant mode from the onset of the plastic deformation in our consolidated materials. Little strain rate sensitivity of the flow stress is observed over a wide strain rate range (up to 5E5/s). These behaviors are contrasted with those of conventional bulk Fe, which shows uniform deformation, significant work hardening, and strong strain rate sensitivity. The underlying deformation and failure mechanisms are discussed based on these observations. With increasing grain sizes and/or temperature, the yield strength decreases and plastic strain increases. These findings are compared with the Hall-Petch relationship, and examined to derive the validity range, in terms of grain size, temperature, strain rate, and particle bonding strength, of different deformation mechanisms (including grain boundary mechanisms). In addition, we comment on the potential advantage of related bcc alloys in military kinetic energy penetrators where the shear banding mode and self-sharpening capability during high rate deformation are desired.

2:30 PM *W4.4/B5.4
THE LIMIT OF GRAIN REFINING STRENGTHENING IN IRON.
Setsuo Takaki, Kenji Kawasaki, Kyushu Univ, Dept of MS&E, Fukuoka, JAPAN; Yuuji Kimura, National Research Institute for Metals, Ibaraki, JAPAN.

Mechanical milling (MM) using a high energy ball mill was applied to iron powder to obtain ultra fine-grained structure. The MM treatment causes a marked increase in the hardness of iron powder from $Hv_{0.5}$GPa to $Hv_{9.5}$GPa. Initial hardening is due to well-known dislocation strengthening but the latter hardening (above Hv_6GPa) is mainly due to grain refining strengtening. The grain size of iron powder with the hardness $Hv_{9.5}$GPa was about 25nm. In the Hall-Petch plot of such ultra fine-grained iron, a linear Hall-Petch relationship was confirmed to around 100nm but the plots below 100nm in the grain size deviated toward lower hardness side from the extended Hall-Petch line. In Fe-0.8%C powder, grain size was refined to 10nm but the hardness was also much smaller than the value expected by the extended Hall-Petch line and showed a tendency of levelling-off at Hv_{12}GPa. As a result, the grain size of iron is seemed to be refined to around 10nm by severe deformation and the hardness achieved by grain refining is thought to be about Hv_{12}GPa (expected yield strength: 3.6GPa).

3:30 PM *W4.5/B5.5
ATOMISTIC STUDIES OF PLASTICITY IN NANOPHASE METALS.
A. Caro, Centro Atomico Bariloche, Bariloche, ARGENTINA; H. Van Swygenhoven, P. Derlet, Paul Scherrer Institute, Villigen, SWITZERLAND; D. Farkas, Dept. of MS&E, Virginia Polytechnic Inst. & State Univ., Blacksburg, VA; M.J. Caturla and T. Diaz de la Rubia, Lawrence Livermore Natl. Lab., Livermore, CA.

We have studied structural, energetic, elastic, and plastic properties of a family of computer generated nanophase samples of Cu and Ni, continuously increasing the average grain size, covering now the range from 20 to 3.5 nm. Properties that can directly be compared to experiments, like density, excess enthalpy, and elastic constants, show a remarkable agreement suggesting that the computer modeled materials are able to capture the essential features of the real nanophase metals. Investigating the structure of the grain boundaries on the atomic level, we found that grain boundaries in nanophase metals are essentially similar to those found at the micro scale, i.e. similar structural units are found, providing evidence against the view of grain boundaries in nano-crystals as amorphous or liquid-like interfaces. One of the most striking features is the observation of a change in plastic regime as the grain size approaches the ~10 nm region. In fact, intragrain dislocation activity ceases to operate below this size and grain boundary sliding, a process based on mechanical and thermally activated single atomic jumps, dominates the contribution to deformation. It in turn implies that deformation becomes easier as the grain size further decreases. This observation is of primary importance in the interpretation of the 'inverse Hall-Petch relation' reported experimentally. The computer simulations provide access to a microscopic view of the deformation processes. In this way we obtain a detailed description of the structure and energetics of grain boundaries and triple junctions, identifying the regions where dislocations are emitted when the grain size is large enough, as well as the mechanism on atomic level of grain boundary sliding. We extract quantitative information about the conditions for dislocation emission and the role of the atomic structure of the grain boundaries.

4:00 PM *W4.6/B5.6
GRAIN-BOUNDARY CONTROLLED DEFORMATION OF NANOCRYSTALLINE MATERIALS BY MOLECULAR-DYNAMICS SIMULATION.
Dieter Wolf, Materials Science Division, Argonne National Laboratory, Argonne, IL.

Molecular-dynamics (MD) simulations of model nanocrystalline microstructures demonstrate that the high-temperature plastic-deformation process involves grain-boundary diffusion creep. In order to suppress grain growth and thus to enable steady-state deformation to be observed on a time scale accessible to MD simulations (of typically 10-9s), our input microstructures were tailored to (i) have a uniform grain shape and a uniform grain size of up to about 10 nm and (ii) contain only high-energy grain boundaries which are known to exhibit rather fast, liquid-like self-diffusion. Our simulations reveal steady-state diffusion creep that is homogenous (i.e., involving no grain sliding), with a strain rate that agrees quantitatively with that given by the Coble-creep formula. The extension of these results to lower temperatures, with emphasis on the crossover in the Hall-Petch effect, is discussed.
Work supported by the U.S. Department of Energy, Office of Science, under Contract W-31-109-Eng-38.

4:30 PM W4.7/B5.7
GRAIN MICROSTRUCTURE EVOLUTION IN NANOSCALE MATERIALS - RECENT SIMULATION RESULTS.
Moneesh Upmanyu[1,2,3] and David J. Srolovitz[1,2]. [1]Princeton Materials Institute, Princeton, Princeton University, NJ. [2]Dept. of Mechanical and Aerospace Engineering, Princeton University, Princeton, NJ. [3]Dept. of MS&E, University of Michigan, Ann Arbor, MI.

The mechanical properties of many materials varies with grain size. For example, the yield strength varies with grain size as in the well-known Hall-Petch relation and creep properties vary with grain size in Coble creep. Such effects are especially important on the nano-scale. In this study, we examine several issues associated with the evolution of the grain structure. In particular, we study several important issues in grain size evolution that only become important when the grain size is very small. These studies were performed using molecular dynamics simulation in two- and three-dimensions. We examine the range of grain sizes over which the classical concepts of grain boundary mobility and curvature driven growth apply and the effects of grain boundary triple junctions and grain rotation on grain boundary migration. These latter effects are commonly neglected in models for grain microstructure evolution. The present results suggest that the boundary velocity is proportional to boundary curvature, at large grain sizes, but increases superlinearly with decreasing grain sizes at very small grain sizes. We have also explicitly extracted grain boundary triple junction mobilities for a large number of grain misorientations. We find that while the triple junctions provide a drag on boundary motion, this drag is negligible at all grain sizes (down to a couple of nanometers). However, we have identified several tri-crystallographies where the triple junction mobility is low enough to significantly slow boundary migration. Finally, simulations of shrinking grains demonstrate that at sufficiently small size, grains rotate as they shrink. Interestingly, this rotation is not completely consistent with expectations based on the misorientation dependence of the boundary energy and does not require the motion of lattice dislocations.

4:45 PM W4.8/B5.8
THE HALL-PETCH RELATION IN NANOCRYSTALLINE METALS.
J. Schiøtz, Center for Atomic-Scale Materials Physics and Department of Physics, Technical University of Denmark, Kongens Lyngby, DENMARK.

It has not yet been settled to what extent nanocrystalline metals obey the Hall-Petch relation, i.e. whether the yield stress and hardness increase with decreasing grain size. There are conflicting experimental data concerning a possible reverse Hall-Petch effect, a softening with decreasing grain size at the smallest grain sizes. The experimental ambiguities are probably caused by the extreme difficulty of manufacturing samples of sufficient quality. We have previously used atomic-scale simulations to demonstrate that nanocrystalline copper and palladium with grain sizes below 13nm exhibit a reverse Hall-Petch effect, caused by sliding in the grain boundaries [Schiøtz et al., *Nature* **391**, 561 (1998); *Phys. Rev. B* **60**, 11971 (1999)]. Based on these simulations, a model is presented that attempts to give a unified description of the yield stress of polycrystalline metals for all grain sizes. The model is based on a competition between two deformation mechanisms: activation of dislocation sources in the grains and grain boundaries, and grain boundary sliding.

SESSION W5: POSTER SESSION
Wednesday Evening, November 29, 2000
8:00 PM
Exhibition Hall D (Hynes)

W5.1
HARDNESS OF SEMICONDUCTORS.
Ichiro Yonenaga, Tetsuya Hoshi, Institute for Materials Research, Tohoku University, Sendai, JAPAN.

Hardness of various semiconductors such as Si, GaAs, ZnSe, GaN, GeSi alloys, etc. was surveyed in a wide temperature range from RT to close to the melting point of the material. The Vickers indentation hardness of (111) surfaces of the crystals of diamond-type and sphalerite-type, shows a typical temperature dependence: a gradual decrease from RT to intermediate temperature, then a steep decrease with increase of the temperature. The hardness of (001) surfaces of GaN and SiC shows a steep decrease at high temperatures following a plateau in the intermediate temperature range. The temperature dependence of the hardness, H_v, of the crystals can be described by a universal relationship H_v/G vs kT/Gb^3, where G is the shear modulus and b the magnitude of the Burgers vector. The steep decrease of H_v at elevated temperatures can be described by the phenomenological relationship $H_v = H_{v0}\exp(U/kT)$. The estimated

value U for various semiconductors is proportional to the activation energy of dislocation motion previously reported for these crystals.

W5.2
THERMALLY-ACTIVATED INITIATION OF PLASTICITY IN SILICON. N. Yang and Y.Q. Sun, Department of MS&E, University of Illinois, Urbana, IL.

Thermally-activated initiation of plastic deformation in single crystal Si is studied. Samples oriented for single slip are loaded by a dead-weight in compression or shear while the sample temperature is continuously increased through the point of brittle-to-ductile transition. The initiation of plastic deformation at the critical temperature, in particular the initial transient plastic strain-rate, is investigated using high-resolution displacement sensors with a fast data-acquisition rate. Particular attention is given to the extent of plastic strain localization and the local strain-rates. The transition temperature is measured against the stress. The relation found between stress and the critical temperature is used to extract the thermal activation parameters. The results are compared with plasticity initiation models based on dislocation nucleation and multiplication.

W5.3
ON THE EFFECTIVE TENSILE STRENGTH OF LIQUIDS UNDER DYNAMIC STRESSING: THEORY AND EXPERIMENT. P. Rhodri Williams, Stephen W.J. Brown, Rhodri L. Williams, University of Wales-Swansea, Centre for Complex Fluids Processing, Dept. of Chemical and Biological Process Engineering, Swansea, UNITED KINGDOM.

We consider some discrepancies arising between different measurements of the effective tensile strength of liquids under dynamic stressing, in experiments involving the creation of negative pressure (or tension) by the reflection of a pressure pulse at a free surface. We critically examine the structure of the pressure records reported in such experiments and conclude that a key feature of them may have been misinterpreted in previous work. A complete explanation of such pressure records is presented here: this explanation suggests a hitherto unsuspected mechanism for the order of magnitude discrepancies in dynamic tensions recorded in previous dynamic stressing experiments. We present a new method of estimating the effective tensile strength of liquids using an extrapolation procedure in the range of static positive pressures. This method, which provides an estimate of the maximum dynamic negative pressure sustained by a liquid, differs from previous techniques which have involved long extrapolations into the negative pressure regime. The results obtained indicate that deionised water can sustain a transient tension of nearly 300 bar, reconciling the results of pulse reflection work with those of other dynamic stressing techniques. Results are also reported for other liquids, including dilute solutions of high molecular weight polymers, liquid mercury and multigrade motor lubricants at elevated temperatures.

W5.4
MOLECULAR-DYNAMICS STUDY OF INSTABILITIES IN CRYSTALS UNDER CONSTANT STRESS. Jianhua Zhao, Frederick Milstein and Dimitrios Maroudas, Univ. of California, Santa Barbara, CA.

Mechanical, geometric, and kinetic characteristics of crystal instabilities are revealed via isostress molecular-dynamics (MD) simulations according to the Lagrangian formulation of Parrinello and Rahman. Results are analyzed in terms of the elastic stability criteria of Hill and Milstein. Fluctuation formulae are used for computation of elastic moduli employed in assessments of elastic stability. Interatomic interactions in metals are expressed both by simple Morse pair potentials and by more accurate semi-empirical embedded-atom-method potentials that have been parametrized specifically for studies of crystal elasticity at finite strain. As an example, the stability of bcc crystals was studied under constant pressure and temperature. It was found that violations of the elastic stability criteria predict the onset of bcc-to-hcp transitions. Computations were carried out of the crystals' shear moduli at each pressure and temperature and of the evolution of the crystals' enthalpies along the transformation paths. Under isothermal-isobaric conditions, the bcc-to-hcp transitions proceeded according to the Burgers mechanism. Most importantly, the transitions were found to originate with thermally activated elastic instabilities that are associated with vanishing or diminishing shear moduli.

W5.5
Abstract Withdrawn.

W5.6
FIRST-PRINCIPLES STUDY OF THE STRENGTH AND FRACTURE OF GRAIN BOUNDARIES IN SILICON CARBIDE. Masanori Kohyama, Dept of Material Physics, Osaka National Research Institute, Ikeda, Osaka, JAPAN.

It is of great importance to understand the mechanical properties of SiC grain boundaries, which seriously affects bulk ceramic properties. First of all, it is essential to clarify the behavior of grain boundaries under various tensile or shear stresses. Currently, first-principles calculations using the pseudopotential method based on the density-functional theory can be applied to such complex problems. We have performed the *ab initio* tensile tests of a coincidence tilt boundary in cubic SiC. One non-polar interface [1] and two polar interfaces of the Σ=9 boundary are examined. These interfaces have the same bonding network, although the polar ones contain either C–C or Si–Si wrong bonds and the non-polar one contains both kinds of wrong bonds. In the tensile tests, the supercell is stretched in a small increment, and all the atoms are relaxed. This cycle is iterated until the interfaces are broken. This corresponds to a real tensile test at T=0K. Young's modulus, tensile strength, fracture energy, and fracture toughness are obtained for these interfaces. Features of fracture, bond breaking and changes in the electronic structure are analysed in detail. Results of each interface are compared with each other and with bulk SiC. We discuss the effects of C–C and Si–Si wrong bonds on the tensile strength and fracture, and the effects of temperature and impurities.
[1] M. Kohyama, Phil. Mag. Lett. 79 (1999) 659.

W5.7
MICROMECHANICAL STRUCTURES MADE FROM HARD STRESS-FREE AMORPHOUS DIAMOND THIN FILMS. T.A. Friedmann, R.J. Hohlfelder, J.P. Sullivan, D.A. Lavan, M.P. de Boer, P.G. Kotula, T.M Alam, C.I.H. Ashby, M.T. Dugger and M. Mitchell, Sandia Nat. Laboratories, Albuquerque, NM.

Amorphous-Diamond (a-D) thin films deposited by pulsed-laser deposition typically have high levels (6-10 GPa) of residual stress. This stress is thought to be intrinsic to the deposition process, but is not intrinsic to hard (> 85 GPa) a-D films, since thermal annealing to moderate temperatures (600°C) can completely remove the stress without significantly changing the 4-fold carbon content. We have taken advantage of these low stress a-D films to create true surface micromechanical structures (cantilever beams, tensile pull tabs, notched beam pull tabs, and friction test structures) to evaluate the micromechanical properties of these films. For example, early tensile test results done with a nanoindentor show that the a-D fracture strength is quite high, > 8 GPa, much higher than CVD diamond films (~1 GPa) which tend to be highly defective. In addition cantilever beam bending experiments show that the modulus of this material is also quite high (> 700 GPa) and does not change significantly with annealing to 950°C. Raman analysis of the annealed a-D films shows that the character of the 3-fold carbon bonds change with annealing while EELS on plan-view samples indicates almost no change in the 4-fold content. We are in the process of making spatially resolved EELS measurements on cross-sectioned samples of annealed a-D to investigate the bonding topology and attempt to relate it to the enhanced mechanical properties reported above for these films. In addition, we have prepared samples with enriched 13C for NMR analysis.
*This work was supported by the U.S. DOE under contract DE-AC04-94AL85000 through the Laboratory Directed Research and Development Program, Sandia National Laboratories.

W5.8
FAILURE OF ELECTRODEPOSITED METALLIC NANO-STRUCTURES. Fereshteh Ebrahimi, MS&E Dept., University of Florida, Gainesville, FL.

We have fabricated nanolayered and nanocrystalline samples of FCC metals via electrodeposition techniques. The properties of these nanostructures are characterized using x-ray diffraction, transmission electron microscopy, scanning electron microscopy, microprobe analysis, and tensile testing techniques. The single-phase nanostructures failed in a ductile manner when the fabricated material did not have weakened paths such as grain boundaries with high concentrations of impurities or inter-nodular boundaries. Excessive co-deposition of hydrogen resulted in formation of ultra-fine voids, which led to microvoid coalescence fracture mechanism. Otherwise, all electrodeposited single-phase FCC nanocrystals fractured by the so-called knife-edge mechanism. The limit strength of these materials will be discussed in terms of work hardening and plastic instability. The laminated nanostructures consisting of two different metallic layers with FCC crystal structures, however, showed cleavage-like facets. One explanation is that local stresses as high as the theoretical strength develop in nanocomposites materials. The other possibility is that delamination at interfaces or a high density of processing voids provides the opportunity for tearing of ultra-fine ligaments, which look like cleavage facets. The possibilities of these mechanisms are discussed in Cu/Ag and Cu/Ni nanolayered structures.

W5.9
THE EVALUATION OF ELASTIC MODULUS AND ADHESION ENERGY OF DIAMOND-LIKE CARBON FILM WITH ENERGY METHOD. Myoung-Woon Moon, Kyu Hwan Oh, School of MS&E, Seoul National Univ., Seoul, KOREA; Jin-Won Chung, Kwang-Ryeol Lee, Thin Film Technology Research Center, Korea Institute of Science and Technology, Seoul, KOREA.

This study presented a new evaluation method of fundamental adhesion energy and elastic modulus of Diamond like Carbon (DLC) film under highly compressive stress state which caused the elastic instability, buckling. The analysis was executed by experiment and theory for DLC film deposited by CH4. To estimate these two elastic properties without other devices or external loads, the two kinds of buckling on the same specimens could be established. The one was the delamination buckling which was self-buckled phenomenon, and the other was the freehang buckling which could be developed with artificial technique of etching the substrate out at the side of delaminated film suggested by K.-R. Lee et al. With analysis of buckling, simple equation for adhesion energy and elastic modulus were taken into account independently with energy method which energy balance could be equivalent between before and after buckling. The adhesion energy at the interface between film and substrate needed to be considered in analysis of delamination buckling, but not to be considered in analysis of freehang buckling because of no interface adhesion which was removed by substrate etched. The freehang buckling could be developed by artificial technique which was the anisotropic etching Si substrate only out. And we could make the edge of DLC overhang free from constaint of Si substrate. The compressive strain resided in deposit process could be released as the form of the sinusoidal buckled wave. And this wave could be experimentally observed as well-developed wavelength and amplitude in etched film and be known as dependent on deposition conditions that affected the elastic properties of film. From experimental data and analytic equation for freehang buckling, the elastic modulus could be easily calculated and were evaluated from 70GPa to 280GPa for DLC films in stress changes from 0.6GPa to 2.1GPa. The adhesion energy could be evaluated by the analysis of buckled geometries of delamination buckling, and in this analysis the calculated elastic modulus of DLC film in the analysis of freehang buckling could be used due to same specimen. Then the fundamental adhesion energy of DLC film on Si substrate could be evaluated such as the range from 0.39 N/m to 1.17 N/m with increasing stress of film from 0.6GPa to 2.1GPa proportionally. In summary, the freehang buckling could be developed at the side of delamination buckled film by substrate etched. By freehang buckling the elastic modulus could be estimated, and the fundamental adhesion energy of thin film could be calculated without no other device by combining with the analysis of delamination buckling and the calculated elastic modulus.

W5.10
HYDROGEN INDUCED MULTI-VACANCY FORMATION IN HYDROGEN EMBRITTLEMENT OF α-Fe. Yoshitaka Tateyama, Takahisa Ohno, Natl. Res. Inst. for Metals, Tsukuba, JAPAN.

Hydrogen effects on void formation at the initial stage of fracture and during the crack propagation has been still one of the most important problems on hydrogen embrittlement of iron-based structural materials. Several effects related with lattice cohesion or dislocation have been already proposed, whereas they are not conclusive yet. Recently, some studies indicating the role of vacancy-hydrogen complexes have been also reported. In this work, we focus on this vacancy-hydrogen system in pure α-Fe and present hydrogen effects on the multi-vacancy formation investigated by first-principles supercell simulations. Concerning the stability of monovacancy, we found that monovacancy is most stabilized by trapping two H atoms inside at ambient condition. This complex '$VacH_2$' leads to about 30% reduction of the monovacancy formation energy due to the hybridization between the H $1s$ and the Fe $3d$ dangling orbitals. On the other hand, the $VacH_6$, which has been believed to be the most stable, is found to be energetically unpreferable to the monovacancy without H. These results provide a new insight for the vacancy clusterization. If the $VacH_6$ is the most stable, their clusterization is expected to be unfavorable due to the decrease of H sites in the vacancy cluster and the occupation of interstitial sites with higher energy by surplus H. On the other hand, this situation does not occur in the case of $VacH_2$, suggesting that the multi-vacancy formation is favorable. The present study clarifies that hydrogen can largely affect the void formation through the vacancy formation and clusterization. By comparing the lattice decohesion mechanism with the present results and considering relation between vacancies and dislocation motions, we discuss possible mechanisms for the hydrogen embrittlement of α-Fe.

W5.11
PLASTIC DEFORMATION AT MICRON-SCALE IN Al AND Cu THIN FILMS STUDIED BY MICROBEAM X-RAY DIFFRACTION (μXRD). Nobumichi Tamura[1], R. Spolenak[2], B.C. Valek[3], A.A. MacDowell[1], R.S. Celestre[1], H.A. Padmore[1], M.D. Morris[2], W.L. Brown[2], J.C. Bravman[3], T. Marieb[4], B.W. Batterman[1] and J.R. Patel[1,5]. [1]ALS/LBNL, Berkeley, CA. [2]Bell Laboratories, Lucent Technologies, Murray Hill, NJ. [3]Dept. MS&E, Stanford University, Stanford, CA. [4]Intel Corporation, Portland, OR. [5] SSRL/SLAC, Stanford University, Stanford, CA.

Plasticity in thin films has been extensively studied by macroscopic averaging techniques such as wafer curvature, tensile testing, bulge testing and conventional x-ray diffraction, but is still only partially understood. In the present work, we have investigated the plastic phenomena by using Microbeam X-Ray Diffraction (μXRD) combining monochromatic and polychromatic beam capabilities. This technique is able to resolve the full strain/stress tensor (6 components) and texture (orientation matrix) at submicron scale in thin films with an accuracy of 2 10^{-4} in strain and less than 0.1° in orientation. In contrast to the complementary technique of CBED (Convergent Beam Electron Diffraction) in Transmission Electron Microscopy, no sample preparation is necessary (i. e. the strain/stress state remains unaltered). Local plastic deformation of single grains in sputtered Cu blanket films (0.3 - 1.5μm thick) was studied between room temperature and 225°C. Furthermore we measured effects of two-dimensional confinement of passivated Al lines (0.7μm wide) and passivated and unpassivated electroplated Cu damascene lines (0.8 to 5μm wide) at temperature varying from room temperature to 300°C. The results indicate noticeable distortional strain and texture variations within individual single grains.

W5.12
DETERMINATION OF STRESS ORIENTATION DISTRIBUTION FUNCTION IN POLYCRYSTALLINE MATERIALS WITH NEUTRON DIFFRACTION. Yandong Wang and Xun-Li Wang, Spallation Neutron Source Project, Oak Ridge National Laboratory, Oak Ridge, TN.

The stress or strain incompatibility between grains having different crystallographic orientations can lead to grain orientation-dependent stress heterogeneity, generally known as the intergranular or type II stress. The appearance of this intergranular stress has severely compromised our ability to specify actual stress levels in the engineering component under examination. It is now generally understood that this incompatibility is mainly caused by the anisotropy of elastic or plastic behaviours in single crystallites. Therefore, determination of the intergranular stress will not only allow us to deduce the macroscopic (or engineering) stress in a component with much more confidence, but also shed light on the fundamental mechanism of grain-to-grain interactions during elastic and plastic deformations. Recently, a new concept called stress orientation distribution function (SODF) was introduced to describe the intergranular stress in polycrystalline materials. Similar to the crystal orientation distribution function (CODF), which is used to quantitatively describe crystallographic texture, SODF describes the mean field stress as a function of the grain orientation, or stress texture. In contrast to previous works that rely on numerical modelling of a specific thermomechanical process, SODF provides an experimental means to characterise the intergranular stress heterogeneity. Stated differently, with SODF, even for materials where the prior deformation history is unknown, the intergranular stress heterogeneity can still be described in a quantitative manner. In this paper, recent progress in determining SODF from neutron diffraction measurement results is presented.

W5.13
INTERFACIAL STRENGTH AND FRACTURE AT Au/Al_2O_3 INTERFACE. S.X. Mao, Department of Mechanical Engineering, University of Pittsburgh, Pittsburgh, PA.

Interfacial strength and fracture at Au/Al_2O_3 interface in layered materials are investigated for mixed mode I and mode II loading. Fracture testing has been carried out to measure the interfacial strength and toughness using $Al_2O_3/Au/Al_2O_3$ sample. The nanoscaled interfacial strength of Au/Al_2O_3 at crack tip has been found to be 14 Gpa and 3 Gpa in dry and moist air environments by measuring the critical crack tip blunting distance using atomic force microscope. Modelling on the interfacial strength and fracture toughness has been carried out. The dislocation plasticity in the metal layer is considered, and the energy for initial emission of a dislocation is assumed to be attained before the cleavage of the interface crack. Superdislocation modeling is employed to obtain the critical strain energy release rate, for interface seperation from crack tip. When a stress separation law based on interfacial strength is satisfied at the interface crack tip, interface debonding occurs and the crack propagates. This model predicts the strong dependence of interfacial fracture toughness on the interfacial strength under mixture of normal and shear loading.

SESSION W6/Q8: JOINT SESSION
LIMITS OF STRENGTH IN INDENTATION
Chairs: Murray S. Daw and Joost J. Vlassak
Thursday Morning, November 30, 2000
Room 309 (Hynes)

8:30 AM *W6.1/Q8.1
EXPERIMENTS ON THEORETICAL STRENGTH AND SIZE EFFECTS IN NANOINDENTATION. William D. Nix, Ranjana Saha, Erica T. Lilleodden, David Barbero and Bruce M. Clemens, Department of MS&E, Stanford University, Stanford, CA.

Nanoindentation permits the study of plasticity of materials in very small volumes, from the atomic and molecular scale, through the mesoscopic scale to the continuum scale. As such it provides a particularly good tool for validating the predictions of multiscale modeling and simulation of material behavior. Various plasticity experiments at small length scales will be described. Here we consider Individual Dislocation Effects, involving the nucleation of dislocations in perfect crystals and Multiple Dislocation Effects, as revealed by various indentation size effects. Nanoindentation of Mo and Ta epitaxial films and Au single crystals at the nanometer depth scale reveals irregular load-displacement curves that appear to be associated with the nucleation of dislocations. The contact pressures at which the first inelastic events are triggered compare favorably with recent calculations of nanometer scale indentations in perfect crystals. We show that these discrete plastic events are strongly affected by the proximity of high angle grain boundaries, suggesting that grain boundaries can play a role in dislocation nucleation. We have shown that the indentation size effect on hardness of crystalline materials can be accurately modeled using the concept of geometrically necessary dislocations and that this can be used to formulate a law for strain gradient plasticity. Here we describe a new type of nanoindentation experiment to show the effect strain gradients on flow strength. A strong plastic strain gradient is created by indenting a soft metal film on a hard substrate with a sharp diamond indenter. The hardness of the film is observed to increase with increasing depth of indentation, in sharp contrast to the falling hardness with increasing depth in bulk materials. We associate this rise in hardness with the strong gradient of plastic strain created between the indenter and the substrate and show that it can be calculated using a recently developed model of strain gradient plasticity.

9:00 AM W6.2/Q8.2
CONNECTING ATOMISTIC AND EXPERIMENTAL ESTIMATES OF IDEAL STRENGTH. C.R. Krenn[1,3], D. Roundy[2,3], Marvin L. Cohen[2,3], D.C. Chrzan[1,3] and J.W. Morris Jr.[1,3]. [1]Univ of California at Berkeley, Dept of Materials Science and Mineral Engineering. [2]Univ of California at Berkeley, Dept of Physics. [3]Lawrence Berkeley National Laboratory, Materials Sciences Division.

Using *ab initio* techniques, it is now possible to calculate the ideal shear strengths of perfect crystals with considerable accuracy. Using nanoindentation techniques, it is also possible to experimentally apply stresses of the order of the ideal shear strength to defect free regions of high purity single crystals. However, realistic determination of the stress fields produced during high stress nanoindentation requires finite element modeling. We use a finite element model incorporating a nonlinear stress-strain curve of the same form as that calculated *ab initio* for bcc tungsten to determine the maximum shear stresses reached beneath a stiff spherical indenter on a nonlinear elastic substrate. This model yields a load-displacement curve very similar to a Hertzian linear-elastic solution, but the peak shear stresses beneath the indentor are only 70% of those obtained from the Hertzian solution. We use these results to compare *ab initio* ideal strengths with the maximum shear stresses reached during nanoindentation of tungsten and molybdenum by Nix *et al.* and Gerberich *et al.* and find very good agreement. We conclude that the upper limit of strength during nanoindentation of initially defect-free tungsten and molybdenum is governed by the limits of elastic stability and suggest that other materials may behave similarly.

9:15 AM W6.3/Q8.3
THE ROLE OF MICROSTRUCTURAL LENGTH-SCALE IN INDENTATION BEHAVIOR OF GOLD. Erica T. Lilleodden, William D. Nix, Stanford University, Dept of MS&E, Stanford, CA.

Observations of depth dependent hardness have been made for various metals, and have been well described, in part, by strain gradient constitutive laws. However, strain-gradient models maintain a continuum framework and cannot be expected to explain discrete load-displacement behavior widely observed at the nanometer scale. Such observations of discontinuities in the initial stages of indentation imply that dislocation nucleation occurs, in agreement with atomistic calculations. However, the two descriptions, strain gradient analyses and dislocation nucleation considerations, rely on opposing limits of the relation between dislocation density and strength, imparting a critical discrepancy between these models. Here, we present experimental evidence of indentation size effects in hardness for gold thin films of various thicknesses. The observations are described in terms of dislocation nucleation and activation, and classical relations between dislocation distributions and strength. It is shown that the grain size affects both the critical loads for the onset of dislocation activity and the evolution of hardness with indentation depth. In particular, a Hall-Petch type strengthening mechanism is shown to play a substantial role in the indentation size effect of the small-grained films, overwhelming the strain gradient effects. Additionally, the competition between dislocation nucleation and activation of pre-existing dislocations is related to the grain structure and the proximity of the indentation to the grain boundary.

9:30 AM W6.4/Q8.4
PHYSICAL ORIGIN OF A SIZE EFFECT IN NANO-INDENTATION. A.J. Bushby[1], J.R. Downes[2], N.B. Jayaweera[2], P. Kidd[2], A. Kelly[3] and D.J. Dunstan[2]. [1]Department of Materials. [2]Department of Physics, Queen Mary and Westfield College, University of London, UNITED KINGDOM. [3]Dept. of Matls Sci & Metallurgy, Univ. of Cambridge, UNITED KINGDOM.

We have reported results of nanoindentation using spherical indenters to observe the full stress-strain curve. We observe the onset of plasticity in semiconductor strained-layer superlattices. These structures have alternating layers with strains of opposite sign. The yield pressure is reduced by the presence of the coherency strain. By varying the thicknesses and strains, we have been able to show that both sets of layers, compressive and tensile, reduce the yield pressure. This requires that a yield criterion must be satisfied over a finite volume, large enough to include layers of both sign. In these studies, we have observed a large and reproducible size effect in the yield pressure. That is, with smaller radius indenters the mean pressure acting over the contact area at the deviation from purely elastic behaviour increases, by up to a factor of two for a 2 micron radius indenter tip. Here we show how the requirement of meeting a yield criterion over a finite volume naturally leads to the size effect. Essentially, with small radius indenters, the peak stresses must be greater in order to achieve a given average stress over a finite volume. A theoretical analysis is given and quantitative agreement with experiment is obtained. This is a crucial result for the understanding of nanoindentation and other systems in which stresses are highly inhomogeneous on a small scale.

9:45 AM W6.5/Q8.5
IN-SITU NANOINDENTATION OF TRANSITION METAL CARBONITRIDES IN A TRANSMISSION ELECTRON MICROSCOPE. A.M. Minor, Department of MS&E, University of California, Berkeley, CA and Center for Advanced Materials, Lawrence Berkeley National Laboratory, Berkeley, CA; E.A. Stach, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; C.R. Krenn, J.W. Morris, Jr., Department of MS&E, University of California, Berkeley, CA and Center for Advanced Materials, Lawrence Berkeley National Laboratory, Berkeley, CA.

The mechanisms of nanomechanical deformation in ultrahard materials such as the transition metal carbonitrides are poorly understood. We have recently developed a nanoindentation TEM specimen holder which gives us the ability to make real-time observations into the nanomechanical response of various materials. We will show results from real-time nanoindentations of transition metal carbonitrides and the subsequent analysis of the resulting indentation damage. Our results will be compared to prior *ex-situ* indentations of ultrahard materials, and to ongoing theoretical calculations of the ideal strength of these materials. We will also discuss issues related to the experimental procedures, including the unique specimen geometry required for the *in-situ* nanoindentations and the effects of the thin foil on the indentation.

10:30 AM W6.6/Q8.6
MECHANICAL RESPONSE OF DIAMOND AT NANOMETER SCALES: DIAMOND POLISHING AND AFM. Rubén Pérez, Murray R. Jarvis and Michael C. Payne, Universidad Autonoma de Madrid, Departamento de Fisica Teorica de la Materia Condensada, Madrid, SPAIN; University of Cambridge, Theory of Condensed Matter, Cavendish Laboratory, Cambridge, UNITED KINGDOM.

Many technological processes and characterization techniques rely on the mechanical response of materials at the nanometer scale. Although computationally demanding, ab-initio methods can now be used to explore the limits of strength under these conditions. In this work, total energy pseudopotential methods are used to study two different processes involving the mechanical interaction of diamond

nanoasperities and diamond surfaces: the wear processes responsible for diamond polishing, and the mechanical deformation of tip and surface during the operation of the Atomic Force Microscope in contact mode (CM-AFM). The strong asymmetry in the rate of polishing between different directions on the diamond (110) surface is explained in terms of an atomistic mechanism for nano–groove formation. Although the direct ab-initio simulation of nanogrooving is still out of reach, the process can be studied in two steps. Separate simulations in which a rigid tip was incident from the soft and the hard polishing directions on a single nano–asperity show pronounced differences in the extent of the induced deformation. Then these differences in asperity removal are related to the process of nanogrooving by changing the boundary conditions at the edge of the asperity to recreate an ideal surface. The post–polishing surface morphology and the nature of the polishing residue predicted by this mechanism are consistent with experimental evidence. In the case of CM-AFM, it is shown that it is possible for a tip terminated in a single atom to sustain forces in excess of 30 nN. The magnitude of the normal force was unexpectedly found to be very similar for the approach on top of an atom or on a hollow position on the surface. This behaviour is due to tip relaxations induced by the interaction with the surface. These forces are also rather insensitive to the chemical nature of the tip apex.

10:45 AM W6.7/Q8.7
SINGLE CRYSTAL INDENTATION: OXIDE RUPTURE, SURFACE ASPERITIES AND THE YIELD POINT PROCESS. Donald E. Kramer, Natl Inst of Standards & Tech, Gaithersburg, MD; Karl B. Yoder and William W. Gerberich, Univ. of Minnesota, Dept. of Chemical Engr & Matls Sci, Minneapolis, MN.

Nanoindentation of metallic single crystals has been a topic of recent investigations. This is a result of their ability to withstand near theoretical contact stresses without showing signs of plastic deformation. When plasticity occurs, it manifests itself as a yield point, a sudden discontinuous increase in indenter displacement and decrease in contact pressure. It has been suggested that dislocation nucleation is the controlling mechanism for the time dependent and instantaneous injection of plasticity under these conditions, while the importance of an oxide or contamination layer has been relatively unexplored. This study combines atomic force microscopy (AFM) with nanoindentation to demonstrate the roles that oxide films and asperities play in the yield point process. Time dependent and instantaneous yield point properties were investigated for single crystals of tungsten and Fe 3%-Si. AFM observations indicate that the presence of asperities has a dramatic effect on the time dependent yield point properties. Measurements on the dependence of yield point load on oxide film thickness are used to develop a fracture mechanics based model in which oxide film fracture controls the yield point process. The results suggest that dislocation nucleation can occur prior to a yield point, but that egress of these dislocations is inhibited by the oxide film. Upon fracture of the oxide film, this constraint is lifted and elastic/plastic indentation ensues.

11:00 AM W6.8/Q8.8
EFFECT OF SURFACE STEPS ON DISLOCATION STRUCTURE DURING NANOINDENTATION. Jonathan A. Zimmerman, Patrick A. Klein, Stephen M. Foiles, Sandia National Labs, Livermore, CA.

The study of dislocation nucleation and plastic behavior during nanoindentation is a prime example in which nanoscopic details play an important role in the evolution of macroscopic mechanical behavior. Experimental studies of nanoindentation suggest that the presence of surface irregularities, such as steps, modify the mechanical response during indentation. However, the experiments did not reveal the details of dislocation creation or how the nucleation process is altered by the irregularities. Through quasi-static atomistic simulations using the embedded atom method, we examine the indentation of a Au(111) crystal that contains a surface step. These simulations show the effect the presence of the step has on both global quantities of indentation force and mean pressure as well as the local atomic stresses. A newly formulated atomistic deformation metric, the slip vector, is used to quantify initial dislocation content. Using this metric, we analyze the shear stresses resolved onto the directions of the partial dislocations that form, improving upon previous analyses which have used the maximum resolved shear stress of all possible slip directions. Our analysis leads to an estimate of critical resolved shear stress to be used as part of a nucleation criterion even at very close distances to atomic-level defects, such as a surface step. In addition, the pre-nucleation stress fields are compared with continuum calculations performed using Cauchy-Born elasticity. These results show that the Cauchy-Born constitutive model works well even at the large deformations close to the indenter. The use of this model in conjunction with a coupled atomistic-continuum approach would allow simulation of systems much closer in size to those studied in experiments.

11:15 AM W6.9/Q8.9
IDENTIFICATION OF PRESSURE-INDUCED PHASE TRANSFORMATIONS USING NANOINDENTATION. Vladislav Domnich, Univ of Illinois - Chicago, Dept of Mechanical Engineering, Chicago, IL; Yury Gogotsi, Drexel Univ, Dept of Materials Engineering, Philadelphia, PA.

Depth-sensing indentation has been successfully used for identification of pressure-induced phase transformations in several brittle materials. Phase transformations during nanoindentation may be revealed through deviations in the shape of load-displacement curves from that of a perfect elastoplastic material. A sudden volume change during fast transformation results in the discontinuity in the load-displacement curve ("pop-in" or "pop-out" events). Sluggish transformation is followed by a gradual change in the slope of the loading or unloading curve (an elbow), which may not always be readily identified if the indentation data are presented as the load-displacement curves. Based on the empirical power law relation between the applied load and the elastic part of the indenter displacement, the average contact pressure (Meyer's hardness) during indentation can be assessed as a function of the contact depth between the indenter and the specimen. Defined in this manner, the pressure - depth relation is linear unless the elastic modulus of the specimen changes in the process of indentation. This greatly facilitates monitoring of possible phase transformations under the indenter and allows assessing the corresponding transformation pressures. Phase changes after indentation are verified by Raman microspectroscopy. The technique is applied to the studies of several single crystal semiconductors and ceramics, including silicon, germanium, boron carbide and zirconia.

11:30 AM W6.10/Q8.10
MECHANICAL DEFORMATION OF CRYSTALLINE SILICON DURING NANOINDENTATION. Jodie Bradby, J.S. Williams and J. Wong-Leung, Australian National University, Department of Electronic Materials Engineering, RSPhySE, Canberra, AUSTRALIA; M.V. Swain, University of Sydney, Biomaterials Science Research Unit, Department of Mechanical and Mechatronics Engineering and Faculty of Dentistry, Eveleigh, NSW, AUSTRALIA; P. Munroe, University of New South Wales, Electron Microscope Unit, Sydney, NSW, AUSTRALIA.

Deformation during spherical and pointed indentation in (100) crystalline silicon using a UMIS-200 nanoindenter has been studied using cross-sectional transmission electron microscopy (XTEM), atomic force microscopy and Raman microspectroscopy. XTEM samples were prepared by focussed ion beam milling to accurately position the cross-section through the indentations. Indentation loads were chosen below and above the yield point for silicon to investigate the modes of plastic deformation. Slip planes (originating from the region of maximum shear stress) are visible in XTEM micrographs for all indentation loads studied but slip is not the main avenue for plastic deformation. A thin layer of poly-crystalline material has been identified (indexed as a high pressure phase from diffraction patterns) on the low load indentation, just prior to yield ('pop-in' during loading). For loading above the yield point, a large region of amorphous silicon was observed directly under the indenter when fast unloading conditions were used. The various microstructures and phases observed below indentations are correlated with load/unload data.

11:45 AM W6.11/Q8.11
AN ASSESSMENT OF THE MICROSTRUCTURES AND MECHANICAL STRENGTHS OF ALUMINIDE-BASED THIN COATINGS. S.Y. Li, H.P. Ng and Alfonso H.W. Ngan, Univ of Hong Kong, Dept of Mechanical Engineering, Hong Kong, PR CHINA.

Titanium and nickel aluminide-based thin coatings were synthesized by magnetron sputtering from intermetallic Ti-50at.%Al and Ni-25at.%Al alloy targets on various substrate materials. Both of the aluminide coatings exhibited high surface hardness values that varied with the degree of heat treatment. Structural characterizations using atomic force microscopy and transmission electron microscopy revealed a typical nanocrystalline structure in the coatings. The hardnesses of the coatings were investigated over a wide range of applied loads using micro- and nano- indentation techniques. It was found that the measured hardness of the coatings depends on the indentation depth, the film thickness as well as the strength of the substrates. In order to estimate the intrinsic strength of the films, the indentation size effects of the apparent hardness were analyzed in terms of the "absolute hardness" models by Jonsson and Hogmark (1984) and Ngan and Ng (submitted). The Jonsson-Hogmark model is more applicable to the situation of hard, brittle films on soft substrates, while the Ngan-Ng model is applicable to soft films on either soft or hard substrates. The analysis indicated that the strengths of the aluminide coatings considerably exceed their strengths in bulk. Plausible strengthening mechanisms are discussed.