SYMPOSIUM Q

Fundamentals of Nanoindentation and Nanotribology II

November 27 – 30, 2000

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*Invited paper
Finite element modeling has proven to be a valuable tool for interpreting nanoindentation measurements obtained from samples such as thin films or ion-implanted layers, allowing the mechanical properties of the films to be separated from those of the underlying substrates. For the case of Si, the measurement is greatly complicated by pressure-induced phase changes and cracking. We have performed extensive indentation testing of both crystalline and self-ion-implanted, amorphous Si, on both bulk Si and Si-on-Insulator substrates. The residual indents were examined with atomic force microscopy (AFM) and both secondary (SE) and secondary electron microscopy (STEM). Detailed finite element simulations of the indentations were performed with the usual 2-dimensional nanometer approximation and with a full 3-dimensional description. By modeling the material as isotropic, elastic-plastic solids with a pure yield criterion, the amorphous Si is shown to have a yield strength and elastic modulus about 20% lower than crystalline Si. Furthermore, comparisons of the extent of cracking and deformation indicate that amorphous Si is more ductile and that it may not undergo phase changes during indentation. This talk will present the results of our measurements and calculations, including the those of ongoing examinations of the effects of phase changes using microscopy and detailed 3-dimensional finite element simulations. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, supported by the U.S. Department of Energy under contract DE-AC04-94AL85000. This work was supported through their Office of Basic Energy Sciences.

9:15 AM Q1.3
A NEW TECHNIQUE FOR CALCULATING THE HARDNESS AND MODULUS FROM INSTRUMENTED INDENTATION DATA.
Warren C. Oliver, MTS Systems Corporation, Nano Instruments Innovation Center, Oak Ridge, TN.

The most generally accepted method for calculating the hardness and modulus using instrumented indentation data uses three empirically determined parameters and a geometric description of the indenter. The three parameters used are the load on the indenter, the displacement of the indenter past the point of contact, and the slope of the unloading curve. The depth is first corrected for the elastic deflection of the surface, using the unloading slope, and then substituted into the geometric description of the indenter to yield the contact area. This contact area, together with the load, yields the hardness. Used together with the unloading slope, the modulus is obtained. This technique requires that the geometric description of the indenter be quite accurate. A new technique that uses the slope of the loading curve instead of the depth of indentation will be presented. This technique significantly reduces the sensitivity of the calculated hardness and modulus values to the geometric description of the indenter, particularly at shallow depths.

9:45 AM Q1.5
LOAD-DISPLACEMENT BEHAVIOR DURING SHARP INDENTATION OF ELASTOMERS AND ELASTOPLASTIC MATERIALS.
Robert F. Cook, Michelle Oyen-Tsimm, Yeete A. Toiwa, University of Minnesota, Dept of Chemical Engineering and Materials Science, Minneapolis, MN.

A constitutive equation is developed for geometrically-similar sharp indentation of a material capable of elastic, viscous, and plastic deformation. The equation is based on a series of elements consisting of a quadratic (reversible) spring, a quadratic (time-dependent, reversible) dashpot, and a quadratic (time-independent, irreversible) slider-essentially modifying a model for an elastic-perfectly plastic material by incorporating a creeping component. Load-displacement solutions to the constitutive equation are obtained for both load-controlled and displacement-controlled indentation during constant and kinematic or increment-rate, respectively. A characteristic of the former is the appearance of a forward-moving "nose" during unloading of load-controlled systems (e.g., magnetic-coated "nanoindenter" systems). Even in the absence of this nose, and the associated initial negative unloading tangents, load-displacement measurements (and hence inferred modulus and hardness values) are significantly perturbed on the addition of the viscous component. A trivial extension of a conventional indentation procedure allows modulus, hardness, and viscosity to be decoupled from a three-step load-unload-indent contact sequence.

10:30 AM Q1.6
MOLECULAR-DYNAMICS SIMULATIONS OF NANOINDENTATION. Huei-Ming Yu, Arizona State University, Science and Engineering of Materials Program, Tempe, AZ; James Adams, Arizona State University, Dept of Chemical, Bio, and Materials Engineering, Tempe, AZ; Yang-Tse Cheng, General Motors Research and Development, Warren, MI; Louis Herson, Microscience Division, ALCOA Technical Center, ALCOA Center, PA.

A series of molecular dynamic simulations have been performed in order to study the indentation of a hard pyramid tip into an Al (100) surface. The effects of several variables are investigated, including temperature, tip-substrate bonding and indentation force. The indentation loading and unloading curves are generated during simulation and we compare it with experimental results. Also, we discuss the relationship between hardness and the indentation depth of Aluminum.

10:45 AM Q1.7
MOLECULAR-DYNAMICS SIMULATIONS OF NANOINDENTATION OF SILICON NITRIDE - PLASTIC DEFORMATION AND MECHANICAL PROPERTIES. Phillip Walsh, Rajiv J. Kalia, Aishah Naqina and Prayas Vishal, Concurrent Computing Laboratory for Materials Simulation, Louisiana State University, Baton Rouge, LA; Subhash Saini, Numerical Aerospace Simulation Facility, NASA Ames Research Center, Moffett Field, CA.

10 million atom molecular dynamics simulations of crystalline and amorphous silicon nitride are performed using various indentation geometries. Mechanical properties such as hardness and elastic moduli are determined and compared with experiments. Local stress profiles are determined using dynamical cut-and-project techniques. Images of atomic configurations, and local bond angle distributions are used to investigate mechanisms of plastic deformation in silicon nitride during nanoindentation. Local mechanical properties are observed in the region of the tip. Hardness of silicon nitride estimated from the simulations is within reasonable agreement with experiment.

SESSION Q2: SCANNING PROBE METHODS
Chair: Nancy A. Burnham
Tuesday Morning, November 28, 2000
Room 309 (Hynes)

11:00 AM *Q2.1
AN ATOMIC FORCE MICROSCOPY PERSPECTIVE ON MEASURING NANOMECHANICAL PROPERTIES. Richard Colton, Naval Research Laboratory, Surface Chemistry Branch, Washington, DC.

In 1896, Binnig, Quate and Gerber introduced the atomic force microscope (AFM) as a complementary tool to the scanning tunneling microscope (STM) capable of imaging the surface of insulators with high spatial resolution. Shortly after the invention of the AFM, two research groups reported other important applications of AFM in nanotechnology and nanomechanics. In 1987, the IBM Almaden group, Binnig, Geissler, Ehrensaft and Chang-reported lateral force measurement showing "atomic" stick-slip phenomena on the surface of graphite. In 1988, the NLG group of Khoury, Burnham and Colton-reported force-distance curves recording attractive, repulsive and adhesive interactions as a function of tip-surface separation. In time both of these AFM-based techniques have matured into quantitative probes of tribological processes, mechanical properties and adhesion at the nanometer scale. This paper will review some of the highlights and pivotal results leading to the development of these quantitative techniques. With respect to development of AFM-based nanomechanical, two important techniques, namely, depth-sensing nanoindentation and force mapping, have been combined to form a new hybrid instrument capable of measuring mechanical properties-stiffness, modulus, hardness, damping losses and creep-of thin films and compliant materials.

Work done under ONR sponsorship and in collaboration with N. Burnham, S. Hues, C. Draper, D. Shaffer, S. Corcoran, K. Wall, and S. Saed Assif.

11:30 AM Q2.2

The atomic force microscope in the lateral force modulation (LFM) mode has been used to characterize the surface modulus. The surface modulus is directly correlated with the surface crosslink density. Brominated poly(2,4-dimethyl-1,3-pentanediyne) (BIM) is a synthetic terpolymer of isobutylene (IB), para-methlyacetamide (PAM), and para-bromomethylstyrene (BPMS). The mechanical properties can be controlled by varying the PMS content and the molecular weight, while the BPMS content is varied to introduce chemical functionalities and chemical crosslinking. BIM can be chemically crosslinked by N,N-Dicarbamylidene-1,6-Hexanedimine at 120°C. Using LFM, we studied the crosslink dynamics of different types of BIMs elastomers of different PMS and BPMS content. LFM appears to be more sensitive to compare different crosslinking techniques such as friction force modulation. The results show the rate of the crosslinking reaction at the surface is sensitively affected by the bromine content of the BIMS, the amount of crosslinker, and the reaction time. The result will be compared with measurements of the bulk modulus on the same samples.

11:45 AM Q2.3

Thermomechanical writing occurs as Joule-heated, cantilevered tips imprint nanometer-scale indentations (bits) in a 50nm-thick polymer (PMMA) film. Thermal data reading incorporates the same cantilevered probes detect a temperature change when a tip follows the contour of a previously written bit. Binnig et al. [1] demonstrated single-cantilever writing and reading density at 400 Gbit/in$^2$. A micromachined 32x32 cantilever array has been fabricated [2] and has demonstrated parallel read/write operation at 150 Gbit/in$^2$ [3]. While much progress has been made to develop a thermomechanical data storage device [4], the fundamental process of thermomechanical bit formation is not well understood. Furthermore, microscopic polymer rheological parameters are unlikely to apply as the bit size approaches the polymer molecule radius of gyration. We have performed detailed investigations of the thermomechanical storage processes for various cantilever designs by applying atomic force microscope (AFM) based force detection during thermal operation. We examine the thermomechanics of polymer indentation with respect to bit size, bit density, and writing speed. Design tradeoffs of data reading signal-to-noise with operational bandwidth and power consumption are discussed and compared with a finite-difference model thermal and electrical simulation [5]. Also, we investigate issues of bit lifetime and wear-induced signal degradation through studies of the rheology of a confined viscoelastic medium. This work impacts the design of AFM cantilevers for combined thermal writing and reading as well as the understanding of fundamental polymer mesoscopic transport.


381
2:30 PM Q3.4
ON THE MEASUREMENT OF STRESS-STRAIN CURVES BY SPHERICAL INDENTATION
Erik G. Herbert, MTI Systems Corporation, Oak Ridge, TN; George M. Pharr, University of Tennessee, Knoxville, TN, and Oak Ridge National Laboratory, Oak Ridge, TN; Warren C. Oliver, MTI Systems Corporation, Oak Ridge, TN; Barry N. Lucas, Fast Forward Devices, LLC, Knoxville, TN.

It has been proposed that with the appropriate models, Instrumented Indentation Test (IIT) data can be used to predict the stress-strain behavior of the test material. However, very little work has been done to directly compare the results from uniaxial tension and spherical indentation experiments. In this work, uniaxial tension, compression, and indentation experiments have been performed on the aluminum alloy 6061-T6. The purpose of these experiments was to specifically explore the accuracy with which the analytical models can be applied to IIT data to predict the uniaxial stress-strain behavior of the metal.

2:45 PM Q3.5
PREPARATION OF TUNGSTEN TIPS FOR NONINDENTATION AND COMPARISON WITH DIAMOND ON SOFT MATERIALS
Jaime C. Graulau, Lorraine F. Francis, William W. Gerberich, Univ of Minnesota, Dept of Chemical Engineering and Materials Science, Minneapolis, MN.

Tungsten tips have been prepared for nonindentation using an electrochemical technique originally developed for field-ion microscopy. Tips with radii of curvature from 0.3 - 3 μm were prepared using 0.38 mm diameter tungsten wire and various applied voltages, generally 1000 V, solution concentration.

The performance of tungsten tips prepared in this manner has been compared to that of commercially produced diamond tips with equivalent radii when indenting bulk polymers. Models of both glass (T<sub>c</sub> = 200°C) and rubber (T<sub>c</sub> < 20°C) polymers, measured using tungsten tips, are comparable but slightly lower than those obtained using an equivalent size diamond tip. Furthermore, comparison of pull-off forces between tip and sample suggest that the greater adhesion between diamond and polymer relative to tungsten-polymer adhesion, may be a contributing factor to mechanical property differences when using a spherical tip approximation. Pull-off forces are twice as great in the diamond-polycarbonate case, on the order of 10% of the polycarbonate yield stress.

When these same tungsten tips are used to indent higher modulus materials, such as single crystal aluminum, large compliances and tip deformation are observed. The calculated result yields Young's moduli that are 50 - 70% of the expected 70 GPa that is obtained when diamond is used.

3:30 PM Q3.6
DETERMINATION OF THE REAL INDENTER SHAPE FOR NONINDENTATION/NANOTRIBOLOGY TESTS BY SURFACE METROLOGICAL AND ANALYTICAL INVESTIGATIONS
Susan Enders, Howard Haworth, NRC Innovation Centre, Vancouver, CANADA; Peter Grau, M-Luther Univ, Dept of Physics, Halle, GERMANY.

In contact experiments under high local loading knowledge of the shape and the expansion of the developing stress field is vitally important for the exact determination and proper interpretation of the mechanical properties of the material under study. To deduce the properties from the stress field it is thus necessary to know the actual shape of the indenter used. We make the assumption that as more tests are made, the indenter will not be evenly abraded and this inhibres the indenter selfsimilarity needed for analysis. This happens not only during scratching experiments but also for the indentation test when uneven wearout can be recorded. In this paper a critical comparison of different kinds of surface metrological and profilometrical methods is made, as well as analytical investigations, to qualify the real indenter shape function. The surface mapping of indenter shapes was done by atomic force microscopy [AFM], scanning electron microscopy [SEM] and optical interferometric profilometry on actual indenters as well as on their indentations in soft materials. The different surface profiling methods are compared to each other. Precise estimation of the deformation inherent in each method. To compare analytical calculations for determining two functions of indenters, their validity was surveyed on results of indentation tests made on standard calibration materials such as fused silica, glass and isotropic single crystals. As an example, calculations based on both Hertz and Sneddon theories of pure elastic contact are thoroughly discussed. Together with various transition functions, thickness theories then yield the complete stress function. The results obtained show the advantages and disadvantages of the respective theories by describing a rounded indenter tip. These investigations should help to avoid inaccurate material property evaluations due to incorrect determination of the real indenter shape.
3:45 PM Q3.7
3D FIB MAPPING OF NANOINDENTATION DEFORMATION ZONES. Timothy J. Milam, Karen W. Jolicoeur, Dept. of Materials, University of Oxford, Oxford, UNITED KINGDOM; Oliver Kraft, Thomas Wagner, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

A novel technique has been developed to examine site-specific, subsurface microstructures in three dimensions (3D). A 3D data set is collected by successive cross-sectional slicing using a gallium focussed ion beam (FIB) and imaging using secondary electrons, enabling a 3D microstructure to be generated using computer-based reconstruction techniques. In the first instance, this 3D FIB imaging technique has been applied to copper-based optical metal multilayer coatings which have been deformed by nanoindentation. Profiles of the deformed subsurface interfaces have been generated in 3D. These individual interface maps allow analysis of the deformation in terms of both the thickness of individual layers and that of the entire film. Material flow, where planarization is pilcetric to the size of the indent, can thus be precisely characterised. The site at which the sectioning is to be carried out can be chosen with high spatial resolution; consequently, nanoscale mechanical properties can be linked directly with an area’s microstructure. In the case of multilayer systems, particular indents were analysed and their deformation characteristics related to the specific hardness and moduli values which were measured during indentation. Since this 3D FIB mapping approach is not limited to the study of interfaces in coatings or deformation caused by nanoindentation, and could be applied to any material with subsurface structure of interest, it provides a unique and exciting insight into site-specific, subsurface microstructure.

4:00 PM Q3.8
DEVELOPMENT OF A NANOINDENTER FOR IN SITU TRANSMISSION ELECTRON MICROSCOPY. E. A. Schne, D. K. Owen, T. P. Freeman, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; M. A. Wall, Materials Science Division, Lawrence Livermore National Laboratory, Livermore, CA; A. M. Malic, Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA; T. Chrunel and R. Hull, Department of Materials Science and Engineering, University of Virginia, Charlottesville, VA.

Nanoindentation has become one of the primary testing techniques used to determine the mechanical properties of thin films and small material volumes. In this technique, a sharp indenter of known geometry is forced into a material surface along a controlled orientation, and the displacement of the material is measured as a function of time. Using this load displacement measurement one can determine the value of certain mechanical properties as well as infer the microstructural response. We have constructed a nanoindenter that resides within a transmission electron microscope sample holder. This permits real time TEM observation of the microstructural response of a material during nanoindentation. We will present results that include fracture of brittle materials (silicon), the indentation response of amorphous germanium, and dislocation motion and yielding in aluminum. The design of the piezoelectrically driven indentation holder will be discussed in detail. Additionally, we will present results on a heated indenter tip and a heated sample with displacement measurement using a visible light laser interferometer.

4:15 PM Q3.9
EXPERIMENTAL CONTACT MECHANICS STUDIES IN THREE DIMENSIONS. Barry N. Luecke, Jack C. Huy, Fast Forward Devices, LLC, Knoxville, TN; and Warren C. Oliver, MTS Systems Corporation, Oak Ridge, TN.

Mechanical and tribological properties at the nanometer-scale are of vital importance to the performance of a number of U.S. industries. This is especially true by the broad range of applications where an adherent, hard, thin, wear-resistant coating plays an essential role in performance of a product or device. In an effort to continue the advancement of techniques required for mechanical characterization of surfaces, a new testing system has been developed that allows independent and dynamic control of the forces on, and the displacements of, a point probe in three dimensions. This capability not only allows studies of dynamic frictional properties during the initial stages of contact between the probe and surface, but also the investigation of the threedimensional elastic response of the surface as the direction of the contact vector is systematically varied. The initial observations and results from this new system when applied to a number of surfaces will be presented and discussed.

4:30 PM Q3.10
LOAD-DISPLACEMENT BEHAVIOR DURING MACROINDENTATION. Yette A. Tavola, Matthew L. Cunningham, Dylan J. Morris, Robert F. Cook, University of Minnesota, Department of Chemical Engineering and Materials Science, Minneapolis, MN.

Load and displacement measurement during indentation provides a means of estimating material modulus and hardness values without direct observation of the indentation contact impression. Such measurements are thus extremely useful for evaluating material properties on extremely small scales at which it is difficult or impossible to observe the contact impression or in which the measurement is constrained to small volumes (e.g., in thin films). Hence, “nanoindentation” techniques have been developed, focusing on deconvoluting load-displacement indentation traces to obtain underlying material properties. In this talk, the design and usage of a macroindenter is described. The instrument allows load and displacement to be measured during indentation events at length and load scales (up to 100 N) at which instrumentation and probe geometry calibration difficulties systemic to nanoindentation are avoided. In addition, the instrument provides the capability for direct observation of contact area and fracture and delamination events to be tracked during simultaneous load displacement measurement. Load-displacement results for a variety of microelectronic materials and film-on-substrate systems are presented and interpreted in terms of conventional modulus-hardness deconvolution procedures. It is suggested that macroscopic measurements, in which there are few instrumentation difficulties, might provide a better vehicle for assessing indentation contact models.

4:45 PM Q3.11
SUB-MICRONINDENTATION AT VERY HIGH TEMPERATURES. Thomas Bell, Alexander Bendell, Leszek Wielinski, Anthony Fischer-Cripps, CSIRO Telecommunications and Industrial Physics, Sydney Australia; Mark Hoffman, University of NSW, Australia.

A critical aspect of understanding wear behaviour of materials is the determination of the mechanical properties of the materials at temperatures corresponding to those resulting from friction and/or inservice environmental conditions. Sub-microindentation testing can be used to determine mechanical properties of materials at the same microstructural level at which wear mechanisms operate. A high-temperature indentation testing instrument, capable of measuring indentation force and displacement at high resolution on samples in situ up to temperatures of 1100°C, has been developed. The device is also suitable for analysing a wide range of mechanical properties including those of the various hard coatings and electronic components at elevated temperatures. The present work gives details of the instrument and preliminary results obtained on a series of metal-ceramics composites.

SESSION Q4 DEFORMATION AND DEFORMATION MECHANISMS

Chair: Donald E. Kramer and Erik A. Schne Wednesday Morning, November 29, 2000
Room 309 (Hyenas)

8:30 AM Q4.1
CHARACTERISING THE PROPERTIES OF VERY THIN (≤100 nm) COATINGS BY LOW LOAD NANOINDENTATION TECHNIQUES: THE BENEFITS OF USING BOTH SHARP AND BLUNT INDENTERS T.E.E. Perl, S.J. Bull, Materials Division, University of Newcastle, Newcastle upon Tyne, UNITED KINGDOM; Department of Chemical Engineering & Materials Science, University of Minnesota, Minneapolis, MN.

We are exploring the use of very low load contiguously-relocating indentation techniques (e.g., ≤10 mN with a Nanotip 2 and ≤1 mN with a Hysitron Park AFM) to measure the properties of thin coatings (≤100 nm thick) on both glass and silicon. This is achieved using a number of monolith and multi-layer CVD and sol-gel materials on glass and magnetron-sputtered & -beam evaporated carbon nitride on silicon. Each sample (~50 mm tip-end radius) and blunter (~200 mm tip-end radius) indentors have been used. Indentation with sharper tips produces well-behaved load-displacement curves from which all the expected quantitative data regarding plastic and elastic work, hardness and modulus values (and their variation with indentor...
Discontinuities during both load and depth controlled continuous indentation tests have been attributed to dislocation nucleation or multiplication during film fracture. In some high dislocation densities, such as annealed single crystals of iron, tungsten, and GaAs, it is very difficult to determine which event is controlling the excision. In most of these cases, the excision has been attributed to dislocation nucleation and multiplication, and any subsequent film fracture is merely a result of the large strains which occur on the surface of the samples during the excision. However, in materials which exhibit permanent deformation prior to a discontinuity in loading, it is more likely that the phenomena is indeed controlled by film fracture, and not the rapid generation of dislocations. The current study has been undertaken to examine the properties of passivating films on engineering alloys as well as model systems such as iron and aluminum. An electrochemical cell coupled with a scanning probe microscope and nanoindentation system allows growth of passive films on a mica tensile stainless steel as well as a titanium alloy. The occurrence of excisions is shown in these materials to be linked directly with film fracture, rather than dislocation multiplication. A complement of set of ex situ experiments shows the presence of deformation prior to film fracture with both load and depth sensing techniques as well as imaging the surface topography.

9:15 AM 9.4.3
INDENTATION OF THIN FILM COATINGS ON SUBSTRATES.
P. D. Beaman and Analysis, M. Do, K. Van Vliet, S. Suresh, Dept. of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA.

The analysis of the mechanics of indentation in thin metal films is complicated by the effect of the underlying substrate on the elasto-plastic deformation of the film. In this paper, we present a combined experimental and computational approach for coated film/substrate systems to address the following fundamental issues: 1) the range of film thicknesses for different film/substrate combinations for which continuum elastoplastic models adequately describe the indentation response; 2) the geometric conditions and material properties for which the substrate strongly influences the deformation of the film, and 3) the effect of residual stress in the film on indentation response. Thin films of aluminum on brittle substrates are subjected to deep-sensing nano-, micro-, and macro-indentation. The ensuing indentation responses are quantitatively assessed by comparisons with the predictions of two-dimensional as well as full three-dimensional finite element results. The validity and limits of continuum mechanics-based models (including physically based crystal plasticity models) and the conditions governing the onset of discrete deformation modes are discussed.

9:30 AM 9.4.4
ARGON IRRADIATION AND ANNEALING TEMPERATURE EFFECTS ON POP IN EVENTS DURING NONINDENTATION OF POLYCRYSTALLINE IRON. Carlos M. Loepensji, Andrea A. Tauros, Neda R. Karamoto, Dept. of Nuclear Engineering, Brazil; Carlos E. Feoerster, Francisco C. Serbena, Dept. of Physics, Univ. Est. de Ponta Grossa, Brazil.

The indentation of metallic materials shows considerable complexity. At small depths, nonindentation loading curves in single crystals shows pop in events which are commonly indicated as yield point loads. We investigated the effect of annealing temperatures and argon irradiation on the occurrence of pop in polycrystalline iron (99.99%) during indentation. Samples of iron were mechanically polished with abrasive processes finishing using diamond powder with diameter of 0.25 μm. After polishing the samples were annealed at 700°C by 20 min, and then the temperature was decreased at a low rate to room temperature. After annealing pop in events were observed at almost all loading curves. Annealed samples were then mechanically polished again with alumina powder (1 μm diameter) and diamond powder (0.25 μm). Pop in events were not observed in nonindentation after second polishing process. Other annealed samples were irradiated with argon ions at 24 keV. In events are still present in these samples of irradiation. The mechanically polished samples were then submitted to annealing at temperatures from 300°C to 700°C, at intervals of 100°C and indentation tests were performed after each annealing. The pop in event frequency increases with annealing temperature. These results are discussed in terms of defects generated by mechanical polishing and argon ion irradiation.

9:45 AM 9.4.5
DETERMINATION OF MATERIAL PROPERTIES OF POLYMERIC MATERIALS THROUGH NONINDENTATION TESTS AND FINITE ELEMENT MODELING. Byung Ho Kim, Timothy O. Ocvirk, The Pennsylvania State University, Dept. of Mechanical and Nuclear Engineering, University Park, PA.

Nonindentation techniques are utilized extensively to characterize a wide variety of thin coatings. In this paper, we present a four-parameter asymmetric visco-elastic/plastic finite element model, a simplified model of five parameter model proposed earlier, has been suggested to explain the behavior of polymer coatings subject to nonindentation tests utilizing a micron sized spherical indenter. The four parameters in modified Kelvin-Voigt model account for both visco-elastic and plastic response in the coating, and are determined by a process that matches the experimental load versus indentation depth plot from the test with that from the finite element model. The model not only provides more useful information than an ordinary method using unloading curve or sinusoidal load but also has an advantage of less iteration time when compared to five-parameter model.

10:30 AM *9.4.6
NANO-MECHANICS OF PLASTIC DEFORMATION DURING INDENTATION. Sahar Saeidi, Massachusetts Institute of Technology, Cambridge, MA.

This presentation will deal with recent experimental and theoretical studies of the nanomechanics and micromechanisms of elasto-plastic deformation in metals subjected to indentation. Particular attention will be devoted to the identification of the transitions from continuous deformation processes to discrete deformation mechanisms as the indentation loads are reduced from macro- to nano-indentation. Experimental studies of indentation will be supplemented with transmission and scanning electron microscopy and atomic force microscopy in an attempt to probe the nanomechanisms of indentation. The effects of grain boundaries, crystallographic texture, surface roughness, initial defect density, and defect nucleation and mobility on the nano-indentation response of a variety of metallic materials will be considered. Experimental and quantitative models will be presented to rationalize the experimentally observed nanomechanistic processes.

11:00 AM 9.4.7
INDENTATION OF AN ALUMINUM THIN FILM ON A GLASS SUBSTRATE: EXPERIMENTAL AND MECHANISM-BASED STRAIN GRADIENT PLASTICITY STUDIES. R. Saha, Stanford Univ, Dept. of Materials Science and Engineering, Palo Alto, CA; Z. Xu, Univ. of Illinois, Dept. of Mechanical Engineering, Urbana, IL; W. D. Nix, Stanford Univ, Dept. of Materials Science and Engineering, Palo Alto, CA; Y. Huang, Univ. of Illinois, Dept of Mechanical Engineering, Urbana, IL.

Micro-indentation hardness test is a reliable experimental method to determine the mechanical properties at the microscale. We present an experimental study of micro-indentation hardness test of an aluminum thin film on a glass substrate. The experiments clearly display very strong size effects for small depths of indentation as well as for large depth of indentation (close to film thickness). We have used the theory of mechanism-based strain gradient (MBSG) plasticity to study micro-indentation experiments in the A1 film/ glass substrate system. It is establised that the MBSG plasticity theory captures the observed size effect very well, while classical plasticity theories clearly fail short for small and large depths of indentation.

11:15 AM 9.4.8
YIELDING AND THE INDENTATION SIZE EFFECT IN IRIDIUM. J. G. Swindener, E.P. George and G. St Pieter, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN; University of Tennessee, Dept. of Materials Science and Engineering.

Iridium alloys have several properties that make them excellent model materials in indentation studies. They have high resistance to oxidation, a large elastic modulus (560 GPa), and a relatively low yield strength (1.0 MPa for 0.2% offset strain). From nano-indentation experiments with a 69 micron radius spherical indentor, the yield
strength of In 0.3% tensile strain was determined to be 2.9 GPa. This value is 18 times the yield strength of the bulk material, but still much lower than the theoretical value (225 GPa) of the material. The large yield stress for the 69 micron spherical indenter appears to be due to the indentation size effect. The effect will be further assessed by using spherical indenters with larger radii.

Nanoindentation experiments with a Berkovich indenter show a large indentation size effect, which extends to much greater depths in indium than in most materials. For a Berkovich indenter, hardness values range from 3.1 GPa at a contact depth of 0.2 microns to 5.9 GPa at a contact depth of 0.37 microns. The indentation size effect appears to extend through the microhardness regime and possibly into the microhardness regime.

Researchers at the Oak Ridge National Laboratory used the NanoMOE User Facility sponsored by the division of Materials Science and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

11:30 AM Q4.9
NANOINDENTATION OF SINGLE AND POLYCRYSTALLINE FCC METALS: Andrew Goldstein, Krystian Van Vleet, Subrah Suresh, Massachusetts Institute of Technology, Dept. of MSE, Cambridge, MA.

Nanoindentation experiments were performed on a variety of single crystal FCC metals, in both thin film and bulk form and of different crystallographic orientation and vastly differing elastic properties. In addition, nanoindentation was conducted on polycrystalline FCC metal thin films with different grain sizes and known crystallographic texture. Deformation under the nanoindenter was studied by recourse to continuous depth-sensing indentation where the load was recorded as a function of depth of penetration. The elastic and plastic portion of deformation under the indenter, along with the grains of discrete and continuous plastic deformation, were documented for all these experiments. The role of dislocation and motion during indentation in facilitating plastic deformation bursts was analyzed by invoking an energetic model. In addition, in bulk model, the model was constructed and tested as a visual tool in interpreting the nucleation and motion of dislocations during nanoindentation. The effects of film thickness, grain size, texture, and the presence of pre-existing defects in the films on the indentation response were analyzed. The effect of these variables on the onset of discontinuous plastic deformation was also examined.

11:45 AM Q4.9
PRODUCING THE CRITICAL STRESS INTENSITY FACTOR FOR SLIP TRANSFER OF GRAIN BOUNDARIES BY NANO-INDENTATION WITH AN AFM: Alfonso H.W. Ngan, The University of Hong Kong, Department of Mechanical Engineering, Hong Kong, PR CHINA.

Based on the concept of dislocation pile-up, a plasticity model has been developed to predict the variation of hardness with grain size and indent size for sub-granular indentation. The predicted relation has a 1.25 slope, which is a similar value to the 1.25 slope found by the work of Sharples et al. (1991) and the additional log-log/log size term representing the relative importance of the pile-up zone to the total deformation volume. A further parameter assumed in the model and appearing in the predicted relation is the critical stress intensity factor for slip transmission across the grain boundary. This parameter is analogous to the Hall-Petch slope in uniaxial testing of polycrystalline specimens, but unlike the latter which is a global average, it is specific to the grain boundary of a single grain.

Two types of experiments have been used to check the validity of this relation. The first type of experiments is microhardness measurement on bicrystals and coarse-grained polycrystals. In these experiments, the distance of the indent from the grain boundary is taken as the ‘grain size’, and plotting the hardness variation according to the model predicted by the present model shows good agreement with the model. The second type of experiments is micro- or nano-indentation on fine-grained polycrystals. Here, the grain size is fixed but the indentation size varies. These data also show qualitative agreement with the predicted relation.

In carrying out the nanoindentation experiments on fine-grained polycrystals, we have used a Hysitron® nanoindenter mounted on a Triboscope® scanning probe microscope. In such a set-up, the sample is scanned by the indenter and the load is applied to the sample. This technique is vital in the current exercise as it enables one to locate precisely the area to be indented, namely, the centre of a selected grain.
Microscopy. Elastic modulus of 110 to 130 GPa and hardness of 1 to 1.6 GPa were measured using the continuous stiffness option (CSM) of the Nanoincident XP. Thicker films appeared to be softer in terms of the lower modulus and hardness, exhibiting a classical Hertz-Patch relationship between the yield stress and grain size. Lower elastic modulus of thicker films is due to the higher porosity and partially due to the surface roughness. Comparison between the mechanical properties of films on the substrates obtained by nanoincident and tensile tests of the freestanding Cu films is made.


High resolution x-ray microbeam measurements have been used to investigate the 3-dimensional deformation microstructure near nanoindentations in single-crystal Cu. Broad-back-scattered (white) synchrotron x-ray microbeams of ~0.7 x 0.7 μm2 cross-section were produced by a Kirkpatrick-Baez mirror on the MBAT-CAT beamline at the Advanced Photon Source. These beams have been used in connection with a CCD detector, interactive laser diffraction software, and a newly developed technique for obtaining microresolution along the penetration depth (~25 μm) to probe lattice rotations in a function of depth and position under Berkovich nanoindentations in <111> oriented Cu. Nearly one-dimensional tilt of up to ~3 degrees were found below and extending beyond the flat faces of the indenter, with a much less-complicated distribution of tilts with compound lattice rotations observed below the tip and the sharp blades of the indenter. The x-ray microbeam methods for performing the measurements and data analysis will be discussed, and the results of the detailed investigations of the fundamental aspects of materials deformation by combining nanoincident techniques with 3-D x-ray microbeam measurements will be considered.

Research sponsored by the Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Department of Energy under contract DE-AC05-00OR22725; the operation of the APS is sponsored by the DOE.

2:30 PM Q5.5 MECHANICAL PROPERTIES OF AS-GROWN AND ION-BEAM-MODIFIED GaN FILMS. S.O. Kucheyev, J.E. Bradly, J.S. Williams, C. Jagdiah, The Australian National Univ., Dept. of Electronic Materials Engineering, Research School of Physical Sciences and Engineering, Canberra, AUSTRALIA; M. Toth, M.R. Phillips, University of Technology, Sydney, Microstructural Analysis Unit, AUSTRALIA; M.V. Swain, The University of Sydney, Department of Mechanical and Mechatronic Engineering, AUSTRALIA.

The deformation behavior of wurtzite GaN films grown on sapphire substrates is studied by nanoincident with spherical indenters. Atomic force microscopy (AFM) and nanoindentation techniques are used to characterize the deformation mode. Slip is identified as one of the physical mechanisms responsible for the "pop-in" events observed during loading of as-grown crystalline GaN indentation with a ~0.2 μm radius indenter. A maximum load up to 900 mN does not produce any cracking visible by AFM in as-grown GaN. Instead, under such loads, indentation results in a pronounced elevation of the material around the impression. Implantation disorder significantly changes the deformation behavior of GaN. In particular, we discuss the mechanical properties of (i) GaN films amorphized by ion bombardment and (ii) GaN films with a high concentration of implantation-produced point defects.

SESSION Q6: ADHESION

Wednesday, November 29, 2000 Room 309 (Hynes)

3:15 PM Q6.1 QUANTIFICATION OF POST-CMP COPPER ADHESION TO A MORPHOUS Si PASSIVATION VIA NANOINDENTATION. J.B. Vella, A.A. Volinsky, I.S. Adibeh, Motorola, DigitalDNA Laboratories, MSL, Mesa, AZ; S.M. Smith, Motorola Labs., P8H5, Tempe, AZ.

Nanoindentation has been used to quantify the practical work of adhesion of oxidized post-CMP copper surface to Si passivation. Poor adhesion of electroplated copper to Si passivation is observed following CMP due to copper oxide growth prior to plasma enhanced silicon nitride deposition. Four point bend testing has shown that failure of test structures occur at the Cu/CuO interface.

Hydrogen and ammonia plasma treatments of the post-CMP copper surface are employed to not only remove the oxide but to increase the surface roughness of copper. Copper oxide reduction is shown by auger electron spectroscopy. Surface roughening of copper is shown by atomic force microscopy. Both effects are shown to increase the adhesion strength of the Cu/Si interface by quantifying the practical work of adhesion and nanoindentation with a one-micron conical indenter was used to induce Si film delaminations and correlate them to the practical work of adhesion [1]. In order to more reliably and repeatably produce the delaminations a TiW (10%) Ti superlayer was sputter-deposited on to the test structures [2]. Mechanical properties, including elastic modulus and hardness of Si, electroplated copper, and TiW were measured by nanoincident. D.B. Abraham, A. Demny, Appl. Phys. Lett., 56(10), 2628-2631, 1984. 2. M.D. Krise, W.W. Gerberich, N.R. Moody, J. Mater. Res., 14(7), 3007-3018, 1998. 3. A.A. Volinsky, N.I. Tymisk, M.D. Krise, W.W. Gerberich and J.W. Hutchinson, Mater. Res. Soc. Proc., 539, pp. 277-290, 1999.

3:30 PM Q6.2 DETECTION OF DISCRETE BONDS UPON RUPTURE OF MICROCONTACTS TO SELF-ASSEMBLED MONOLAYERS. Hjalti Skulason, C. Daniel Frisbie, University of Minnesota, Department of Chemical Engineering and Materials Science, Minneapolis, MN.

Pull-off forces were measured under load for Au coated atomic force microscopy (AFM) tips in contact with self-assembled monolayers (SAMs) bearing S-containing end groups known to bond to Au. In these experiments, the tip-SAM microcontacts involve approximately 100 molecules. The mean pull-off force required to break the Au-SAM microcontacts was seven times greater than with centered SAMs having no S-containing groups. Further, rupture force histograms for the Au/S-containing SAM microcontacts showed 0.1 nN periodicity. We have assigned this 0.1 nN force quantum to rupture of individual chemical bonds and have estimated the bond energy to be on the order of 10 kJ/mol. The specific interaction corresponding to this energy appears to be abstraction of Au atoms from the tip surface upon pull-off. Force quantums were also observed in other microcontact experiments involving chemically modified tips and SAMs. Our ability to detect force quantums is a function of the solvent. In order to observe single bond rupture forces directly, the tip-substrate interfacial energy must be negative and larger in absolute value than the substrate-solvent and tip-solvent interfacial energies. Otherwise, non-specific solvent exclusion effects dominate the microcontact adhesion. These measurements demonstrate that pull-off forces can be sensitive to fluctuations in the number of discrete chemical interactions.

3:45 PM Q6.3 ADHESIVE FAILRE OF THIN EPOXY FILMS ON ALUMINIZED SUBSTRATES. N.R. Moody, Sandia National Laboratories, Livermore, CA; D.T. Biddle, Washington State University, Pullman, WA; M.S. Kent, J.A. Emerson, E.D. Reed, Jr., Sandia National Laboratories, Albuquerque, NM.

Composition and structure are two of the most important factors controlling performance and reliability of thin film components. They are particularly important in components with thin polymer films where changes in composition and structure during processing and service can lead to interfacial failure. However, our understanding of interfacial failure in these systems is limited by the lack of established thin polymer film test techniques. We have therefore begun a program to determine the properties and adhesion of Epon 838/T400 using nanoincident and stressed overlayers. The films were spin coated onto a aluminumized substrate to four thicknesses ranging from 24 nm to 11.8 μm. The indentation tests showed that the near surface properties of all four films were essentially the same. In contrast, susceptibility to fracture appeared to vary with film thickness where the thickest film delaminated readily during indentation while the thinner films required deposition of highly stressed overlayers to trigger delamination. Nevertheless, fracture in all samples occurred along the film-substrate interface. Mechanical-based models are then used to determine interfacial fracture energies. In this presentation, the test and analysis techniques will be discussed and used to show that practical works of adhesion can be obtained for the very thin polymer films used in this study. This work supported by U.S. DOE Contract DE-AC04-94AR45000.

4:00 PM Q6.4 EPOXY/ALUMINUM ADHESION AS MEASURED BY CONTACT MECHANICS (JKR) IN THE PRESENCE OF AN ORGANIC CONTAMINANT. John A. Emerson, Dana L. Woerdeman", Rachel K. Guinn, Sandia National Laboratories, Albuquerque, NM; Virginia Commonwealth University, Richmond, VA.

Bonding of microgarnries gives rise to a variety of technical barriers, including dispensing, alignment, and facturing. In these minute length scales, knowledge of interfacial properties such as...
roughness and surface energies of the various components is critical. Surface contamination is a common occurrence, and therefore calls for a convenient method to remove the contamination before introducing adhesive to the microsystem. Photoelectro surface analytical techniques are routinely used for characterizing substrates, however they are impractical because the contaminants are evaporated in high vacuum. The use of this contact mechanics technique has a number of advantages for probing micro-scale areas in a processing environment, since it is relatively inexpensive, versatile, and easy to use. In a theoretical study by Bremond Wyatt and de Gennes, they considered the dewetting phenomena of water between a hydrophilic solid and a rubber. In the present work, we investigate the behavior of a model organic contaminant, hexadecane, in contact with aminophenolamine PM. We use hexadecane because it resembles a typical machining fluid, is nonreactive with aluminum surfaces, and should not dissolve readily into the adhesive systems of interest. Preliminary results have shown that the hexadecane does not wet the aluminum oxide surface, but instead, forms a discontinuous film. One particular interest is understanding why we observe dewetting of a low surface tension fluid in contact with a relatively high energy surface. We also examine the effect of the contaminant on the adhesive forces between an epoxy cyanoacrylate (the probe) and anodized aluminum using the JK contact mechanics approach. The extent of hysteresis in the contact mechanics curves illustrates the sensitivity of the technique to minute quantities of hexadecane on the surface. This work supported by U.S. DOE Contract DE-A101-84AI15500.

Q7.1 THE EFFECTS OF CREEP ON ELASTIC MODULUS MEASUREMENT USING NANOINDENTATION. Gang Feng, Alfonso H.W. Ngan, The University of Hong Kong, Department of Mechanical Engineering, Hong Kong, PR, CHINA.

In nanoindentation the moduli of materials are usually calculated using the Oliver-Pharr scheme in which it is assumed that the unloading process is purely elastic. However, time dependent deformation (TDD) can happen alongside elastic recovery, and consequently, the modulus calculated may be over or underestimated. One form of TDD is thermal drift and this can be corrected for by subtracting from the observed displacement rate a thermal drift rate, which is measured in a load held period. Another form of TDD is creep. In this paper we show that creep always results in overestimation of the modulus. We also present evidence showing that the effects of creep cannot be eliminated simply by using very long holding time, since even though steady state creep may be achieved, the creep rate is still larger than zero (e.g. about 0.05nm/s for Cu (111) plane under 6 mN). A method is proposed to correct for the effect of TDD by extrapolating the TDD law in the holding process to the unloading one if the TDD is assumed to exhibit either time-hardening or strain-hardening constitutive characteristics. Thermal drift is correctable by this method as it trivially obeys time-hardening constitutive laws. Under this assumption, the error introduced by TDD in the contact compliance is shown to be equal to the displacement rate (creep plus thermal drift) at the end of the holding period divided by the unloading rate during the unloading process that follows. Thus, the methods errors may be introduced if a short holding period or a slow unloading rate is used. This correction method is applied to compute the elastic moduli of three materials including single crystal copper, single crystal Ni3Al and polycrystalline Al, and the results are compared with the values obtained without considering the TDD effects.

Q7.2 NANOINDENTATION AND NANOSCRAVING OF SILICON CARBIDE ALLOYED PYROLYTIC CARBON. Martin A. Wiedenmeier, Sulzer Carbonides Inc., Austin, TX; Steven G. Thomas, George M. Pharr, Univ of Tennessee, Dept of Materials Science and Engineering, Knoxville, TN and Oak Ridge National Laboratory, Oak Ridge, TN.

Pyrolytic carbon (PyC) alloyed with silicon carbide is the principle material used in the manufacture of mechanical heart valves. PyC is made by cracking hydrocarbon and silane gases in a fluidized bed reactor at relatively low temperatures (~1350°C). The mechanical behavior of films of PyC alloyed with up to 14 wt% silicon deposited on graphite substrates was examined by nanoindentation and nanoscratching with a Berkovich indenter. Several unusual behaviors were observed, including fully elastic contact at all loads (up to 300mN) and a sharp reduction in scratch resistance at silicon concentrations above ~8 wt%. Results are presented and discussed in terms of pertinent microstructural observations.


Q7.3 NANOINDENTATION OF THIN FILMS: EXPERIMENTS AND ANALYSIS. Kajiwong Zeng, Lu Shen, Institute of Materials Research and Engineering, SINGAPORE.

This work is focused on the nanoindentation of thin film materials. Several different thickness of Al2O3 film on Al, Al film on graphite and Al film on Si were tested by nano-indentation techniques. The experiments covered two kinds of thin film structures, i.e., hard film on soft substrate, and soft film on hard substrate. It is generally believed that, for nano-indentation of thin films, the indentation depth should be less than 1/10 of the film thickness in order to avoid the effects of substrate to the measurement. This was examined through the current experiments. Our experiments showed that the maximum indentation depth, whereas no influence the substrate to nano-indentation, is in fact different from the cases of hard film on soft substrate to the soft film on hard substrate. This maximum depth is also controlled by the degree of differences between the film and substrate. The larger differences, the smaller the indentation depth. The results were also analyzed using the newly developed analysis method.

The mechanics of surface force driven contact growth between viscoelastic particles show the presence of three distinct stages. The first is the formation of a contact governed by the marginal balance of stored elastic and surface energies. The second stage is one of sintering driven by surface tension. Contact growth in sufficiently small particles is predicted to be dominated by an intermediate "zipping" mode driven by direct attraction across the gap ahead of the contact edge. The zipping mode of contact growth has been analyzed using results from the cohesive-zone theory of viscoelastic fracture and computational using surface finite elements. Unlike the first and third stages of contact growth, the zipping stage depends on the details of the cohesive force distribution, specifically the characteristic cohesive stress. It is predicted to dominate contact growth kinetics for submicrometer particles. The results from the simulation will be presented at this conference along with numerical results from this study, compared with existing theories and experimental data on the coalescence of polymeric particles.

Q7.5 MOLECULAR DYNAMICS SIMULATION OF ASPERITY SHEAR IN ALUMINUM. Jun Zheng, James B. Adams, Arizona State University, Dept. of Chemical and Materials Engineering, Tempe, AZ; Huaqiang Yu, Arizona State University, Science and Engineering of Materials Program, Tempe, AZ.

One important mechanism of wear involves the shear of asperities by other asperities. Here we use molecular dynamics to simulate the shearing of aluminum (57151) (Lennard-Jones) asperity. The simulations were repeated for a wide range of conditions, including different asperity drift velocities, temperatures, asperity shapes, degree of intersection, crystal orientations, and adhesive strength, to determine their effects on the wear process. These simulations involve the use of a reliable EAM potential for Al that was developed by Force Matching to a large database of DFT forces.

Q7.6 NANOINDENTATION EVALUATION OF BRITTLE FILMS ON HARD AND SOFT SUBSTRATES. Natasha Tymisk, Dept. of Chem. Eng. and Mat. Sci., University of Minnesota, Minneapolis, MN; Antranig Dankel, Hyspark Inc., Minneapolis, MN; Trevor F. Page, Materials Division, The University of Newcastle, Newcastle Upon Tyne, UNITED KINGDOM; William W. Gerberich, Dept. of Chem. Eng. and Mat. Sci., University of Minnesota, Minneapolis, MN.

In most cases, depth sensing indentation is the only practical means of evaluating the mechanical properties of thin films and coatings. While a number of experimental strategies and models for analysing data exist for general situations of this type, indentations into brittle porous films raise some added problems. Not least because deformation of such films involves a variety of concurrent processes such as plasticity, densification, and fracture. As this poses quite a challenge for the analytical solution, supplementary techniques such as acoustic emission become increasingly critical and useful. The present study addresses several cases of brittle films on different types of substrates. First, mechanical behavior of nanocrystalline carbon nanotube loaded substrates is considered. For the evaluation of indentation induced densification and intrinsic mechanical property deconvolution, repeated loading-unloading cycles have been utilized. This analysis was based on the P−d2 approach. With this method, indentation curves for elastoplastic materials obtained with sharp pyramidal indenters may be represented as P = Kd3 where P and d denote indentation load and displacement respectively. The parameter K includes materials hardness/modulus ratio and indent geometry. This approach was applied to both loading and unloading enabled evaluation of mechanical property gradients through the film thickness. The method also allowed detection of transient events corresponding to indentation induced fracture. In addition acoustic emission (AE) was attempted to provide separation of deformation and fracture induced events. Second, yield initiation phenomena have been evaluated for oxidized W single crystal surfaces under normal and sliding contact with the assistance of AE as a supplementary technique.

Q7.7 3D FIB MAPPING OF CRACK ZONES IN AL2O3−SiC NANOCOMPOSITES. R.W. Respass, W.G. Webster, W.B. Roberts, Beverley Inskan, Dept. of Materials, University of Oxford, Oxford, UNITED KINGDOM.

Al2O3−SiC nanocomposites exhibit dramatically improved strength and wear properties compared to monolithic Al2O3 ceramics with similar grain sizes. In particular, abrasive wear a transition from intergranular cracking in Al2O3 to intragranular cracking in Al2O3−SiC nanocomposites is observed. This change in fracture mode results in improved wear resistance in these nanocomposites (as compared to both monolithic Al2O3) and improved surface finish. In order to investigate the mechanisms causing the change in fracture mode and nanosurface damage behavior during wear, Al2O3−SiC nanocomposites and monolithic Al2O3 have been locally deformed by indentation. The subsurface damage beneath the indent sites has been analysed in 3D using a new 3D focused ion beam (FIB) mapping technique. This technique enables 3D maps of the crack zones to be extracted, directly under the indent where the mechanical properties have been measured. Using this method, the 3D nature of the crack zones in the Al2O3−SiC nanocomposites and monolithic Al2O3 may be directly compared.

Q7.8 STRESSES AND IMPACT TESTS FATIGUE OF PVD HARD COATINGS ON CEMENTED CARBIDES DEPENDENT ON COATING PARAMETERS. S. Tsapatsis, G. R. Dubois, Materials Science Institute, University of Technology Aachen, GERMANY; T. Leyendecker, G. Ersens, CemeCon GmbH, Aachen, GERMANY; A. Bouzakis, Laboratory for Mechanical Engineering, University of Thessaloniki, GREECE.

For quality control of PVD hard coatings applied on carbide machining or tooling tools the scratch test gives a first impression of the adhesion under constant or increasing loads. Coated carbide machining tools are often working under dynamic load, which has a great influence on the adhesion properties of the hard coatings [TiAlN and CrN-coated]. To get detailed information about the coatings characteristics and its dynamic behaviour the impact tester was developed and further improved. This test method is used for research and assessment of failure mechanisms of thin films and statements about the adherence of hard material coatings under dynamic compressive stress. Therefore a hard metal ball strikes with a frequency of up to 50 Hz onto the surface. The altitude stress can be varied to get a detailed evaluation of fatigue under reversal strain. The appearance of surface fatigue is based upon structural transformation, cracking and cracking growth processes and ends with the separation of debris particles caused by the above mentioned permanent changing strain. Selected hard material coatings were analyzed after testing with the described method. The results of the study as well as other studies were compared with the measured data of the stress distribution by nanoindentation. It will be shown, that the results of the fatigue test and the adhesion properties are dependant on deposition parameters. Different types of hard coatings were analyzed and compared.
to investigate materials with depth or scale-dependent mechanical properties. Matsubara and colleagues [3] have very recently shown that the same approach for the inverse problem to the standard equations currently used to analyze nanoindentation data. Results from finite element calculations showed excellent agreement with the analytical expression for the case of a material with a relatively large values of the elastic modulus and hardness. In the current paper, it is shown how this approach can be extended to polymers. Experimental data are presented for the indentation of PMMA sheet with Berkovich indenter. Each indentation was done using an exponentially increasing load in order to ensure that the imposed strain rate was constant as in penetration depth [4]. A simple manipulation of the equations also allows the ratio of the contact depth to the total penetration depth in terms of material and geometric parameters. It is shown how this ratio can be used to aid the interpretation of creep and recovery data for viscoelastic materials [1]. J.L. Loubet, J.M. Georges and G. Melle, in Mechanical Measurements in Materials Science and Engineering, ed. J.J. Bland and B.R. Law, ASTM, Philadelphia, p. 72 (1984) [2].


Q7.11 CORRELATION OF NANOINDENTATION AND CONVENTIONAL MECHANICAL PROPERTIES MEASUREMENTS. Philip M. Rice, IBM Research Division, Almaden Research Center, San Jose, CA, and Roger E. Stoller, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN.

A series of model ferritic and commercial alloys was used in an investigation of solute effects on irradiation-induced hardening. The model alloys were irradiated with both light and heavy ions and subjected to conventional Vickers hardness measurements to obtain measured hardness values. Measurements on commercial alloys were used to further extend this range. Nanohardness with loads as low as 0.05 g were obtained with the NanoIndenter-II and compared with conventional Vickers hardness measurements using a 200 g load. Two methods were used to obtain the nanohardness data for the comparison with Vickers hardness: (1) constant displacement depth and (2) constant load. When the nanoindentation data was corrected to account for the difference between projected and actual indenter contact area, good correlation between the Vickers and nanohardness measurements was obtained for hardness values between 0.7 and 3 GPa. The correlation based on constant nanohardness load was slightly better than that based on constant nanoindentation displacement. Tensile property measurements were also made on these same alloys, and the expected linear relationship between hardness and yield strength was found. Thus, a correlation was developed between measured changes in nanohardness and yield strength changes.

Q7.12 MECHANISM OF Al2Cu5Fe2 QUMCRYSTAL PLASTIC DEFORMATION STUDIED BY INSTRUMENTED SHARP INDENTATION. Sergey N. Dab, Institute for Superhard Materials, Kiev, UKRAINE; Yuli V. Milman, Dina V. Lsects, Anton N. Belous, Institute for Problems of Material Science, Kiev, UKRAINE.

Nanohardness tests of Al2Cu5Fe2 quasicrystal reveals a different mechanism of plastic deformation as compared with a regular metal. For a metal, the indenter tip is characterized by a load, while for a quasicrystal, it increases stepwise. Penetration of the indent into the metal occurs at the approximate constant average contact pressure. For quasicrystal the pressure under the Berkovich indenter is constant. It grows during several seconds and drop suddenly on about 300 MPa at 0.3 s. We think that the steps formation is not related to the indentation-induced cracks. Regular steps formation not observed for such brittle materials as sapphire, boron carbide and cubic boron nitride during loading up to 120 mN. Something close to these steps was observed earlier for germanium for which pressure-induced phase transformation in the indent takes place. Probably, the step formation in the load-displacement curve in Al2Cu5Fe2 quasicrystal is due to the structure transformation in the indent. It is known that the quasicrystal structure destroys at high deformation and transforms into a regular crystalline structure. Therefore, it is possible to attribute the pressure drop in the indent to the transformation of the quasicrystal structure into the crystalline one. In this case the plastic polycrystalline metallic phase is pressed between a diamond indenter and a rather hard quasicrystalline bulk. In result, the plastic phase is extruded out of the crystalline one. The total process decreases the volume of the phase and grows with the further loading until a new portion of the quasicrystal structure transforms into the crystalline one. The observation of thin layers extruded out of the indent in Al2Cu5Fe2 quasicrystals supports this assumption.

Q7.13 DETERMINING THE INTERPHONE SIZE AND PROPERTIES IN POLYMER-MATRIX COMPOSITES USING PHASE IMAGING ATOMIC FORCE MICROSCOPY AND NANOINDENTATION. Lukvan Kjærsgaard, Dept of Mechanical Engineering, William Cross, Testing Engineering, Ritupam Kumar, and Jan Koller, Dept of Materials and Metallurgical Engineering, South Dakota School of Mines and Technology, Rapid City, SD.

In polymer matrix composites the interface between the reinforcing phase and the bulk phase is paramount to the overall performance of the composite as a structural material. This interface is now thought to be a distinct, three-dimensional phase surrounding the reinforcing phase called the interphase. The development of the atomic force microscope and nanoindentation devices have facilitated the investigation of the interphase. Previously, force modulation AFM and nanoindentation were the primary methods used to determine the size of the interphase and its stiffness relative to the bulk phase. The present investigation utilized phase imaging AFM and nanoindentation to examine the interphase in a glass fiber-reinforced epoxy matrix composite. Nanoindentation experiments indicated that the relatively stiff fiber might have caused a gradient in the modulus across the interphase region. Specifically, the modulus next to the fiber approached that of the fiber and decreased to that of the bulk polymer as the distance away from the fiber increased. Once the fiber was removed by chemical etching this gradient reversed itself, hence, nanoindentation, due to the fiber bias, was not found to be adequate for measuring actual interphase properties. It was found that phase imaging AFM was a highly useful tool for probing the interphase, because it involves much lower interaction forces between the probe and the sample than force modulation or nanoindentation. The interphase in the model composite investigated was found to be softer than the bulk phase with a size of 2.4-2.9 microns, and was independent of fiber volume content, for similar treatments between 0.1% and 5.0% (initial aqueous concentration).


Johnson's cavity model relating indenter geometry and deformation resulting from elastic-plastic indentations is appropriate for a wide variety of materials. In the case of nanoindentations in single crystal BCC metals, limitations are reached when creep is not fully accounted for. Both the standard Berkovich and cube corner geometries show changes in the ratio of plastic zone radius to contact radius increases with the duration of time at the peak load. Indenter tip geometry is shown to play an important role in this phenomenon. Length scale phenomena, such as the indentation size effect, are also subject to various interpretations. The traditional definition of hardness does not produce similar trends with indentation length scale between the blunt Berkovich geometry and the sharper cube corner geometry. However, the ratio of the plastic zone radius to contact radius proves to be a tip geometry independent method of assessing the plasticity of these materials. These data are shown to hold for both low dislocation density materials (which exhibit a 'yield point') as well as large numbers of available dislocation sources in the region of the indent tip.

Q7.15 NANODENTATION USING TUNNELING PROBE WITH SEM CONDUCTING DIAMOND TIP. Novikov Nikolaus, Lynska Oleg, Grushko Vladimir, Institute for Superhard Materials, Kiev, UKRAINE.

The mechanical properties of thin films can be measured by a variety of different techniques of depth sensing nanoindentation. At the nanometer level, the actual contact area must be carefully determined to obtain reliable values of mechanical properties by indentation. At this scale, the contact area is greatly affected by local surface roughness. Therefore an indentation technique at the nanometer level does to be associated with imaging technique. Measuring the topography of an indent using SEM and combining this information with a load-displacement nanoindentation data is one of the most recent developments in this growing field. Scanning tunneling microscopes or atomic force microscopes are frequently used for imaging surfaces before and after indentation. Simultaneously these microscopes with diamond tip have been used for indentation (atomic force microscopes are preferred because of their versatility). The disadvantage of the tunneling pressure is the limitation in studying materials with low conductivity. Increasing the tunneling probe sensitivity to the tunneling current up to 0.01 nA can solve this problem. However in the case of traditional using a diamond tip for both indentation and scanning the obstacle of the low
conductivity of a diamond arises. For a solution of this problem semiconducting diamond monocrystals were synthesized. A unique feature of this material is the yields of the diamond films increasing with increasing the substrate temperature from 0.00001 mN to 10 mN. The experimental procedure includes three main stages. At the first stage scanning the surface with an indentor makes a pyramidal topographic image. At the second stage the indentation test is performed without the simple displacement. At the third stage the indentated area is scanned again with the indentor tip in order to visualize the indent. The same tip is used for and sliding with further scanning of the surface. The relation between the dimension parameters, the properties, and the surface morphology was studied.

Q.7.16
MECHANICAL PROPERTIES OF t-C FILMS PREPARED BY PULSED FILTERED VACUUM ARC DEPOSITION. W.F. Liu, S.P. Wong, N. Ke, W.Y. Cheung, Department of Electronic Engineering, The Chinese University of Hong Kong, Hong Kong, CHINA; N.Y. Wu, Institute of Nuclear Energy Physics, Beijing Normal University, Beijing, CHINA.

Hydrogen-free tetrahedral amorphous carbon (t-C) films were deposited on silicon substrates at various substrate bias voltages. Thermal annealing was performed in vacuum at various temperatures. The s3 fraction of the films was characterized by Raman spectroscopy. The Raman results confirmed that these t-C films exhibited high s3 fraction of over 80%. The hardness of the film was measured by the nanoindentation method. The friction and wear properties of the films were studied using a pin-on-disc tribometer. The stress in the films was studied by the curvature method and the stress-strain curves was studied by the infrared photelastic method. The optical properties of the t-C films were also studied using spectroscopic ellipsometry (SE). The complex refractive indices and the optical band gap of the t-C films were obtained by analyzing the SE spectra using the Foradi and Bloomer model. Results on the issues such as the variations of the properties of these films with the deposition conditions and the film thickness, the stress relaxation with annealing conditions, the correlation between mechanical properties and the s3 fraction, will be presented and discussed. This work is partially supported by the Research Grants Council of Hong Kong SAR (Ref. No.: CUHK 4155/97E).

Q.7.17
DETERMINING THE AREA FUNCTION OF SPHERICAL INDENTERS FOR NANOINDENTATION. Andrew J. Bashby1, Nigel M. Jennett2, Department of Materials, Queen Mary and Westfield College, University of London, UNITED KINGDOM; 2National Physical Laboratory, Materials Centre, Teddington, Middlesex, UNITED KINGDOM.

Nanoindentation with spherical tipped indenters provides a powerful technique for exploring surface mechanical properties through the application of Hertzian contact mechanics. The full range of mechanical response can be obtained from elastic, through the yield point, to permanent deformation. However, to be fully quantifiable and reproducible the technique requires accurate calibration of the indentor tip geometry. In nanoindentation diamond indenters tend to be chosen for their high hardness and elastic modulus. However, the nominally spherical diamond indenters deviate from an ideal spherical shape, due to the difficulty of polishing a monocrystalline crystal into a perfectly spherical geometry. Significant errors may be introduced by using a constant (especially the nominal) radius in calculations. Use of a continuous function describing radius (or area) as a function of depth is usually necessary. Calculation of the effective shape, or area function, for pointed indenters is often biased, for convenience, on indentation into reference materials. Methods using two reference materials have shown agreement with direct imaging using atomic force microscope (AFM) techniques. In this paper indentation methods are used to characterise a range of spherical tipped indenters with nominal radii from 2 to 30 microns. A traceably calibrated metrological AFM is also used to determine the actual shape of one of the indenters. Comparisons of the two methods are made and the sensitivity of both methods to measurement parameters are discussed.

Q.7.18
APPLICABILITY OF SNEEDON'S EQUATION TO THE ANALYSIS OF NANOINDENTATION DATA. L. Matthew, M. Tommasoni, Materials Science and Engineering Department, Rensselaer Polytechnic Institute, Troy, NY.

Sneddon derived equations that describe the elastic response of a flat specimen under a conical indentor. The Oliver and Pharr method of analysis of nanoindentation data applies these equations to the unload data and evaluates the projected area of contact to determine the indentation hardness. Hardness values obtained by this method are observed to be independent of the maximum load. However, hardness values determined by this method show an indentation size effect (ISE) where the hardness increases as the load decreases. Careful analysis of nanoindentation data obtained from different materials shows that Sneddon's equations do not apply over the entire range of indentation depths at which nanoindentation experiments were performed. Results show that the projected area of contact evaluated using this method at maximum load is larger than the cross-sectional area of the indentor for some depth of indentation, even when the non-dimensional area factor is used. This deviation in the projected area of contact at maximum load is a function of the indentation depth for a given material and is different for different materials. It has to be taken into account for accurate analysis of nanoindentation data.

Q.7.19
NANOINDENTATION RESPONSE OF CARBON NITRIDE THIN FILMS. G. Ciarletta, P. Patanos, S. Logothetidis Aristotle Univ, Dept of Physics, Thessaloniki, GREECE.

Nanoindentation test provides a simple, versatile and rapid means of measuring the scratch resistance of thin films with thickness below 1μm, information on the surface elastic-plastic deformation modes and their adherence to the substrate. We investigated the scratch response of thin (300 nm) Carbon Nitride (CNx) films, deposited on Si [001] by sputtering under intense ion irradiation, in the load range from 2 to 20 mN. The film mechanical properties was studied by nanoindentation and nano-arching. We have found a load dependent transition in the scratch and friction responses. Below 5 mN, the scratches showed a complex Abrahams-like behavior. Above 10 mN the scratches showed a mixed elastic-plastic behavior and at 20 mN exhibited areas with permanent grooves. However, Atomic Force Microscopy and in-situ profiling of the surface of the film before and after the scratch event observed no evidence of film failure. Coefficient of friction (μ) between 0.1 and 0.3) and plastic deformation increased with increasing load (L). The relation between μ and L is given by an expression of the form μL=α. The exponent b arises from the type (elastic-plastic) of film deformation after scratch-test. The film recovery after nanoindentation testing at 10 mN manifested variations in the film properties, which correspond to different stages in the deformation of the CNx film and hence determines transitions between different failure modes. The nanoindentation testing behavior of the CNx films was found to vary with the microstructure as it was studied by X-Ray Diffraction (XRD) and Reflectivity. XRD suggested that CNx is not homogeneous, but there are crystalline grains embedded in an amorphous matrix. The latter is supported by Continuous Stiffness Measurements revealing considerable variations of hardness and elastic modulus values (local values up to 45 GPa and 230 GPa, respectively) indicating that the crystalline regions are superhard and elastic.

Q.7.20

We have investigated the effect of the film thickness on the nanoindentation measurements of superhard diamondlike carbon films. The DLC films were deposited on Si [100] substrates by pulsed laser deposition (KrF, λ=248 nm, duration =25 ns, energy density about 3.0 J/cm2) in high vacuum (exceeding 10-7 torr) at room temperature for various periods of time. The nanohardness and elastic modulus of these films were measured by Nanoindentor 900P. It was found that the nanoindentation results are in agreement with the film thickness. In order to obtain more realistic mechanical properties of the DLC films, we used finite element analysis to analyze the nanoindentation process, Menas5.5 was used to simulate the nanoindentation process to obtain the calculated load-displacement curves for the DLC films and the simulated results were fitted to the experimental load-displacement curves. It was found that the combination of nanoindentation experiments with finite element modeling can give more information of the superhard DLC films than nanoindentation tests alone.

Q.7.21
NANOINDENTATION MEASURES OF POLYSTYRENE ADHESION. M. Adhison, M. Li, C. Berry Carter, William W. Gerberich, Univ of Minnesota, Dept of Chemical Engineering and Materials Science, Minneapolis, MN; Marc A. Hillmyer, Univ of Minnesota, Dept of Chemistry, Minneapolis, MN.

Indentation combined with atomic force microscopy (AFM) has been utilized for the evaluation of polystyrene (PS)/glass adhesion.

390
Adhesion energy calculations have been made using AFM measurements of the deformations induced by indentation. To enhance the resolution for deforming dielectrics (polymer-composite) (PMMA) overlayers were applied on top of PS films. The PS (Mw = 180K) film thickness 0.6 μm with a 1.2 μm PMMA overlay has a measured adhesion energy of ~0.65 J/m². Fracture surfaces were also characterized to determine the mechanism of interface fracture. In addition, using a pull-off method, the effect of PS molecular weight on the adhesion energy between tip and PS was studied. Above a certain indentation load, low molecular weight PS appears to have a higher adhesion energy.

Q7.22
NANOINDENTATION STUDIES OF LOW K DIELECTRIC MATERIALS. Ashok Kumar, M. Anthony, I. M. Erbas, Center for Microelectronics Research, University of South Florida, Tampa, FL.

Low-K dielectric films have reduced hardness and modulus values relative to traditional dielectrics and they are many potential challenges associate with these materials to integrate with IC technologies. In this investigation, we have used nanoindentation studies to evaluate the mechanical properties of doped oxides dielectrics (PSG, HSQ, MSQ, HGPS), organic dielectrics (BCB, SILK, FLARE, PAE-2), highly fluorinated dielectrics (parylene-AF4, a-CF, PTFE), porous dielectrics (xerogels, xerogels, nanogels). The structural properties of the films have been also investigated using analytical techniques.

Q7.23
THE EFFECT OF SURFACE FORCES ON APPARENT CONTACT COMPLIANCE AND THE IMPLICATIONS FOR FRAME COMPLIANCE DETERMINATION. Nigel M. Jennett, Andrew J. Bushby. 1 National Physical Laboratory, Material Centre, Teddington, Middlesex, UNITED KINGDOM 2 Department of Materials, Queen Mary and Westfield College, University of London, UNITED KINGDOM.

Depth sensing indentation (DSI) is one of the few techniques capable of probing both the elastic and plastic responses of very small volumes, such as thin layers. For elasticity, DSI measures the total contact compliance, which is a composite of the mechanical responses of the instrument frame, indenter and all components of the sample. Isolation of the elastic response of the target material requires a precise and accurate determination of all other compliance in the contact. The determination of frame compliance, Cf, of DSI instruments is therefore a crucial pre-requisite for obtaining reproducible, quantified measurements of indentation modulus. Determination of Cf by indentation into high compliance materials (e.g. BCB, widely used and can be repeatable. It is shown, however, that the CF value produced is a reference material dependent. Rearrangement of the contact mechanics equation has been suggested, but this introduces additional assumptions, such as hardness being invariant with depth. Often the same data will give different CF values depending on the analysis used. In this paper we present results from the European Commission funded project: "Determine and model the contact compliance of thin films by nanoindentation - INDICOAT". Indentation experiments into a wide range of reference materials have been performed using Berkovich and large radius spherical indenters. Reliable direct measurement of indenter area functions and surface modulus values for reference materials are used, to allow more accurate subtraction of the contribution of the sample from the total contact compliance. However, the CF values obtained in this way are not local and may be described by a function of the area of contact. Supplementary experiments are described which show that surface forces affect the measured contact compliance. The implication of this for the determination of CF are discussed.

Q7.24
NANODENTATION OF PRESSURE QUENCHED FULLERENES AND ZIRCONIUM METAL FROM A DIAMOND ANVIL CELL. Shane A. Cattell, Alexander T. Spencer, Jeremy R. Paterson, Yogesh K. Vohra. Dept. of Physics, University of Alabama at Birmingham, Birmingham, AL.

Due to the advent of diamond anvil cells, the physical properties of materials can be investigated under pressures of millions of atmospheres and temperatures of several thousand Kelvin. The sample size employed in these high pressure cells is limited to a diameter of typically 25 to 150 microns. While this size is often sufficient for studies using x-ray diffraction and Raman scattering, exact measurement of mechanical properties using conventional microhardness indentation techniques is not feasible. For some materials, the high pressure phase(s) can be quenched to room temperature allowing further characterization by other techniques. In this paper, we make use of the small probe volume available by nanoindentation to investigate the pressure-quenched structures of both C-70 fullerene and zirconium. For the case of C-70, we show that the amorphous phase established above 35 GPa can be quenched, and that it shows a largely elastic indentation loading behavior with a hardness of 18 GPa (compared to 7 GPa of the surrounding steel gasket). We establish that this hard carbon phase can be produced from C-70 fullerene by application of pressure at room temperature. Also of interest is the pressure quenching of zirconium metal from the unique omega-phase (hexagonal symmetry with AI3 structure). The use of pressure quenching in a diamond anvil cell along with subsequent nanoindentation of the small sample volume opens up new windows of opportunity for mechanical property measurements of materials after processing in high pressure/high temperature conditions.

Supported by National Science Foundation (NSF)

Q7.25
COMPUTER SIMULATION OF FRICTION OF POLYMER SURFACES. Dieter W. Heermann, Institut für Theoretische Physik, Universität Heidelberg, Heidelberg, GERMANY.

I propose a model to simulate friction of amorphous polymer surface. To model friction I use a tip-shaped tool that is dragged across the surface of a polymer film. In the contribution I focus on the region around the tip and exclude shear forces other than the internal. To be independent from the finite film thickness I introduce a coupling parameter which is a measure of the frictional force. After that I examine the dependence of friction on the load, the roughness of the tool. Finally I present the results of the structural modifications caused by the tool.

Q7.26
VIEWING A MOVING SURFACE CONTACT: A SCANNING PROBE MICROSCOPY AND QUARTZ CRYSTAL MICROBALANCE STUDY OF SLIDING FRICTION IN ABSORBED MOLECULES. B. Borovsky, T. Colley, M. Abdelsalam, and J. Krim, North Carolina State University, Department of Physics, Raleigh, NC.

Experimental investigations of friction, lubrication, and adhesion at nanometer length scales have traditionally been performed by employing scanning probe microscopy (SPM), surface forces apparatus (SFA), or quartz crystal microbalance (QCM) techniques. The QCM has been quantitative information on fundamental energy dissipation mechanisms associated with the sliding friction of atoms along surfaces, but its results have never been cross-referenced with SPM or SFA measurements. In order to perform such a cross-referencing, we have carried out two investigations: (1) a STM-QCM study of triarylphosphate (TCP) as well as a ternary blend of phenol phosphates (TPBP), two common molecular additives known lubricating properties on the macroscopic scale, and (2) a study, using several techniques, of the changes in interfacial friction of toluene on single crystal substrates in both the presence and absence of C60 adsorbed layers. In the first study, the results of previous macroscopic friction measurements [1] have been correlated with sliding friction measurements obtained by means of a joint STM-QCM apparatus [2]. In the second study, the results of previous SFA measurements [3] have been correlated with SPM and QCM macroscopic contact angle [4] measurements.

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. Nucleation of dislocations in Mo and Ta epilayers 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 inclasves effects 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 pre-existing dislocations and conclude that nanometer scale plasticity 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. We describe a new type of nanindentation experiment to show the effect strain gradients on flow strength. A strong plastic strain gradient is created by indenting a soft metal film on an 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 presence of dislocations and hardening at the film substrate interface. This can be calculated using a recently developed model of strain gradient plasticity.

9:00 AM Q8.2/W6.2

CONNECTING ACOUSTIC AND EXPERIMENTAL ESTIMATES OF IDEAL STRENGTH. C.R. Krenn1,2, D. Roundy2,3, Y.M. Cohen1,2, D.C. Chrzan1,2, and J.W. Morris Jr.1,2,3. 1Univ of California at Berkeley, Dept of Materials Science and Engineering. 2Univ of California at Berkeley, Dept of Astronomy. 3Lawrence Berkeley National Laboratory, Materials Sciences Division.

Using ab initio techniques, it is now possible to calculate the ideal strength of perfect crystals with considerable accuracy. Using nanindentation techniques, it is also possible to experimentally apply stresses of the order of the ideal strength to defect free regions of high purity single crystals. However, realistic determination of the stress field in a perfect high strength nanocrystalline material 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 spherical indenter as a function of the applied load. This model yields a load-displacement curve very similar to a Hertzian linear-elastic solution, but the peak stresses beneath the indenter are only 70% of those obtained from the Hertzian solution. We use these results to compare an ab initio ideal strength curve with the maximum shear stress reached during nanindentation 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 nanindentation of initially defect-free tungsten and molybdenum is governed by the limits of plastic stability and suggest that other materials may behave similarly.

9:15 AM Q8.3/W6.3

THE ROLE OF MICROSTRUCTURE LENGTH SCALE IN INDENTATION BEHAVIOR OF GOLD. Erica T. Lilleodden, William D. Nix, Stanford University, Dept of MS&E, Stanford, CA.

Observations of dependent hardness have been made for various metals, and have been well described, in part, by strain gradient constitutive laws. However, strain-gradient models must 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, implying 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 strain size affects the critical load 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 Au films, explaining 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 Q8.4/W6.4

PHYSICAL ORIGIN OF A SIZE EFFECT IN NANO-INDENTATION. J.J. Owusu, R. Dominguez, N.I. Skroban, E.A. Geim, and P. Kevser1, A. Kelly1, and D.J. Durran. 1Department of Materials, 2Department of Physics, Queen Mary and Westfield College, University of London, UNITED KINGDOM. 3Department of Materials Science and Metallurgy, University of Cambridge, UNITED KINGDOM.

We have reported results of nanindentation 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 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 understanding of nanindentation and other systems in which stresses are highly inhomogeneous on a small scale.

9:45 AM Q8.5/W6.5

IN-SITU NANOMATERIALS OBSERVATION OF TRANSITION METAL CARBONITRIDES IN A TRANSMISSION ELECTRON MICROSCOPE. A.M. Miner, Department of MS&E, University of California, Berkeley, CA and Lawrence Berkeley National Laboratory, Berkeley, CA; E.A. Stach, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; C.R. Krenn and J.W. Morris Jr. Department of MS&E, University of California, Berkeley, CA; and Lawrence Berkeley National Laboratory, Berkeley, CA.

The mechanisms of nanomechanical deformation in ultrathin materials such as the transition metal carbides are poorly understood. We have recently developed a nanindentation 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 nanindentations of transition metal carbides and the subsequent analysis of the resulting indentation damage. Our results will be compared to prior ex-situ indentations of ultrathin 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 nanindentations and the effects of the thin foil on the indentation.

10:30 AM Q8.6/W6.6

MECHANICAL RESPONSE OF DIAMOND AT NANO- AND MICRO-SCALE. M.J. Murray, R. Jami, and M. J. P. Pegg, 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 characterisation 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 nanoparticles 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 (C-AFM). The strong asymmetry in the rate of polishing between different directions on the diamond (110) surface is explained in terms of an anisotropy 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 is pushed into the solid and the hard polishing directions on a single nano-aperture 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 contact size, phase 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 CMAFM, 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 behavior 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 Q8.7/W6.7
SINGLE CRYSTAL INDENTATION: OXIDE RUPTURE, SURFACE ASPIRITIES AND THE YIELD POINT PROCESS. Donald E. Kramer, National Institute of Standards and Technology, Gaithersburg, MD; Karl B. Yoder and William W. Gerberich, University of Minnesota, Dept. of Chemical Engineering and Materials Science, Minneapolis, MN.

Nanoindentation of metallic single crystals has been a topic of recent investigations as 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 this displacement 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 aspirities play in the yield point process. Time dependent and instantaneous yield point properties were investigated for single crystal tungsten samples by AFM observation and nanoindentation testing. The results suggest that displacement nucleation can occur prior to a yield point, but often 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 Q8/8/W6.8
EFFECT OF SURFACE STEPS ON DISLOCATION STRUCTURE DURING NANOINDENTATION. Jonathan A. Zimmerman, Patrick A. Klein, Stephen M. Fales, Sandia National Laboratories, Livermore, CA.

The study of dislocation nucleation and plastic behavior during nanoindentation is a prime example in which nanostructural details play an important role in the evolution of macroscopic mechanical behavior. Experimental studies of the 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 atomic simulation using 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 response. The simulation revealed a 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 a nucleation criterion even at very close distances to atomic-level defects, such as a surface step. In addition, the precoupling stress field 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 indent. The use of this model in conjunction with embedded atom method should allow simulation of systems much closer in size to those studied in experiments.

11:15 AM Q8.9/W6.9
IDENTIFICATION OF PRESSURE-INDUCED PHASE TRANSFORMATIONS USING NANOINDENTATION. Vladimir Demchenko, Univ. of Illinois - Chicago, Dept. of Mechanical Engineering, Chicago, IL; Yong Gogotis, 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 elastic-plastic material. A sudden volume change during fast transformation results in the discontinuity in the load-displacement curve (‘pop-in’ or ‘pop-out’ events). Sigmoidal 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 (Mayer’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 relations 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 Q8.10/W6.10
MECHANICAL DEFORMATION OF CRYSTALLINE SILICON DURING NANOINDENTATION. Jodie Bradley, J. S. Williams and J. Wong-Leong, Australian National University, Department of Electronic Materials Engineering, RSM/B4, Canberra, AUSTRALIA; M.V. Swan, University of Sydney, Biomaterials Science Research Unit, Department of Mechanical and Mechatronics Engineering and Faculty of Dentistry, Beverly, 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 UMT-200 nanoincident 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-sectional plane through the indentation. Generation 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 source 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 loads above the yield point, a large region of amorphous silicon was observed directly under the indenter when fast unloading conditions were satisfied. The various microstructures and phases observed below indentations are correlated with load/unload data.

11:45 AM Q8.11/W6.11
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 spattering from intermetallic Ti50at.%Al and Ni25at.%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 hardness 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 “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.
Unexpected friction and wear transitions occur in transition metal coated with dislocation emission, dislocation storage, and oxide break-off phenomena. Both normal incipient and kinetic aspects will be reported along with current but speculative ideas on multiple friction and wear transitions. Preliminary results show that yielding under constant load contact can be substantially influenced by normal contact loading whereas a normal contact into [100]W might produce a 100 nm displacement excursion upon large scale dislocation release, a 100 nm/s sliding contact at the same load can produce a 250 nm displacement excursion. Ramifications are seen in terms of friction coefficients which can double during the non-continuous yield excursion, and then continue to triple from about 0.05 to 0.15 in the pile-up phase in front of the sliding contact. Implications of how nanotribological issues such as the transition contact phase to macroscopic friction and wear are discussed.

260 PM Q9.2
FEW ATOMS CONTACTS AND LIQUID LAYER FRICTION MEASURED BY AN ULTRA-HIGH SENSITIVITY AFM
Peter M. Hoffmann, Steve Jeffery, Ralph A. Grimble and John B. Pethick, University of Oxford, Department of Materials, Oxford, UNITED KINGDOM, Ahmet Oral and H. Ougier-Ozer, Department of Physics, Bilkent University, Ankara, TURKEY.

An interferometry-based ultrahigh-sensitivity AFM technique was used in both UHV and liquid to investigate sub-nanometer mechanical properties. We present recent results from UHV experiments showing the mechanical behavior of a few atom contact between a metal tip and clean semiconductor or metal surfaces. It is seen that atomic rearrangements occur once the surface has been disturbed in such a contact and extends a critical value of the order of 1 eV/atom. Other useful parameters can also be extracted and the limits of a single analytical models to describe the obtained results are discussed. We have also carried out experiments in liquid, in which we measure the variation of lateral stiffness as a function of separation. We find that, in a liquid which is known to undergo ordering at solid surfaces, there are periodic variations in the lateral stiffness with separation corresponding to the molecular dimensions. In these experiments, we are able to examine the mechanical behavior of a lubricating film on the molecular scale prior to the onset of frictional sliding.

2:15 PM Q9.3
INFLUENCE OF TIP RADIUS AND SUBSTRATE ON THE NANO-TRIBOLOGICAL CHARACTERIZATION OF THIN DLC COATINGS Th. Rassfeldt, K. Schaffmann, Fraunhofer Institute for Thin Films and Surface Engineering, Braunschweig, GERMANY.

In this work DLC films with thickness of 20 and 350 nm on different substrates were examined by nanotribology and microscopy in combination with scanning probe microscopy. The substrates (Si, photoresist, quartz glass, Si[100] andAITC ceramic) include very soft high hardnes of 4 GPa up to very hard high hardness of 30 GPa materials providing a wide range of combinations between film and substrate properties (typical hardness of DLC = 27 GPa). The experimental configuration allows the testing of samples with loads between 0.001 and 10 mN and a high local resolution. The tribological characteristics of the various film/substrate systems are analyzed in two different ways. On the one hand a single scan test with linear increasing load is used and on the other hand the samples are exposed to oscillating wear tests at constant loads. The tip radius of the conical diamond tip utilized in these experimental configurations vary between 0.5 and 8 micrometer. Both, the substrates as well as the tip radii, show a significant influence on the tribological behavior of the systems. Experimental results of the friction wear depth, the friction coefficient, the critical load of an onset of irreversible surface deformation as well as the critical cycle number of complete film failure are correlated to parameters of the tribological system like substrate properties, tip radii and film thickness.

2:30 PM Q9.4
MECHANICAL AND MOLECULAR FILM PROPERTIES OF Si-N and Y-O Films: A COMPARATIVE STUDY, Paul M. Seeger, Seeger Research, San Jose, CA; James Kirby, Vacuum-Tech, Inc., Seeger Research, Pittsburgh, PA.

Magnetic recording media are thin film overcoat improved to both their mechanical reliability and to create a chemically inert layer. Typical overcoats are comprised of sprayed carbon with various degrees of either Hydrogen or Nitrogen doping. Due to increasingly stringent recording requirements these protective layers are approaching one nanometer in protective thickness. Both Si-N and Y-O films present an alternative path to these typical overcoats. Si-N are in general use as a dielectric-encapulant in magneto-optical media and Y-O has been used as an overcoat on magnetic media (~200 - 3000A). Molecular calculations using all electron Density Functional Theory (DFT) were performed using cluster models of Si-N, Zr-O and (Diamond) crystalline forms. The derived electronic density of states provides indications of the degree of covalency in these idealized structures and also the degree of charge transfer. Parameters of the electronic structure calculations to properties such as cohesive energy and hardness. Predictions of mechanical properties from DFT are compared with nanoindentation measurements of hardness and wear resistance. An approach to the theoretical properties, measurements are performed on single crystals of modeled material, including Si-N and Zr-O. These results were extended to microscopic wear testing of manufacturable materials (i.e., sputtered thin films on magnetic media) to gain a better understanding of the relationship of DFT predictions and nanoindentation to material performance currently in use.

2:45 PM Q9.5

Self-assembled monolayers (SAMs) consisting of hydrocarbon and fluorocarbon chains attached to silicon wafer were evaluated computationally for their high temperature stability, life cycle and performance for MEMS aerospace applications. High level ab initio calculations of surface interaction were conducted on model compounds to predict the bond strengths holding the monolayer tethered to the MEMS device and relate them to its thermal stability. Non-equilibrium molecular dynamics (NEMD) simulations under sliding periodic boundary conditions were employed to find the frictional force as a function of applied load. The NEMD trajectories were analyzed for the structure and chain dynamics of the SAMs and compared with equilibrium MD results for the SAMs in fluid. The dependence of monolayer penetration, monolayer gasket fraction, wall thermal characteristics and the size of the simulation box on the computed results will be discussed.

3:30 PM Q9.6
MOLECULAR TRIBOLOGY OF highly ORDERED MONOLAYERS. D. Guordon, University of Santa Barbara, Santa Barbara CA; C. Dachly, Swiss Federal Institute of Technology, Lausanne, SWITZERLAND, N.A. Burshtein, Department of Physics, Worcester Polytechnic Institute, Worcester MA.

In order to investigate friction at a fundamental level, atomic force microscopy (AFM) in the wearless regime was performed on a model system - a highly ordered thiol-gold monolayer on mica. In the monolayer, condensed domains with long-range orientational order were present. These domains revealed strong friction anisotropies as well as negligible asymmetry in friction around the long axis. The directionality of these two effects appeared to correlate with the tilt direction of the molecules (more specifically of their terminal alkyl chains) in the monolayer. The molecular tilt responsible for the sliding friction effects was surprisingly small - less than 10 degrees - demonstrating that even small tilts can make a major contribution to friction at the molecular scale. The friction was measured on our model system as a function of applied load. The measurements versus load revealed two or three different frictional regimes (depending on the load range), that correlated well with a systematic stepwise behavior of the height of the domain as measured simultaneously. These discrete effects were attributed to molecular gauche defects created under the stress applied by the tip. Other studies include the friction as a function of sliding velocity, chemical preparation of the tip, alternative monolayers, and substrate. Our work suggests that friction on this system is primarily a mechanical phenomenon.

4:00 PM Q9.7
FRICITION MEASUREMENTS OF MOLECULARLY THIN LUBRICANT FILMS USING QUARTZ CRYSTAL RESONATORS. Alexander T. Lischack1,2, Larry E. Bailey2, George W. Tindall3, Curt W. Frank2, Diethelm Johansen1; 1 MacPherson-Institute for Polymer Research, Mainz, GERMANY, 2IBM Research Division, Stanford, CA, 3TAM Almaden Research Center, San Jose, CA.

Although physics has dealt with the issue of friction since Amonton, its understanding still is mostly phenomenological. It was recently that the classical friction laws have been in the perspective because of investigations with the Scanning Force Microscope and the Surface Forces Apparatus. Typical surface speeds of these methods are approximately 1 mm/s, and frequencies are in the range of f =
0.01-100 Hz. For lubrication purposes polymers are often in use. Because of their
fluid properties they reduce energy loss and wear of the surfaces that is
carried by friction. In modern applications ultra thin (d < 30 Å)
films are frequently required. However, due to this confined geometry
many polymers lose their fluid properties (boundary lubrication).
Perfluoropolyethers are molecular lubricants that keep these
properties even when subjected to extremely working conditions (speeds
at 1 m/s). We developed a quartz resonator method that comes very
close to these conditions. Using quartz crystal resonators, the
dependence on film thickness of the frictional properties of
molecularly thin (d = 4-35 Å) perfluoropolyether films was
investigated. For complete monolayer coverage (d ≥ 25 Å) no energy
loss in friction is detected. It increases, however, with decreasing
film thickness although the surface stays completely wetted.

4:15 PM Q9.8
STRUCTURAL CHARACTERIZATION OF THE NEAR-SURFACE
PLASTIC DEFORMATION OF SEMICRYSTALLINE POLYMER
BLENDs. Houxiang Tang, David C. Martin, The Univ. of Michigan,
Dept of Materials Science and Engineering, Ann Arbor, MI.

Near-surface deformation was introduced to polymer blends with a
Telemede Table1 scratch testing apparatus. The deformation field was
characterized by polarized light optical microscopy, microfocus
pinhole X-ray diffraction, scanning electron microscopy (SEM), and
transmission electron microscopy (TEM). Images of the change in
elecrwice to plastic deformation were obtained around the
scratch, from which a mapping of the polymer chain orientation could be
obtained. The localization of the deformation into shear band
structures around the scratch was revealed through TEM imaging.
Micromechanics models describing the nucleation and movement of the
shear bands will be discussed. Geometrical parameters such as the
scratch width, scratch depth, and recovered scratch depth were
 correlated with the anisotropy in the microcosmic mechanical
properties of materials tested.

4:30 PM Q9.9
TRIBOLOGY OF ALKANE MONOLAYERS: THE EFFECTS OF
TIP FLEXIBILITY AND DEFECTS. Judith A. Harrison, Paul T.
Mukulski, and Alan B. Tutein, United States Naval Academy,
Chemistry Department, Annapolis, MD; Steven J. Stuart, Clemson
University, Chemistry Department, Clemson, SC.

The molecular structure, mechanical properties, and tribological
properties of self-assembled monolayer (SAM) materials have been
studied in great detail using scanning probe microscopies due to their
potential usefulness as boundary-layer lubricants. Salmonon and
coworkers were among the first to suggest that the measured friction
in these systems was linked to the energy dissipated by the formation
of gauche defects. With that in mind, we have used molecular
 dynamics (MD) and a new, adaptive intermolecular reactive empirical
bond-order potential (AIREBO) to examine friction in monolayers
composed of n-alkane chains. Particular attention was paid to the
formation of gauche defects and to the quantification of their
contribution to the total energy dissipated during sliding. In addition,
the utility of carbon nanotubes as scanning probe microscopy (SPM)
tips has received considerable attention. The experiments to date are
non-contact (or tapping) mode experiments that rely on the slender
nature of the nanotubes for increased resolution or accuracy. We have
also used MD simulations to examine the utility of several types of
carbon nanotubes as SPM tips for the study of tribology in contact
mode.

4:45 PM Q9.10
CONSEQUENCES OF COMBINED CHEMICAL AND
TRIBOLOGICAL SIMULATION OF SINGLE CRYSTAL
SURFACES: AN EXAMPLE OF NANOMETER-SCALE
TRIBO-CHEMICAL SURFACE MODIFICATION.
Thomas Dickinson, Rizal Hariadi, and Stephen Longford, Physics
Department, Washington State University, Pullman, WA.

We have examined the effects of exposing slightly soluble inorganic
crystals to aqueous solutions of a wide range of under- and
super-saturations to tribological loading with a scanning probe
microscope (SPM) at various normal stresses, tip radii, and lateral
velocities. The SPM can be used to monitor the wear or deposition
processes. We find well defined conditions for either removal or
deposition at atomic (monolayer) steps. These effects are synergistic
in the sense that the rates are fastest for particular combinations of
these parameters. Crystal structure strongly influences the rates of
both removal and growth at steps, showing unique dependencies on
the bonding geometries of individual steps. These unique phenomena
can be exploited in the production of both atomically flat surfaces
and nanometer-dimension surface modification. Extension of this work
to polymer surfaces in weak solvents will also be reported. This work
is supported in part by the National Science Foundation, ENG/CMS.