SYMPOSIUM BB
Mechanisms of Mechanical Deformation in Brittle Materials

November 28 - 30, 2005

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* Invited paper
SESSION BB1/L4.1 Joint Session: Hard Tissue II  
Chair: Michael Swain  
Monday Afternoon, November 28, 2005  
Room 206 (Hynes)

3:30 PM *BB1.1/L4.1  
Micromechanical and Structural Analysis of Compromised Dental Tissues. 
Erik K. Maloney1, Nicky Kilpatrick2 and Michael Swain3.  
1Paediatric Dentistry, University of British Columbia, Vancouver, British Columbia, Canada; 2Royal Childrens Hospital, Melbourne, Victoria, Australia; 3Biomaterials, University of Otago, Dunedin, New Zealand.

With the continual development of new dental materials and treatment techniques, dental materials research is now more important than ever. Although the traditional method of materials and treatment technique assessment was laboratory based, there is a need for investigation and understanding of vitro research. The aim of this paper is to discuss the micromechanical and structural analysis of compromised dental tissues that affect young children, using a biomaterial and clinical approach. There are a number of causes of developmental defects in children. The most common defect is dental caries which is the bacterial degradation of dental hard tissues. Enamel caries is well understood, although much less work has been conducted on dentine caries. Enamel hypomineralisation and hypoplasia occurs much less commonly than dental caries but is very difficult to treat in affected children.

Although together these conditions account for the majority of work of a dentist treating young children, there is presently a lack of both biomaterials and clinical research on the affected tissues. Therefore, the mechanical and microstructural properties of first, carious dentine from primary incisors and, then, of both enamel and dentine in developmentally defective permanent molar teeth were determined using (among others micro-indentation) ultrasound microscopy (UMIS). These studies improved the biomechanical understanding of these compromised tissues such that it was then possible to develop a unique in vivo study to evaluate the changes in the mechanical and microstructural properties of tooth tissue brought about through some conventional restorative treatments. Each of these investigations will be discussed as, together they contribute to the understanding of the physical properties of compromised tissues and the effects of dental treatment on such properties.

4:00 PM *BB1.2/L4.2  

As early as the end of 19 century, G.V. Black realized the importance of characterizing the mechanical properties of enamel and dentin in order to enable to set requirements on restorative materials. Elastic and shear modulus, tensile and compressive strength, proportional limit, and Poisson's ratio have been determined. Due to the complexity of the structures and to the small samples available, fracture mechanics and fatigue crack propagation (FCP) methodology has only recently been extensively applied to their study. This paper focuses on dentinal fracture toughness (KIC of dentin) and dentino-enamel junction (DEJ). Ritchie and coworkers reported the work of fracture of dentin and identified its anisotropy as a function of the orientation of tubules relative to the plane of crack propagation (PCP). A KIC of 3.08 MPa·m1/2 was determined for human dentin using a compact tension specimen. The determination of KIC of human dentin and the study of the effect of the orientation of the plane of dentinal tubules (PDT) relative to PCP (perpendicular (PE)); parallel and aligned (PAA); parallel and transverse (PAT)] has been reported. A significant anisotropy in relation to KIC of human dentin has been identified/confirmed, with values of ~2 in both PAA and PAT directions and ~1 MPa·m1/2 in PE direction. Kinney concluded, based on AFM indentation results, that tubule orientation had no effect on the elastic behaviour of normal dentin. However, significant differences between peritubular and intertubular dentin with respect to elastic moduli and hardness were identified. Ritchie, Kinney, and Nalla have reported on their detailed fracture mechanics studies on the mechanism of crack growth in elephant and human dentin. The results of two teams on bovine, elephant, and human dentin suggest that simple Paris power-law can describe FCP behaviour. A wide range of m exponents, 4.3-32, have been reported, suggesting that further studies need to be performed in this area.

3:40 PM *BB1.3/L4.3  
1Oral Science, University of Minnesota, Minneapolis, Minnesota; 2Developmental and Surgical Sciences, University of Minnesota, Minneapolis, Minnesota; 3R & D, Foxconn Electronics, Inc., Santa Clara, California.

A white spot lesion is an area of demineralized enamel that frequently occurs around orthodontic appliances. Early stages of lesion formation involve a low pH which causes undersaturation of mineral in the microenvironment with the concomitant dissolution of the boundaries of enamel prisms. The result is a porous mineral structure that is susceptible to acid attack and mineral loss from subsurface enamel, creating a subsurface lesion. The ions secreted to the microenvironment through diffusion reconstitute on the surface and form a hard, moderately dense mineral skin. Previous studies focused on quantifying optical properties of this lesion for diagnostic purposes. Mechanical properties, which determine further the clinical character of white spot lesion, are not well characterized. This study reported an indentation method to measure crush energy absorption - Specific Volume Absorbing Energy (SVAE). Crushing along the lesion body pronounced to the enamel surface was examined to determine the effects of progressive mineral loss. Twenty four enamel slabs were isolated from bovine incisors and polished to create flat surfaces for micro-indentation tests. Twelve (Group I) were used for characterization of white spot lesions. The other 12 samples were immersed in acid gel to create lesions: three samples for each of three demineralization conditions (5, 10, and 15 days). The remaining three un-demineralized samples were used as controls. Another twelve samples (Group II) were used to evaluate the effects of Vitremer, a fluoride releasing restorative material, on caries prevention; they were divided in the same demineralization conditions as described in Group I. Repeat micro-indentations with incremental loads (25g, 50g, 100g, 200g, 300g, and 500g) were performed on each sample. Indentation areas and depths were recorded. Load (force, F) versus displacement (depth, d) curve was plotted. Work (crushing) energy was calculated by integrating areas underneath the F-d curve. SVAE equals the work energy divided by the indentation volume. In Group I, SVAE at high load (500g) decreased monotonically as the subsurface demineralization increased. Higher load broke the skin layer of the remineralized tissue and the subsurface structure dominated the crush behavior. Under load (<100g), SVAE, however, increased as demineralization increased. This was primarily due to the reconstitution of the skin layer remineralization. In Group II, Vitremer appeared to slow down calcium leaching out at short demineralization time; this resulted in a slightly higher SVAE value in 5-day demineralization in Group II than in Group I. At low load, however, SVAE was relatively higher in Group I (0.013 MJ/m3) compared to Group II (0.002 MJ/m3). Vitremer might provide chemical protection while the smooth and slightly rough enamel surface may not possess optimal mechanical strength, i.e., SVAE. (Support by MDRCCB, 3M ESPE Dental Products, and NIH/NIDCR R21DE015410).

4:45 PM BB1.4/L4.4  
Site specific effects of commercial whitening treatments on nano-mechanical properties of human dental enamel.  
Michelle Emma Dickinson, Hysitron Inc, Minneapolis, Minnesota.

With increasing awareness of aesthetic appearance, commercial tooth whitening treatments have become a lucrative component of the oral health care sector. The many products currently on the market include toothpastes and in-tray-gels most of which have a bleaching component to lighten the enamel surface. The availability and cost of these treatments varies greatly depending on the composition of the bleach used and its concentration. Commonly, peroxide based solutions are used as surface bleaching agents in both over-the-counter and professional dental treatment products. There has been concern that these products may be detrimental to the mechanical properties of the enamel surface causing permanent sensitivity. Although there have been numerous studies on the effects of these treatments on the hardness of enamel, their results have been conflicting. Some studies state that there is no effect on either the hardness or modulus of the enamel, and some indicate that the products are detrimental to the mechanical properties of the enamel surface. This investigation studies the effect of different commercial whitening
Porous SiO2 Low-Dielectric-Constant Films. Shou-Yi Chang, Yi-Chung Huang and Bo-Kang Yang; Department of Materials Engineering, National Chung Hsing University, Taichung, Taiwan.

Porous low-dielectric-constant (low-k) materials have been widely developed as inter-metal dielectric layers in the multilevel-interconnect structures of ultrahigh-scale integrated circuits to overcome the problem of serious resistance-capacitance delay. However, mechanical damages of these layers such as film cracking and delamination severely suppress the yield and reliability of microelectronic devices. The mechanical properties of these porous low-k films are especially important and need to be clarified. Nanoinindentation test is now the most popular method for the evaluation of thin-film mechanical properties. However, the hardness of thin films does not sufficiently prevent the failure behavior of interconnects. The real stress-strain behavior of thin films as well as fracture toughness should be thoroughly considered. Essentially the pore content, size, geometry and distribution in the porous films will affect the mechanical properties. Therefore in this paper the porous SiO2 films with different porosity and pore sizes were prepared by TEOS/TEtOH/H2O/BCl3 sol-gel method and spin coating, followed by thermal curing. The mechanical properties of these porous films were measured using nanoindentaion test. True flow stress and fracture energy release rate as well as the deformation behavior of these films were analyzed. Through Fourier transform infrared spectroscopy and energy dispersive spectrometry, pure SiO2 films with mostly Si-O bonding and little OH adsorption were obtained. These films exhibited a very small roughness below 1 nm and possessed a large amount of roughly spherical pores with uniform distribution. For the porous SiO2 films prepared with EtOH = 34% and aging time = 10 hours, the refractive index was measured as low as 1.38. It is observed that the hardness of the porous SiO2 films gradually decreased. With increasing aging time from 2 to 64 hours before curing, the pore size in the cured films increased from several nanometers to several hundred nanometers. The hardness of the films decreased from the maximum values of about 1.2 and 33 GPa with the aging time of 16 hours because of complete bonding construction and then dropped due to the enlarged pore sizes. With increasing solvent content, the porosity slightly increased, and the mechanical properties decreased. From the load-indentation depth curve and converted true flow stress-depth curve, it was observed that the porous SiO2 films deformed elastically at first and then yielded at a stress of about 2 GPa. The maximum fracture energy release rate of these porous films was obtained as about 0.4 J/m2. After yielding, the flow stress of the porous SiO2 films gradually decreased. No obvious dislocation burst phenomenon was observed, and the plastic deformation of the films was expected through the crack initiation and propagation along the large amount of pores.

9:30 AM BB2-4
Small Angle X-ray Scattering Study of Tantalum Oxide Foams. Tony van Buuren1, Trevor Willey2, John Kinney1, Cheng Saw1, Jan Havskey2, Ted Baumann1 and Joe Satcher1; 1LLNL, Livermore, California; 2Advanced Photon Source, Argonne, Illinois.

Although a great amount of study has been devoted to the physical properties of porous structures, it is not clear whether the theoretical models developed to date can be extended to nanoscopic length scales. We have measured how the structure of highly porous tantalum oxide foams changes with uniaxial pressure, and surface environment. How these porous materials react under such conditions (e.g., how they deform with mechanical stresses) will be examined in-situ with a combination of small-angle x-ray scattering (SAXS) and high resolution synchrotron-radiation-computed tomography (SRCT). In particular small angle x-ray scattering, was performed on the Ta oxide foams as a function of preparation conditions. For the 100mg/cc foam the x-ray scattering is consistent with a random orientation of 40 nm thick disk-like structures. We measure how the structure of these materials change in increasing uniaxial pressure. Input from both SAXS and SRCT will be used to generate microstructural models of these foams for analysis with finite-element modeling. The work was supported by the Division of Materials Sciences, Office of Basic Energy Science, and performed under the auspices of the U.S.-DOE by LLNL under contract No. W-7405-Eng-48. The UNICAT facility at the Advanced Photon Source (APS) is supported by the U.S. DOE under Award No. DFM02-91ER45439 and the APS is supported by the U.S. DOE, Basic Energy Sciences, Office of Science under contract No. W-31-109-ENG-38.

9:45 AM BB2-5
Measuring Residual Stresses in Glasses and Ceramics using Instrumented Indentation. Thomas Buchheit and Rajan Tandon; Sandia National Laboratories, Albuquerque, New Mexico.

Residual stresses in glasses and ceramics readily develop during thermal processing, joining or sintering operations, promoting premature failure or long-term reliability problems for engineered
components containing these materials. The goal of this study is to investigate the viability of instrumented indentation as a means of locally measuring and mapping residual stresses. This investigation is rooted in a recently demonstrated approach using spherical indenter tips on metals. [1] Results from that study indicated that the geometry of the indentation experiment, e.g., a relatively blunt tip, produced load slightly exceeding the elastic limit of the material underneath the tip, was required to realize a significant effect of residual stresses on the recorded load-displacement response. Relative to metals, many glasses and ceramics have a high hardness-to-modulus ratio, and therefore are naturally amenable to this method as a technique to measure residual stress. This presentation demonstrates the viability of the technique with finite element simulations and experiments on stressed and un-stressed glass. Results will be presented demonstrating that for successful measurements, when compared to metals, a wider range of tip geometries is acceptable. However, the indentation load must be kept within a relatively narrow range of the elastic limit, but below the cracking threshold. The presentation will conclude with demonstrating the viability of this method for mapping residual stress in a cross-sectional ceramic-metal component. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000. [1] Swadener, J.G., Taljit, B., and Pharr, G.M., J. Mater. Res., Vol. 16, No. 7, p. 2091, 2001.

10:30 AM **BB2.6
Mechanical Properties and Fracture Behavior of Nanoporous Au. Juergen Biener, Andrea M. Hodge, Yinmin Wang, Joel R. Hayes and Alex V. Hanza; Nanoscale Synthesis and Characterization Laboratory, Lawrence Livermore National Laboratory, Livermore, California.

Nanoporous metals have recently attracted considerable interest fueled by potential sensor and actuator applications. From a material science point of view, one of the key issues in this context is the synthesis of nanoporous metals with both high tensile and compressive strength. Nanoporous gold (np-Au) has been suggested as a candidate material for this application due to its monolithic character. The material can be synthesized by electrochemical-driven deaetylating of Ag-Au alloys, and exhibits an open sponge-like structure of interconnecting ligaments with a typical pore size distribution on the nanometer length scale. However, beside the observation of a ductile-brittle transition very little is known about the mechanical behavior of this material. Here, we present our results regarding the mechanical properties and the fracture behavior of np-Au. Depth-sensing nanoindentation reveals that the yield strength of np-Au is almost one order of magnitude higher than the value predicted by scaling laws developed for macroscopic open-cell foams. The unexpectedly high value of the yield strength indicates the presence of a distinct size effect of the mechanical properties due to the sub-nanometer dimensions of the ligaments, thus potentially opening a door to a new class of high yield strength - low density materials. The failure of np-Au under nanoindentation stress was evaluated in microscopic examination of fracture surfaces using scanning electron microscopy. On a macroscopic level, np-Au is a brittle material. However, microscopically np-Au is very ductile as ligaments strained by as much as a factor of 100 were observed in the nanoindentation scale. Cell-size effects on the microscopic failure mechanism were studied by annealing experiments whereby increasing the typical pore size/ligament diameter from ~100 nm to ~1 micron. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract of No.W-7405-Eng-48.

11:00 AM BB2.7
Brittle Behavior in Nanoporous Au. Cynthia A. Volkert and Erica T. Lilleodden; Forschungszentrum Karlsruhe, Karlsruhe, Germany.

We have investigated the mechanical behavior of coarse-grained, nanoporous Au. This material is made by electrochemical dissolution of AuAg sheets with a nominal grain size of 100 μm and results in a structure with wall size of 30 nm. On macroscopic length scales and under general loading conditions, the material behaves in a brittle fashion. This is evidenced by the many cracks in the as-processed material and the fact that it is very difficult to handle. However, on the microscopic length scale and during compression, the material behaves in a ductile fashion. These uniaxial compression experiments are conducted with a nanoindenter, outfitted with a flat punch, on micron-sized cylindrical columns machined using a focused Ga beam. The columns deform uniformly in the driving compression and do not form cracks. The results suggest that the apparent brittle behavior of the Au either requires a certain flow distribution which may be absent in the micron-sized specimens, or requires loading conditions other than uniaxial compression. The formation of circumferential cracks around micron-sized indents into a flat nanoporous Au surface points towards the importance of the loading conditions in causing brittle behavior. To more clearly distinguish between these two possibilities, experiments are in progress in the micrometer-sized, notched cantilever beams with a nanoindenter tip. As supported by experimental observations, the effect of flaw distribution and loading configuration on the brittle behavior will be discussed within the context of porous solids.

11:15 AM BB2.8

Hard thin films are increasingly employed for applications where wear resistance is important; however, many current deposition techniques for candidate film systems produce high residual growth stresses which can markedly decrease the coating’s mechanical stability. Consequently, it is essential to have a reliable method for examining the elastic, plastic and fracture behavior of thin, hard films in order to optimize coating choice, proper deposition parameters, and device design. In the current study, the fracture behavior and toughness of thin amorphous diamond films were examined using acoustic sensing nanoindentation. Laser annealing was used to induce several levels of film stress ranging from ~3.7 GPa (compressive) to ~2.4 GPa (tensile). Indentations into the films, using a cube-corner indenter geometry, produced radial cracks that extended into channel cracks with increasing indentation depth. It was found that both the indentation load as well as the sign and magnitude of the residual film stress, with tensile stresses promoting crack extension. Established fracture toughness models for channel crack morphologies, accounting for both residual and indentation driving stresses, were evaluated. (Thurston and Cook, 2004; Lain, 1993) and calculations of the diamond films displayed fracture toughness values ranging from 3.3 to 3.7 MPa·m1/2. These results are in excellent agreement with experimental findings from a similar amorphous diamond system obtained using MEMS-based micro-toughness tests. Results from an in-situ acoustic sensor also showed a relationship between the released acoustic energy and the total crack length. This was used to formulate an expression for the fracture toughness of the thin films as measured from the released acoustic energy with a good toughness agreement compared to the standard indentation-based measurement. *Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

11:30 AM BB2.9
In situ high temperature x-ray diffraction (HTXRD) study of the oxidation behavior of the microwave sintered Si3N4. Sreekumar Cheolkalingam, David A. Earl and Scott Miiatree; NYSER at Alfred University, Alfred, New York.

An investigation on the oxidation behavior of microwave sintered Si3N4 was carried out. Along with excellent thermal, wear resistance and mechanical properties, high temperature oxidation behavior is another important property to be studied in the case of Si3N4 ceramics due to its high temperature structural applications. Dense beta-Si3N4 samples were prepared from alpha-Si3N4 powder by sintering at 1650°C for 20 min using 2.45 GHz microwave energy. High temperature XRD (HTXRD) has been successfully applied to study the oxidation behavior of microwave sintered specimens containing Si3N4, 6 wt %Y2O3, 4 wt %MgO, 0-12 wt %ZrO2 at various temperatures. The samples were heated to 900°C from 20°C and cooled to room temperature. No variation in the diffraction patterns was observed. The same sample was exposed to air at 900°C suggesting the sample is oxidation resistant. In contrast, the conventionally sintered samples showed surface oxidation after 6 hours at 900°C. The oxidation behavior is attributed to the fraction of alpha vs beta Si3N4 and compared between the samples processed via conventional and microwave sintering.

11:45 AM BB2.10

The hardness of hot pressed boron carbide/carbon nanotube (B4C/CNT) composites usually decreases with increasing amount of CNTs due to bad adhesion of the CNTs to the B4C matrix. Since CNTs possess significant potential to improve the mechanical properties of such composites, the surfaces of the CNTs were reacted...
with B and B$_2$O$_3$ vapor to strengthen the adhesion between the CNTs and B$_4$C. This treatment resulted in either a B$_4$C coating on the CNTs or B$_4$C nanorods, as observed by TEM, depending on the processing conditions. Electron diffraction patterns indicated a preferred crystallographic orientation of the B$_4$C coating with respect to the graphitic surface layers of the CNTs. The modified NTs (mNTs) were further coated with B$_4$C (i.e., HCC Starke) with a grain size of about 500 nm in various compositions up to 10 w% mNTs. The density of low temperature, hot pressed composites critically depended on the structure and kind of mNTs; the maximum increase in the density of composites with increasing mNT content was observed for coated CNTs rather than for polycrystalline B$_4$C nanorods. The deformation mechanisms of the composites containing different kinds of nanotubes are being studied by a comparison of different hardness indentations and by additional characterization. The results will be reported. This work was supported by the Nanoscale Science and Engineering Initiative of the National Science Foundation under NSF Award Number DMR-0117792 and by the U.S. Army under Contract Number W911QY-04-C-0111.

SESSION BB3: Modelling and Simulations
Tuesday Afternoon, November 29, 2005
Liberty (Sherron)

1:30 PM *BB3.1

Atomatic mechanisms of nanoindentation of nanocrystalline ceramic thin films have been studied by multimillion-atom molecular dynamics simulations on parallel computers. The increased volume fraction of highly disordered intergranular films as compared to nanocrystalline metals manifests itself in unique deformation mechanisms. Our study of nanocrystalline silicon carbide provides a scenario for the interplay between grain rotation, corporate grain motion, sliding at grain boundaries and intergranular deformation to produce a rich load-displacement response. We predict a crossover from continuous corporate grain response to discrete inter-grain plasticity at a critical depth that is a fraction of the grain size. The crossover phenomenon is expected to play an important role in the design of nanocrystalline materials with enhanced mechanical properties.

2:00 PM BB3.2
Dislocations Deflect and Perturb Dynamically Propagating Cracks, Dov Sherman, Materials Engineering, Technion-Israel Institute of Technology, Haifa, Israel.

We demonstrate that in single crystal silicon, short-range collisions of a dynamically propagating crack with stationary intrinsic inclined dislocations generate local crack deflections that grow to a large surface perturbation. Experiments show that when the crack collides with a single dislocation, the perturbation height is about 8 nm, but it collides with a large array of dislocations, the perturbation can extend up to 80 nm in height (~200 b), 250µm in length, and visible to the naked eye. A model was developed formulating the maximum velocity at which the crack climbs into the dislocations’ wake. This model predicts that when a dislocation’s trace line is perpendicular to the crack surface, no interaction takes place.

2:15 PM BB3.3
Atomaticic Configurations and Energetics of Crack Extension in Silicon, Ting Zhu1, Ju Li2 and Sidney Yip1; 1Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; 2Department of Materials Science and Engineering, Ohio State University, Columbus, Ohio.

A fundamental question in understanding fracture at the atomic level is how a sharp crack advances by a sequence of bond breaking events. Such insights would elucidate a wide range of phenomena, such as slow crack growth and environmental effects. In this work we apply a reaction pathway analysis to determine the minimum energy path for bond breaking along 3D, atomically sharp crack fronts in Si. Treating this path as a reaction coordinate we investigate the atomic configurations as well as the energetics of the crack front during the advancement of the crack tip by one atomic spacing. We find the crack-front extension occurs through a kink mechanism, the nucleation of a double kink followed by the spreading of this kink pair across the front. This scenario is essentially the same as the mechanism for glide of partial dislocations in a SiC cubic lattice of Si. We are able to extract activation barriers in terms of kink nucleation and migration energies. Our investigation also leads to manifestations of lattice trapping and directional cleavage anisotropy effects beyond those discussed in 2D simulations.

3:30 PM *BB3.4
Investigating the Deformation Mechanisms in Shocked Crystals by Large-Scale Molecular Dynamics. Ramon Bayoño, 1Physics Department, University of Texas, El Paso, Texas; 2Los Alamos National Laboratory, Los Alamos, New Mexico.

The physical phenomena associated with failure, deformation or phase changes in brittle or hard solids under shock loading are still not well understood. Employing large-scale molecular dynamics simulations with 10^12 to 10^13 atoms, we have begun to elucidate the response of crystalline solids to shock wave and high strain-rate loading. A brief overview of the main characteristics of shock-induced deformation observed in models of brittle solids will be presented and contrasted with those observed in defect-free metallic single crystals which exhibit a high Hugoniot elastic limit (HEL). The observed deformation mechanisms correlate with the shock-induced stress state which depends strongly on the shock propagation direction. Good agreement with experimental observations, the deformation in fcc metals shocked along the (100) crystallographic direction, proceeds via nucleation of partial dislocation loops at the shock front. This is in contrast to (111) and (110) shocks, which exhibit different plastic states with an elastic-plastic transition characterized by a two-wave structure and a large volume collapse. Work supported by the U.S. Department of Energy under Contract No. W-7405-ENG-36.

4:00 PM BB3.5
Dynamical Fracture Instabilities due to Local Hyperelasticity at Crack Tips, Markus J. Buehler1 and Huajian Gao2; 1Department of Civil and Environmental Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; 2Max Planck Institute, Stuttgart, Germany.

Cracks moving at low speeds create atomically flat mirror-like surfaces, whereas cracks at higher speeds leave increasingly rough fracture surfaces. This change in fracture surface morphology is a universal phenomenon found in a wide range of different brittle materials. The underlying physical reason of this instability has been debated over an extensive period of time. Most existing theories of fracture assume a linear elastic stress-strain law. However, the relation of stress and strain in real solids is strongly nonlinear due to local deformation near a moving crack tip, a phenomenon referred to as hyperelasticity. Using massively parallel large-scale atomistic simulations, we show that hyperelasticity can play a governing role in dynamical crack tip instabilities in fracture of brittle materials. We report a simple analytical model that treats the instability problem as a competition between energy flow and deformation field controlled mechanisms, and allows predicting the onset of instability for different material behaviour. We demonstrate that the recently discovered characteristic energy length scale is critical not only to understand the limiting speed of cracks as suggested earlier (Buehler et al., Nature, 2003), but also to form a complete picture of the role of hyperelasticity in the instability dynamics. Our results help to explain controversial experimental and computational results, as for example mode I cracks in homogeneous materials moving at speeds faster than the shear wave speed. We demonstrate that such behaviour, strongly contrasting the classical theories, can only be understood from a viewpoint of hyperelasticity. The work reported in the present paper helps to form a more complete picture of dynamic fracture, and strongly suggest that hyperelasticity is crucial for dynamic fracture, both in order to understand the instability dynamics as well as to comprehend the crack limiting speed.

4:15 PM BB3.6
Anisotropic plasticity in NiAl alloy from molecular dynamics. Alejandro Strachan1 and Shen-Nian Luo2; 1Materials Engineering, Purdue University, West Lafayette, Indiana; 2Los Alamos National Lab, Los Alamos, New Mexico.

We use molecular dynamics with a first principles-based interatomic potential to characterize the orientational dependence of plastic deformation in NiAl B2 alloy under dynamical and quasi-static loading. For all directions studied plasticity starts with the nucleation of superpartial loops encircling 1/2[111]> slip but the subsequent events exhibit marked anisotropy. For shocks in the [110] direction we find an intricate pattern of <111>[110] and <100>[110] slip with the plastic wave moving at the shock velocity. In the case of [111] shocks plastic deformation is dominated by <100>[110] slip that forms when trailing superpartials nucleate inside the initial 1/2[111]> loops. For shocks in the [100] direction (the hard direction) much stronger shocks [(uniaxial stress almost twice larger than for [110]) and [111]] required before plastic deformation occurs, our MDs help to explain controversial experimental results, as for example, parallel superpartials nucleating in [111] but not in [110] directions, slip that forms when trailing superpartials nucleate inside the initial 1/2[111]> loops. For shocks in the [110] direction (the hard direction) much stronger shocks [(uniaxial stress almost twice larger than for [110]) and [111]] required before plastic deformation occurs, our MDs help to explain controversial experimental results, as for example, parallel superpartials nucleating in [111] but not in [110] directions, slip that forms when trailing superpartials nucleate inside the initial 1/2[111]> loops.
4:30 PM BB4.7
Autocatalytic Avalanches of Unit Inelastic Shearing Events are the Mechanism of Plastic Deformation in Amorphous Silicon. Michael John Demkowicz1,2 and Ali S. Argon2; 1MST-8: Structure-Property Relations, Los Alamos National Laboratory, Los Alamos, New Mexico; 2Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Discrete stress relaxations were found to be the source of low-temperature plastic flow in amorphous silicon (a-Si) as modeled by atomistic simulation using the Stillinger-Weber potential. These relaxations are triggered when a local yielding criterion is satisfied in a small cluster of atoms. The atomic rearrangements accompanying discrete stress relaxations are describable as autocatalytic avalanches of unit shearing events. Every such unit event centers on a clearly identifiable change in bond length between the two split peaks of the second nearest neighbor shell in the radial distribution function (RDF) of bulk a-Si in steady-state flow.

4:45 PM BB8.8
Densification of Silica under Pressure. Liping Huang and John Kieffer; Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan.

Inorganic compounds with rigid polyhedral building blocks, like silica, often possess significant capacity for densification under pressure due to their open structures. Our molecular dynamics (MD) simulations reveal that this densification follows a ubiquitous two-stage mechanism. First, a compact high-symmetry anion sub-lattice forms, governed by the repulsion between the anions and the attractive forces between cations redistribute onto interstices of this sub-lattice. Our simulations demonstrate that anions form a hexagonal close-packed (hcp) and a body-centred cubic (bcc) sublattice in the high-pressure cristobalite and quartz silica, respectively. Lower-energy pathways are created in the closed-packed anion sub-lattices for cations to move from low- to high-coordination states. Depending on the synchronization of cation movement between interstices different transformations can be observed. We therefore submit that the formation of such close-packed anion sub-lattices, common to at least the two major polymorphs of silica under pressure, may constitute a universal mechanism for the densification of compounds with polyhedral building blocks.

SESSION BB4: Poster Session: Mechanisms of Deformation in Brittle Materials

Chairs: Jodie Bradby and Sergei Kucheyev
Tuesday Evening, November 29, 2005
8:00 PM
Exhibition Hall D (Hyves)

BB4.1
Reverse Taylor Anvil-on-Rod Impact Tests for Validation of Constitutive Equations for Brittle Materials. Louis Ferranti, Daniel Eakinis, Morgana Martin and Naresh N. Thadani; Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Reverse Taylor anvil-on-rod impact tests were performed on samples of various materials including OFHC copper, shock-compacted Ni-Al powder mixture, Ni-Al-epoxy composites, Al+Fe2O3+epoxy composites, and W-reinforced Zr-based metallic glass. The dynamic yield strengths and deformation and failure mechanisms of recovered impacted specimens were characterized and correlated with their structure and processing conditions. Constitutive equations used to describe the deformation and failure response of the materials were validated by matching simulated velocity traces with those obtained via velocity interferometry, as well as by correlating transient deformation profiles captured using high speed digital photography with simulated deformation geometries based on constitutive models.

In this paper, we will describe the applicability of the reverse Taylor anvil-on-rod impact experiments for developing and validating constitutive equations for ductile and brittle materials.

BB4.2
Abstract Withdrawn

BB4.3
Processing and characterization of RF magnetron sputtered TiN films on AISI 420 stainless steel. Youngman Kim and Seungwoo Song; Materials Science and Engineering, Chonnam National University, Gwangju, South Korea.

Titanium nitride (TiN) coatings were produced on AISI 420 stainless steel by magnetron sputtering of a TiN target changing the processing variables, such as the flow rate of N2/Ar, substrate temperature and the existence of Ti interlayer between TiN coatings and substrates. The hardness and the residual stress in the films were investigated using nanoindentation and a laser scattering method, respectively. The stoichiometry, surface morphology and adhesion were investigated using glancing - angle X-Ray Diffraction, Scanning Electron Microscopy, Atomic Force Microscope and Scratch tests. The corrosion property of the films was also studied. A polarization method in NaCl (0.9%) solution. Mechanical properties including hardness and residual stress were related to the ratio of N2/Ar flow rate. The corrosion resistance also was related to the processing variables. The adhesion property was improved when Ti interlayer was employed. Acknowledgment: This study was sponsored by the KOSEF CAPST and the authors are grateful for this support.

BB4.4
Mechanical and Thermal Properties of Well-Dispersed Alumina/Epoxy Nanocomposite. Su Zhang1, Linda S. Schadler1, Richard W. Siegel2, Carina Omeley2, Henrik Hilborg2 and Tommaso Auricica1; 1Materials Science and Engineering Department, Rensselaer Polytechnic Institute, Troy, New York; 2ABB Corporate Research, VSTERS, Sweden.

One of the potential advantages of nanoparticle filled thermosets is the unique combination of mechanical properties that can be obtained. In this study, well-dispersed Al2O3/epoxy nanocomposites were obtained by shear mixing using a HaakeSpeedMixer. There was no change in the glass transition temperature of the composites, but the Young's modulus, strength, strain-to-failure, and thermal conductivity of the nanocomposites all increased upon addition of nanoscale Al2O3 and were optimized at a particle loading of 10 phr (w/w). The fracture surfaces of the tensile bars were characterized by SEM. The fracture morphology of the 10 phr nanocomposites showed a marked difference compared to other nanocomposites. Two different crack morphology regions were observed. The center of the circle region is composed of cracks. Cracks were straighter and sharper in this region compared to the second region. The outer region showed small scale-like cracks, which deflected around the nanoparticles. These two morphologies suggest that the crack growth was abated. This may be the reason why 10 phr nanocomposites had a higher strain-to-failure compared to the others. Finally, the effects of the nanoparticle/matrix interface are also under investigation by functionalizing Al2O3 nanoparticles with an epoxy compatible silane coupling agent. Results will be reported. This work was supported by ABB Group Service Center and the Nanoscale Science and Engineering Initiative of the National Science Foundation under NSF Award Number DMR-0117792.

BB4.5
Fractal Dimension for Ceramic Fracture Surface. Piotr Kotowski1 and Jerzy Bielecki2; 1Wrocław University of Technology, Wrocław, Poland; 2Energy Institute, Warszawa, Poland.

The study of the morphology of fracture surfaces is lately very active field of research. It is well established that the fracture properties of heterogeneous materials depend on the microstructure and the damage process of the material. Both these parameters may have a strong influence on the local deviations of the main crack and thus on the shape of fracture surface. Relatively not so long ago has the concept of fractals revealed useful tool in that respect. Many experiments on materials as different as ductile aluminum alloys to brittle materials like rock have shown that the topology of fracture surfaces is self-affine. However, the fractal dimension characterising the surface D is ~2.2 (or fractal exponent H = 0.8) has been reported in all cases. These results seem to support the idea that the fractal dimension of fracture might have a universal value, i.e., independent of the material. To our knowledge the most experiments in this field relate to study of the fracture surfaces obtained under conditions of static torn. In this paper we present experiments for fracture obtained under conditions of cyclic loading as well. The fractal dimension of electrotechnical ceramic fracture surface has been estimated. This material is commonly used for the high voltage insulators in the power transmission. The specimens were breaking under uniaxial cyclic and static loading conditions. Another part of the specimens were subjected to impact bending for the fractal dimension estimation a method used for metals fracture had been adapted. In the method the fractal dimension was determined for the lateral profiles obtained by the profile technique cross-section. On the basis of the value of the fractal dimension for the fracture surface was estimated. The image of the profiles recorded at proper magnification are subjected to binarization and contouring. During these operations the tests which enable minimization of the error are carried out. For calculation of the same fractal dimension, the FFT technique has been used, which is available through the Internet and it allows computing of the capacity fractal dimension (box-counting). Above mentioned method allows determination of the fractal dimension with an accuracy of 0.05. The results obtained in the work seem to support idea, that fractal dimension of fracture surface is a universal value. For
all tested specimens identical fractal dimensions equal 2.10 were obtained irrespective of the loading conditions. The results for ceramic and iron alloys were compared. In all cases no correlation between the fractal dimension and materials properties neither with loading conditions was observed.

BB4.6 Indentation-induced Crystallization and Phase Transformation of Amorphous Germanium. Gilles Patriarchi1, Eric Le Bourhis2, M. M. O. Khayyat3 and M. Mumaw Chaudhri2.
1Laboratoire de Cristallographie et de Nanosciences, CNRS UMR 5160, Marcoussis, France; 2Laboratoire de Metallurgie Physique UMR 6630 CNRS, Universite Poitiers, Futuroscope-Chasseneuil Cedex, France; 3Cavendish Laboratory Department of Physics, University of Cambridge, Cambridge, United Kingdom.

Silicon and germanium are of considerable technological and scientific interest. When subjected to sufficiently high hydrostatic and nonhydrostatic pressures and temperatures, the amorphous phase can transform to a crystalline phase through a process of crystallization. In this transformation, the amorphous material is first converted into a partially crystalline state, and then the crystalline phase is formed by the growth of well-defined nuclei. The crystallization process is typically characterized by a decrease in the free energy of the system, which drives the transformation. The rate of crystallization depends on the temperature, pressure, and the structural and chemical properties of the amorphous material. The transformation from the amorphous to the crystalline phase can occur over a range of temperatures and pressures, and the resulting crystalline structure can be highly dependent on the conditions of the transformation.

BB4.7 Compositionally induced stresses in non-stoichiometric oxide films. Brian W. Sheldon, Siddharth Bhatia, Sunil Mandowara, Tai Hee Eun and Jane Rankin; Division of Engineering, Brown University, Providence, Rhode Island.

Variations in the oxygen content of non-stoichiometric films have been widely studied in many materials, with particular attention to point defects and transport processes. Composition changes can also lead to stresses in thin film materials that are constrained by an underlying substrate. This phenomena was investigated with precise in situ curvature measurements at elevated temperatures, in both TiO$_2$-x and CeO$_2$-x films produced by metal organic chemical vapor deposition and sol gel methods. In addition to the potential technological applications of these materials, they were specifically chosen because they have chemical and transport properties. Some of these systems have been widely investigated by others, and because the range of non-stoichiometry in ceria is particularly large. The experimental results with these films provide direct information about relationships between composition, point defects, films growth, and diffusion. In many cases, the observed stresses were too large to be caused solely by the molar volume change associated with changes in just the point defect concentrations. This suggests that stresses are also induced by Kirkendall-type operations operating in these materials. To interpret the stress data, a theoretical framework was developed to describe compositionally induced stresses in both single crystal and polycrystalline films. The results show that the direction (tensile or compressive) and magnitude of the stress depends on both the dominant point defects and the relative diffusivities of different species. The effects of dopants and impurities were also considered. The overall framework developed in this work should be applicable to a wide variety of different oxide materials.

BB4.8 Investigation of Inelastic Behavior Observed in Thermally Sprayed YSZ, Yagie Li, Andrew Nakamura2, Sanjay Samplph2,1 Department of Mechanical Engineering, State University of New York at Stony Brook, Stony Brook, New York; 2Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, New York.

Thermally sprayed ceramics are commonly used as thermal barrier coatings in high temperature environments. Their attractive feature, low thermal conductivity, is due to the presence of many voids and inclusions found in their microstructure. However, these geometrical inhomogeneities also cause unique deformation characteristics that are different from those of bulk ceramics. In this paper, substrate curvature-temperature measurements obtained during thermal cycles are used to investigate the inelastic behavior of Yttria (Y2O3) Stabilized Zirconia (ZrO2) coatings (YSZ). There appears to be two classifications. One is nonlinear response of coating as stresses change during thermal loading, and the other is anelastic hysterisis of stress-strain-related during complete thermal cycle. The existence of many cracks probably explain the former behavior while the mechanism leading to the second characteristic is still unclear although one may speculate the cause as frictional sliding of splot interfaces. The nonlinear response occurs when some cracks close under large compression but open during tensile condition. In other words, the stiffness of coating varies as the internal stress transition from compression to tension or vice versa. To clarify the cause of crack opening and closing, we have developed an inverse technique for the effective stress-strain relation of coating from substrate curvature measurements. It is a versatile procedure that utilizes Kalman filter technique. Its accuracy is verified by detailed finite element simulation. The technique can also be used to estimate residual stress in coating after fabrication at room temperature.

BB4.9 Using Femtosecond Pulseshaping to Characterize Polymer Structure and Dynamics: Deathstar GHz Spectrosopy. Neal A. Vachani2,3,4, Jyotirmoy D. Chou1, Kenji Katayama2, Keith A. Nelson1,2 and Edwin L. Thomas1,2.
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A femtosecond pulseshaper called the Deathstar has been used to generate optica pulse sequences with repetition rates in the 5 - 500 GHz range, and these are used to excite acoustic waves in the same frequency range in amorphous polymer samples. With straints of roughly 10^-4, this corresponds to ultrahigh strain rates (10^4 to 10^6 s^-1). The frequency dependence of the longitudinal acoustic velocity and the frequency dependence of the acoustic attenuation rate have been measured under such conditions. The frequency range of longitudinal phonons studies is not directly accessible by other spectroscopies. Probing polymer responses in this intermediate regime is valuable because it helps characterize secondary transitions and energy dissipation mechanisms. Bandwidth experiments have been done to study the temperature dependence of the longitudinal acoustic velocity (for polystyrene and poly(methyl methacrylate)) from 20 to 300 K. The observed dynamic impedance at small strains is relevant for evaluation of potential transparent armor materials. The results are in line with literature values and the predictions of a model based on acoustic impedance mismatch theory. Narrowband studies with the technique used were previously limited to amorphous silicon. They are extended for the first time to amorphous polymers. Deathstar GHz spectroscopy is used to determine the absolute acoustic attenuation coefficient for PMMA as a function of frequency from 55 to 180 GHz. To explore additional dynamics, attenuation is also measured at temperatures above and below the polymer glass transition. The variation of this technique for different polymers and its potential for future studies in this research area.
have been of great interest recently. This paper presents effect of loading conditions on pop-up phenomena on the load-depth curves for BCC and FCC crystalline structures (W, Fe, Ni) and nanocrystalline Ni. Change in deformation mechanisms due to variations in strain rate has received attention mostly in amorphous materials. In an attempt to understand incipient plasticity and the change in deformation mechanisms with a change in strain rate, nanoinindentation tests were carried out with different strain rates using a pyramidal indenter up to a depth of 100 nm. A strong dependency on crystallite structure and type of pop-ups is observed. Results indicate grain size and microstructure play important roles in small-scale incipient plasticity.

BB4.12 Deformation Facilitated by Symmetric Tilt Grain Boundaries of Rocksalt-Structured Ceramics at High Pressure
James Palko1,2,3 and John Kieffer2; 1Aerospace Corporation, El Segundo, California; 2Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan; 3Materials Science and Engineering, University of Illinois, Urbana, Illinois.

Grain boundaries have long been known to be vitally important to the deformation behavior of ceramics. Interest was generally centered on their roles as sources of intrinsic defects that act as crack nucleation centers or as reservoirs of amorphous phases that soften drastically with temperature allowing grain boundary sliding. As ceramic processing technology has advanced and resulted in much-improved microstructures, other, smaller scale, processes occurring at grain boundaries have become significant. Further advances in grain boundary engineering aimed at improving the mechanical behavior of ceramics increasingly rely on insights into the atomic level details of the structure and dynamics of the grain boundaries. Here we present the results of some recent atomistic simulations dealing with symmetric (1 0 0) tilt grain boundaries in rocksalt structured ceramics subjected to various simple loading conditions. These simulations have corresponded to the atomistic scale and enabled us to number processes, including a pressure induced rearrangement in these grain boundaries that may have broad implications for diffusional and dislocation modes of deformation in ceramic materials at high pressures. A remarkable reduction in sliding resistance (of the (1 0 0) grain boundaries accompanies this transformation in rocksalt-structured ceramics. We present atomic and continuum models that account for the boundaries relative stability and mobility.

BB4.13 Surface and subsurface nanomechanical properties of amorphous carbon thin films. Spiros Kassavetis and Stergios Logothetidis; Physics Department, Aristotle University of Thessaloniki, Thessaloniki, Greece.

The amorphous carbon thin films, hydrogenated (a-C:H) or non-hydrogenated (a-C), have attracted the interest of the research community due to the wide range of different applications. The a-C and a-C:H thin films are excellent candidates as protective overcoats for optical devices and barrier coatings for packaging, because of the combination of optical, electrical, mechanical, and thermal properties. The a-C and a-C:H thin films were grown on c-Si substrates from the vapour phase using rf biased and unbiased (MS) rf magnetron sputtering. The thin films are characterized by in-situ spectroscopic ellipsometry (SE), in the energy region 1.5-5.5 eV, in order to control their thickness, optical properties and structural composition (content of sp2 and sp3 hybridized carbon bonds). The surface topography was studied using Atomic Force Microscopy in contact mode. The imaging of the surface nanomechanical properties of the a-C and a-C:H was realized using the surface sensitive, non-destructive Atomic Force Acoustic Microscopy (AFAM), which is a sophisticated technique, based on atomic force microscopy principles, that enables comparative nanomechanical properties imaging through the simultaneous imaging of the variations of the surface nanomechanical properties (acoustic image) and the surface topography. On the other hand, the depth sensing or contamination (DNI) was employed for the study of the variations of the hardness (H) and the elastic modulus (E) of the thin film/substrate system versus the indentation depth. The adhesion of the samples to the substrate was determined using Scratch Test (ST) combined with lateral force measurements. Acoustic imaging showed intense variations of the nanomechanical properties at the surface for the MS a-C and a-C:H thin films, which were eliminated by increasing the applied bias (Vb) at the substrate. Also, the comparison between the topographic and acoustic image showed that the surface picks/valleys were corresponding to areas with lower/higher elastic properties. The NI results revealed the presence of fracture at 2mN for the MS a-C:H, which was also eliminated by increasing Vb to 40V. These observations were also confirmed by the ST, which showed the delamination of the MS a-C:H thin films for scratching performed by applying 0.02 to 25mN normal load. Moreover, the H, E values measured by NI were found to significant increase with increasing Vb. Finally, we validate the measured nanomechanical properties with the structural characteristics revealed by SE.

BB4.14 Non-Linear Deformation Behavior of La1-xCaxCoO3 Perovskites as a Function of Ca Doping. Siddharta Pathak1, Nina Orlowskaya2, Jakob Kuebler3 and Mike Reece2; 1Materials Science and Engineering, University of Pennsylvania; 2Lab for High Performance Ceramics, EMPA, Swiss Federal Lab for Materials Testing and Research, Dübendorf, CH-8600 Switzerland; 3Department of Materials, Queen Mary University of London, London, United Kingdom.

The non-linear ferroelastic behavior of Ca doped LaCoO3 perovskites has been recently discovered. A basic understanding of the origin of ferroelastic properties is not currently available for lanthanum cobaltites, this study explores the ferroelasticity and hysteresis in these perovskites as a function of external load (compression and bending), temperature and materials composition in terms of various levels of Ca doping (Pure LaCoO3, 20%, 40% and 55% Ca doped LaCoO3). First, the mechanical properties of the cobaltites were characterized in terms of their bending strength and fracture toughness, both at room temperature and at elevated temperatures (700 and 800°C). The load-deformation and stress-strain curves for these compositions, at room temperature and 800°C, help to compare the ferroelastic property as a function of Ca doping. In compression tests at room temperature, the inflexion point for the pure (undoped) compounds was found to be around 70 MPa. The inflexion point increased with increased Ca doping, around 150 MPa for 20% Ca doped LaCoO3 and about 300 MPa for each of 40 and 55% Ca doped LaCoO3. The cycling loading/unloading tests under compression corresponded to 4 point bending tests. Domain reorientation under loading was confirmed by XRD analysis.


Nanindentation is a powerful technique to study sub-micron constrained deformation behavior in materials. To date the nanoindentor of choice appears to be the Berkovich indenter; less work has been carried out with spherical nanoindenters. The major advantage of the latter is that the load displacement curves can be converted to stress-strain plots from which crucial information concerning the mechanical response of the indented solids can be obtained. From the stress-strain results the elastic moduli, yield stresses and energy dissipated per cycle are readily obtainable. Herein, we describe how repeated loading on the same location is a powerful technique in identifying the non-linear and the elastic (KNE) solids. This newly classified class of solids is quite large and includes all solids with a c/a ratio greater than about 1.5. The stress-strain curves of KNE solids are non-linear, and outline fully reversible or closed hysteresis loops. The energy dissipated per cycle is substantial. This work describes an analytic model that is capable of estimating the number, shape and distribution of dislocations under the indenter, as well as their evolution with increased load. The agreement between the experimental and theoretical results is excellent. The implications of using this technique to the characterization of KNE solids are discussed. 1. M. W. Barsoum, A. Murugaiyah, S. R. Kalidindi and T. Zhen, "Kinking nonlinear elastic solids, nanoindentations, and geology", Phys. Rev. Lett. 92 (25) 255508 (2004). 2. M. W. Barsoum, T. Zhen, A. Zhou, S. Basu and S. R. Kalidindi, "Micrscopic modeling of kinking nonlinear elastic solids". Phys. Rev. B 71, 134101 (2005).


In the past few years significant research interest has been attracted to single crystal ZnO as a potential material for short-wavelength optoelectronics. A good understanding of the properties is needed for a successful fabrication of these (opto)electronic devices. Depth sensing spherical nanoinindentation is an effective tool to characterize the stress-strain behavior, yield point, modulus and energy dissipation during deformation in a single crystal material. In this study we show the significant advantages of repeatedly loading spherical indenters of two different sizes - into the same location. And while what is occurring during the first cycle can be ambiguous, the hardening, reversibility and closed loops observed in subsequent cycles
BB4.17
A Numerical Model for Intergranular and Cleavage Fracture in Ceramic Materials. Reuben H. Kraft, Derek W. Warner and Jean-Francois Molinsari; Mechanical Engineering, Johns Hopkins University, Baltimore, Maryland.

The performance of ceramic materials is tightly linked to damage mechanisms at the microstructural length scale. In the case of high-performance ceramics, substantial grain in ductility may be obtained by allowing a certain amount of microcracking at the grain level while preserving macroscopic cohesion. With a focus on the intergranular and cleavage fracture mechanisms in ceramic materials, this presentation proposes a numerical framework for investigating novel microstructural design strategies. A finite-element model is developed to explicitly capture damage evolution at the grain level. Microstructural uniform and bimodal grain size distributions are obtained through homogenization methods. Cohesive elements with well-characterized material parameters are used to model cracking at the grain boundaries as well as cleavage fracture. We validate the approach by considering a transition between the two mechanisms for increasing grain sizes. In the case of alumina, and for varying grain size distributions, we discuss the link between these macroscopic damage mechanisms and macroscopic strength and ductility. We analyze the effect of the reinforcement pressure on the increase of compressive failure strength and as a mechanism to delay crack coalescence. We conclude the presentation with a grain boundary engineering approach for optimizing macroscopic properties.

BB4.18
Abstract Withdrawn

BB4.19
Rate Dependent Deformation Mechanisms of POSS-filled PVC and Plasticized PVC. Sharon Yu-Wen Soong1, Robert E. Cohen1 and Mary C. Boyce2; 1Chemical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts; 2Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Polymers are known to exhibit strong time-dependent mechanical behavior, as evidenced by rate-dependent elastic moduli, yield strength, post-yield behavior, and failure mechanisms. In different temperatures or frequency regimes, the rate sensitivities of polymers change as various primary and secondary molecular mobility mechanisms are involved. As nanoparticles are incorporated into the polymer matrix, their influence on the microstructure is enhanced, thus offering an opportunity to tailor the rate-dependent mechanical deformation and failure behavior of the polymer, which potentially could enhance the energy absorbing ability of the material. In order to design the deformation and fracture behavior of nanocomposite laminates at high strain rates, it is important to understand the rate-dependent mechanical deformation behavior of polymer nanocomposites. Current research efforts are focused on polyhedral oligomeric silsesquioxanes (POSS) enhanced polymeric nanocomposites. POSS has received much attention due to its hybrid organic-inorganic structure, which consists of a silica cage with organic functional groups attached to the cage corners. Methacryl-POSS (C₇H₈O₃S₄Al) is incorporated into poly(vinyl chloride) (PVC) through a melt blending process using a lab-scale extruder. Uniform polymer nanocomposites with different POSS contents are prepared from the melt blending process, which are then compression molded into circular disks of various thicknesses for mechanical and thermal analyses. Diocyl phthalate (DOP) plasticized PVC is also prepared using the same method as a comparison to POSS-filled PVC. It was found that the indentation loading/unloading rate dependence of the POSS nanocomposites is stronger than that of PVC. The indentation loading/unloading rate dependence of the POSS nanocomposites is stronger than that of PVC and an increase in POSS content was observed. The rate-dependent behavior of PVC/POSS and PVC/DOP is then characterized by the material yield strength in compression tests. Zwick Mechanical Tester is used for low to moderate strain rates (10⁻⁴ to 10⁻⁴/s) and Split Hopkinson Pressure Bar is used for high strain rates (500 to 3000/s). It was found that PVC with POSS shows a delay in yield strength rate-sensitivity transition, which is consistent with the observation in the 𝛽-transition and increased rate-sensitivity transition in yield strength faded away. In the near future, the possibility of obtaining stress-strain data in intermediate strain rates regime will be performed to cover a wider range of frequencies, which will provide a good reference to the DMA data. Other nanofillers such as nanoclays and carbon nanotubes are also being considered in polymeric systems.

BB4.20

Ceria is one of the electrolyte material candidates for the intermediate temperature operation of solid oxide fuel cells (SOFCs). During the operation of SOFCs, the partial pressure of oxygen varies across the electrolyte, which induces lattice defects such as oxygen vacancies in the material. This presentation discusses the effects of heat treatments in a high oxygen partial pressure environment on the mechanical properties of nanocomposites. Nanindentation was employed for measuring the intrinsic elastic modulus and hardness of the material. Bulk elastic modulus and fracture toughness were evaluated using compression and Brazilian disc testing methods, respectively. It is shown that the elastic modulus reduces significantly with increasing the defect concentration, however the toughness and the hardness follow different trends. The financial support by DOE under contract DE-FS26-02NT41562 for conducting this research is greatly appreciated.

BB4.21
Influence of Size Effect on Strain Rate Sensitivity and Creep in Plasma-Enhanced Chemical Vapor Deposited Silicon Oxide Films. Zhijiang Cao1, Tong-Yi Zhang1 and Xin Zhang1; 1Manufacturing Engineering, Boston University, Brookline, Massachusetts; 2Department of Mechanical Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Kowloon, Hong Kong.

Plasma-enhanced chemical vapor deposition (PECVD) silicon-based oxide(SiOx) films have been widely used in both microelectronics and MEMS (MicroElectroMechanical Systems) to form electrical and/or mechanical components. In particular, we are interested in its applications as the heat/electrical insulation layers in Power MEMS, e.g., microscale heat engines and related components. In these applications, time-dependent mechanical properties of the material, such as strain-rate sensitivity and creep etc. are crucial design parameters. Especially, whether these properties are influenced by size scale need to be investigated. In this paper, as a first step toward better understanding of these properties, we systematically studied the influence of size effect on the strain rate sensitivity and creep of the PECVD SiOx films at room temperature by using the nanoindentation method. The tests were conducted on a Hysitron TriboIndenter nanoindentation system equipped with 3 different types of tips: 1) Berkovich (sharp) tip, which has a nominal radius of 100 nm; 2) 5mm tip, with a radius of ~5mm and 3) 20mm tip, with a radius of ~20mm. Load/unload nanoindentation is employed and the indentation loading/unloading rate ranges from 50 to 2500 m/s, corresponding to a loading/unloading time of 4~100 s. The thermal drift was less than 0.05 nm/s. Our experimental results demonstrated that the indentation with the sharp tip led to more pronounced plastic deformation during the loading at the same high loading rates. Due to the high indentation velocity, it is believed that the effect of time-dependent processes is minimal, and the differences showed an elastic-plastic-like rate-independent response of the material. In addition, with a sharp tip (~100 nm radius), a pronounced rate-effect was observed for as-deposited PECVD SiOx films. As the loading/unloading rate decreased, the load vs. indentation depth curves appeared to shift toward larger total displacements. However, with larger nanindentor tips, such as 5mm and 20mm, the rate sensitivity was much reduced, and the differences among the curves are not significant. Similar influences were also observed on the creep phenomena. Nanoindentation results on as-deposited PECVD SiOx films with a sharp tip produced a much more pronounced creep than those obtained with blunt tips. After rapid thermal annealing at 800°C for 30 min, however, creep was significantly reduced for all cases. Similar effect has been reported by Suresh et al. for Nano-crystalline Ni, who attributed the effect to the larger volume fraction of grain boundary affected material in the nanocrystalline sample. We believe the physical origin of the enhanced rate sensitivity in our sample is fundamentally different than that of n-Ni because PECVD SiOx is amorphous. A microstructure-based mechanism was proposed to explain the experimental results.

BB4.22
The Role of Multiple Polytypes in Determining the Catastrophic Failure of Boron Carbide at High Shock Velocities. Giovanni Fabbroni and Manish Chhowalla; Materials Science and Engineering, Rutgers University, Piscataway, New Jersey.
The absence of a plastic phase in boron carbide and its failure at shock impact velocities just above the Hugoniot elastic limit (HEL) has been a puzzle for a long time [1]. Also the location of the B and C atoms in the lattice and, hence, its exact microstructure is not well understood, despite the x-ray data show it to be formed by 12-fold icosahedra [[B12]C2B12] and 3-fold icosahedra [CB12] in the 120 GPa work, using self-consistent field density functional simulations [2,3] we are able to account for many experimental observations by noticing that several boron carbide polytypes [B12]C2B12, [B12]C2B12, etc... coexist without significant lattice distortions. Our analysis also indicates that above a threshold pressure all the candidate microstructures are less stable than a phase involving segregated boron and amorphous carbon (c-B). The energetic barrier between boron carbide and borazine [12-B2 + 4C2H6 = 3CaH2 + 2CO2 + 3H2O] is 7.6 eV per atom [4], requiring the lowest atomic displacement for a transformation B4C->3B+C-a-C, occurring at pressures of 5.5GPa = P(HEL). For such a configuration, segregation of a-C occurs in layers orthogonal to the lattice direction and recent with transmission electron microscopy (TEM) analysis [4], [1]


BB4.23 A Study of Damage in Amorphous Silica Using Experiments and Simulations. Cindy Lynn Rountree, Mehdi Talanai, Daniel Bonamy, respectively, and a member of the silicon quantum dot group.


Quasicrystalline materials are, as a rule, drastically brittle at low and intermediate temperatures. A serious drawback for industrial applications. Typically, plastic deformation only takes place at temperatures above 70% of their melting temperature, for a deformation rate of 10^-5 s^-1. Among them, Al-Pd-Mn is certainly the most investigated alloy in particular since high quality quasicrystal specimens can be readily produced. It has been established that, in this quasicrystalline alloy, high temperature plasticity may be ascribed to dislocation movements. In addition, its very high brittle-to-ductile transition temperature strongly suggests that diffusion processes are requested for plastic flow. It is therefore of prime importance to investigate AlPdMn plasticity at low temperature, in particular to establish if plastic deformation can occur in this quasicrystalline alloy without the occurrence of diffusion. Techniques commonly used to investigate plastic behaviour of brittle materials below their brittle-to-ductile transition (BDT) temperature are micro-indentation and deformation under confining pressure. In the present work, we have used several types of the latter technique (multi-anvil, Paterson press), which consist to apply to a specimen a deviatoric stress in addition to a confining pressure. These techniques have been successfully applied to plastically deform AlPdMn quasicrystal specimens in the temperature range 300K - 900K, i.e. below and above the BDT temperature respectively. The microstructures have been examined, prior to and after a plastic deformation, by means of a field emission SEM and by transmission electron microscopy (TEM) operating at 200 K. At all deformation temperatures, the dominant feature of dislocation microstructures is the identification of dislocation climb events. Pure dislocation glide is predominantly observed at room temperature, but its occurrence rapidly decreases with increasing temperature. Dislocation climb appears to be an easy process in this non-periodic structure. The occurrence of climb at such low temperatures addresses questions concerning vacancy concentration and diffusion process in quasi-periodic structure. Nevertheless, all deformation temperatures, it does exist dislocation configurations which arrangements require dislocation glide. Consequently, a glide component is also involved in dislocation movements. The present results confirm the recent idea that glide mechanisms are difficult to initiate in icosahedral quasicrystals. Another characteristic concerns the dislocation shapes that evolve from curled lines at low temperature to segmental shapes at high temperatures. These experimental results are interpreted in the framework of a dislocation model that takes into account the specificity of the icosahedral structure by introducing an evolving extension of the dislocation cores with increasing temperature.

BB4.25 Deformation Mechanisms in Refractory Rare-Earth Phosphates. Randall Hay and Pavel Mogilevsky; USAF, WPAFB, Dayton, Ohio.

Rare-earth phosphates form monazite (monoclinic) and xenotime (tetracapped cubic) crystal structures. They have relatively low density, which makes them good candidates for refractory materials. We characterize fracture, twinning, and dislocation deformation mechanisms by TEM in polycrystalline monazite indented at temperatures between -196°C and 800°C and in monazite fiber-coatings sheared during fiber push-out at room temperature, 500°C, and 800°C. Unusual deformation twinning and climb disassociated dislocation mechanisms were observed. Monazite deformation mechanisms are compared with those observed for xenotime and scheelite. Predictive methods developed for deformation twinning in monazite were applied to tungstates and other rare-earth phosphates. Results are discussed.

BB4.26 Non-Linear Deformation of LaCoO3-Based Ferroelastic Ceramics. Johann Mestin, Mari-Ann Einarsrud, Tor Grøndal, Per Erik Vullum and Randi Holmestad; Materials Science and Engineering, NTNU, Trondheim, Norway; Physics, NTNU, Trondheim, Norway.

Ceramic materials are usually brittle and deform elastically under stress. Ferroelastic ceramics are rare exceptions and these materials respond non-elastically to mechanical stresses. A transition from a high temperature high symmetry paraelastic phase to a low temperature low symmetry ferroelastic phase introduces two or more domain states in the low temperature phase and reorientation of the ferroelastic domains cause the nonlinear stress-strain behavior. Here, we report on the ferroelastic properties of La1-xCaxCoO3 (A= Ca,Sr) materials. La1-xCaxCoO3 (A= Ca,Sr) materials undergo a phase transition from a paraelastic cubic perovskite structure to the ferroelastic body-centered tetragonal structure space group Pn3m at high temperatures to a ferroelastic rhombohedral structure with space group R-3c. High temperature X-ray diffraction of La1-xCaxCoO3 (A= Ca,Sr) materials have shown that the phase transition is reduced down to ambient temperature. Joel, M. Miličić and M. Antonioli, Nature, 1999, 399, 181, and the substitution of Ca and Sr have lead to establish different structural properties due to the significant different ionic radii of Ca and Sr. Reduction of the valence state of Co leading to oxygen deficiency of the materials is also becoming important at high substitution levels and correlated temperature, particularly to the case of LaCoO3 in the BaCoO3. Mechanical stress-strain behavior of La1-xCaxCoO3 (A= Ca,Sr) materials have also been studied under compression. A hysteresis in the stress-strain relationship due to reorientation of ferroelastic domains (deformation to strain) is confirmed and the permanent strain is measured after unloading. The cohesive stress, defined as the
maximum in effective elastic compliance during first loading, increases with substitution level and decreases with increasing temperature. Domain reorientation and the material's mechanical behavior are further constrained by in-situ X-ray diffraction using synchrotron X-ray diffraction during compression/decompression. Domain reorientation is observed continuously up to about 1 GPa. Domains with the c-axis parallel to the stress axis have to be thermally most stable due to the cryostat orientation during since the unit cell is compressed along the c-axis compared to the cubic cell. In the direction parallel to the stress field (220) reflection vanished with increasing stress, while the intensity of (006) reflections increased with increasing stress. In the stress axis the opposite trend was observed and the intensity of (006) reflection was strongly depressed and the (220) reflection enhanced with increasing stress. The relationship between the cohesive stress and the Ca/Sr substitution level is finally discussed.

**BB4.27**

Mechanical and Thermal Properties of LaGaO3 Single Crystals. Siddhartha Pathak1, Surya Kalidindi, Nina Orlovskaya1, Christine Klemens2, Miladin Radovic3 and Edgar Lara-Curroz.3

LaGaO3 based perovskites are a new promising material as an electrolyte for SOFC. While a significant amount of work has been done to characterize ionic conductivity and structure of these materials, there are only a few reports on the mechanical properties of lanthanum gallate based ceramics. Furthermore, the mechanical properties reported in the literature are for the polycrystalline LaGaO3 ceramics. The goal of the present research is to characterize the mechanical properties of the lanthanum gallate single crystals in an effort to understand and quantify the inherent anisotropy of physical properties in single crystalline samples. Single crystals of LaGaO3 grown by Czochralski technique in the [100] and [001] direction were used to perform micro and nanoindentation as well as measurements of thermal expansion. Single crystals of LaGaO3 in the [100] direction were well defined twins. Thermal expansion measurements show a strong shrinkage of the LaGaO3 single crystal in the [100] direction and an expansion in the [001] direction during heating at 1450°C. This transition is reversible and the [100] and [001] direction exhibit expand and contract, respectively, on heating. This can explain the small volume change in these single crystals around the (orthorhombic to rhombohedral) phase transition temperature of 1450°C. In the present study of the phase transition using Raman spectroscopy, an intermediate phase has been detected that has not been reported before. The values of hardness and fracture toughness, measured by Vickers indentation technique, are reported to be in the range 8-9 GPa and 0.3-0.5 MPam1/2 respectively for both the crystal directions. Raman spectra taken from the inside of the impressions show the existence of LaGaO3 rhombohedral structure which indicate that orthorhombic phase transformation to rhombohedral takes place at 110°C and 300°C in the [001] and [001] planes in LaGaO3 differ in their yield stress and modulus values (~160 GPa in the [100] direction and ~110 GPa in the [001] direction) as calculated from nanoindentation load-displacement curves. The same trends are observed for indentation load-displacement curves obtained by nanoindentation using spherical diamond indenters. A new definition for nanoindentation strain (total penetration depth of indentation divided by contact radius) was used for these calculations. This method was found to be consistent for indenters of different radii. The penetration of cube corner diamond indenters in LaGaO3 single crystals were recorded by in-situ SEM technique and a formation of lateral and radial cracking along with a squeezing of the materials during the indentation are shown.

**BB4.28**


In this paper we summarize recent progress in modeling of dynamic crack propagation in a silicon single crystal based on a multi-scale multi-paradigm modeling approach consisting of a sequence of hierarchical, thermodynamically based force field training, with subsequent concurrent coupling of reactive and nonreactive potentials of different levels of accuracy. This newly developed hybrid multi-scale approach combines a Tersoff potential and an improved ReaxFF force field for silicon derived exclusively from quantum mechanical calculations without any additional empirical fitting. The simulations are carried out within our newly developed Computational Materials Design Facility (CMDF). The CMDF framework is capable of performing multi-paradigm multi-scale simulations, and allows coupling various paradigms ranging from quantum mechanics to continuum scales within a single simulation environment (Buehler et al., 2005). Our modeling suggests that our hybrid ReaxFF-Τersoff approach can reproduce some experimental observations of fracture in silicon, such as crack orientation dependence and dynamic instabilities when the crack driving force is much larger than that needed to create new surfaces. We observe formation of secondary microcracks ahead of the primary crack when the system is heavily overloaded. The results suggest that our first principles based ReaxFF force field framework can be successfully applied to model mechanical properties of materials. We suggest that a similar approach could be applied to many other problems in which there exists a strong coupling between chemistry and mechanical properties, as ReaxFF is capable of properly handling complex chemistry.

**BB4.29**

Insight into the Deformation Mechanisms under a Sharp Contact Loading in Glass by Atomic Force Microscopy. Thomas Rouxel, Larmarn FZC-CNRS 2177, University of Rennes, Rennes, France.

The formation of a permanent indentation under a sharp contact loading at the surface of a material is a complex phenomenon involving both reversible and irreversible matter displacements. At the macroscopic scale, these matter displacements control the mechanical characteristics which are further used to built up a constitutive law accounting for the elastic, plastic and viscous contributions. Soda-lime-silica, silicon oxynitride, chalcogenide, and bulk metallic glasses exhibit tremendous differences regarding the indentation behavior. A detailed analysis of the morphology of the residual indentation is for the Atomic Force Microscopy (AFM), including depth, volume, width, extent of the piling-up process, allows for quantitative estimations of elastic, viscous and densification-flow contributions. AFM appears as a remarkable tool to get insight into the matter flows beneath the indent. Much different deformation mechanisms were brought to light depending on the glass system and Poisson’s ratio seems to play a major both on the effective contact area and on the amount of densification beneath the indenter.

**BB4.30**

Mechanical Cyclic Fatigue and Raman Microscopy Resolved Domain Movement in Lead Zirconate-Titanate (PZT). S. Hazzazi, R. Mauban, J. Jacobs, J. S. September. School of Materials Sci. & Eng., The University of New South Wales, UNSW, New South Wales, Australia.

Cyclic fatigue crack growth under electromechanical loading of PZT may lead to component failure, however, the cyclic fatigue crack growth rates and mechanisms are poorly understood. In this work, cracks are grown in PZT under cyclic mechanical load at 1 Hz. It is found that fatigue crack growth rates follow a Paris power law relationship. Crack growth rates under environmental slow crack growth at constant load are orders of magnitude lower than under cyclic loading, confirming the fatigue effect. Peeling is found to have no significant influence upon crack growth rates. Fatigue crack growth is then considered in terms of extrinsic toughening induced by domain movement in the crack tip process zone. An experiment, in which the stress intensity factor for crack extension is monitored as a function of the time of the unload cycle reveals that negligible reversal of domain movement in the process zone occurs at a frequency of 1 Hz. The expected behavior of the material in the process zone is then analyzed by considering the mechanical stress-strain behavior of the material via cyclic loading of bend bars. It is revealed that plastic strain is cumulative indicating progressive reduction in domain movement with cyclic loading. This is confirmed with X-ray diffraction. Neutron diffraction under bulk compressive mechanical cyclic loading, analogous to one side of the bend bar, reveals domain movement decreases with cycles in a process which may be considered as cyclic mechanical poling. Concurrently, it is observed that the orientation of the domains may be revealed in situ in the Debye-Scherrer spectra from the surface of mechanically or electrically poled samples. It is envisaged that this technique will allow analysis of the domain movement within crack process zones.

**BB4.31**


We have recently identified a huge class of solids that we termed kinking nonlinear elastic, KNE, because one of their important - and in many cases only - deformation mode is the formation of fully reversible dislocation-based kink bands, KBS. In this paper we further claim that most, if not all, solids with c/a ratios > 1.0 - which perforce are plastically deforming - will display kinking behavior. In this paper we present compelling evidence obtained from simple...
compression and nanoindentation experiments that KNE solids include most layered solids, some of which, like the layered silicates are geologically important, Mg, Ti, Zn, as well as sulphur, ZnO and AlN, among many others. The ramifications of these results are far-reaching. First, the hysteresis units invoked to explain the behavior of nonlinear mesoscopic elastic solids in geology - have been identified as including KBEs 2 Second, they elucidate, for the first time, why graphite responds to stress the way it does; a 50 40 year old problem. 3 Third, if the factors that result in the nucleation of KBEs are identified and manipulated to significantly reduce the stress at which they nucleate it may be, in principle, possible to reduce otherwise brittle solids with some limited plasticity; an exciting possibility. Lastly given the diversity and ubiquity of KNE solids it is clear that KBEs play a much more important role in our daily life than has hitherto been appreciated. 1 M. W. Barsoum, T. Zhen, S. Kalindiri, M. Radovic & A. Muruganah, Nature Materials, 2, 107-111 (2003). 2 M. W. Barsoum, A. Muruganah, S. R. Kalindiri and T. Zhen, Phys. Rev. Letters, 92, 235508-1 (2004). 3. M. W. Barsoum, A. Muruganah, S. R. Kalindiri and Y. Gogotsi, Carbon, 42, 1435-1440 (2004).

SESSION BB5: Deformation in Semiconductors
Chairs: Sergey Kuchev and James S. Williams
Wednesday Morning, November 30, 2005
Liberty (Sheraton)

8:30 AM *BB5.1
Effect of Thin Films on the Micromechanical Properties of Silicon. Brian B. Law2, Materials Science and Engineering Laboratory, NIST, Gaithersburg, Maryland.

Silicon remains the principal material component of MEMS and NEMS devices. These devices can be fabricated in near-defect-free form with strengths well in excess of 1 GPa. However, real devices are subject to submicroscopic damage under long-term operating conditions, especially from small-scale contacts. An important issue is the retention of strength during the lifetime of the device as small-scale flaws evolve in the structures. Another important issue is how strength is influenced by the presence of thin films on the silicon surface. The present study uses nanoindentation to introduce controlled flaw sets and investigate their interaction with oxide and nitride surface films. Strength tests are conducted by bonding the indented plate surfaces to a polycarbonate substrate, and then applying a concentrated axial load at the top surfaces to induce fracture from the indentation sites. The measured strengths for pristine silicon systematically increase with decreasing flaw size, the increase becoming more rapid below an indentation threshold at which well-developed indentation pre-cracks become suppressed. The strengths for silicon with oxide films are greater than the overall indentation load range, but especially in the subthreshold region. Conversely, the strengths for silicon with nitride films are diminished over the load range. These trends are consistent with the existence of compressive surface residual stresses in the oxide and nitride. A simple fracture analysis is used to quantify the residual stress levels, and the role of film deposition conditions is examined.

9:00 AM BB5.2
Nanoindentation Studies on Silicon and MgO. Bernard J. Hocke,2 Renato Machado3 and Jean-Pierre Guin4.
1Ceramics, NIST, Gaithersburg, Maryland; 2Nat. Inst. of Metrology, Standardization and Industrial Quality, Rio de Janeiro, Brazil; 3MSEL, NIST, Gaithersburg, Maryland.

The nanoindentation behavior of various materials at low loads is currently being investigated with the immediate goal of determining the initial stages of material response to these elastic-plastic contacts. Results relating to the indentation behavior of (111) Si and (001) MgO at loads of 0.1 mN to 5 mN using a Berkhovich indenter are presented. Plane-Section Transmission Electron Microscopy (TEM) observations are used to describe the residual indentation structures produced in MgO at loads down to 0.1 mN and to describe the elastic-plastic response (i.e., indentations, phase transformations and fracture) of silicon at loads down to 0.25 mN. The TEM results, together with associated Atomic Force Microscopy (AFM) images of indentations and the indenter tip, are directly related to the initiation of plastic deformation in these materials and, more generally, to the elastic-plastic response seen in acquired load-displacement plots.

9:15 AM BB5.3
In-situ electrical measurements of phase transformations in Si during nanoindentation. Jodie E. Bradby, Simon Ruffell, Bianca Haberl and James S. Williams; Electronic Materials Engineering, The Australian National University, Canberra, Australian Capital Territory, Australia.

Nanoindentation-induced phase transformations in Si have been well studied in recent years using a variety of techniques including Raman microscopy, acousto-ultrasonic spectroscopy, and transmission electron microscopy. However the majority of these studies are ex-situ and do not provide any detailed information regarding the nature of the phase transformations during the deformation process. In this study we have exploited the fact that the electrical properties of Si change dramatically during indentation. From the starting point of a semiconductor, silicon is well known to transform to a metallic state on loading, and to either a semi-metallic crystalline phase or an insulating amorphous phase following pressure release. In this study we have measured the electrical properties of silicon during nanoindentation using two methods. The first method relies on the switching of the properties of deposited surface contacts from Schottky to Ohmic as the underlying silicon is transformed into the metallic state. This method has the advantage of requiring little additional instrumentation aside from a voltage source and current meter. However it was found that the sensitivity of the measurement decreased on the unloading cycle of each sample as a result of the particular experimental conditions. The second method uses a new in-situ electrical probe where the tip is utilized as both the mechanical and electrical probe by engineering conducting indenter tips (Berkovich) from artificially grown doped diamonds. A bias is applied between the tip and the sample and the resulting current flow measured during indentation. This measurement is sensitive to changes in the resistivity in the indentation zone and/or changes in the barrier potential across different material phases in this zone. Interestingly, such electrical measurements are sensitive to structural changes throughout the entire unloading curve.

9:30 AM BB5.4
On the nucleation of dislocations in silicon and the relation with the brittle to ductile transition. Jacques Rabier and Jean-Luc Demenet; Laboratoire de Metallurgie physique, UMR 6630 CNRS-Universite de Poitiers, Futuroscope, France.

Deformation experiments have been conducted under high confining pressure (5 GPa) on PZ silicon single crystals in order to pass the unloading brittle to TEM transition. TEM observations of these deformed crystals provide evidence for the nucleation of different type of dislocations depending on stress and temperature domains. In the low temperature high stress domain perfect shuffle dislocations are nucleated whereas dissociated glide dislocations are nucleated at lower stresses and higher temperatures. In situ TEM annealing experiments failed to provide any evidence of core transformation from shuffle type dislocations into glide ones. Another striking feature of the high stress deformation substructures is the zigzag configurations shown by perfect shuffle dislocations. This demonstrates the existence of very strong pinning points for these dislocations as compared to glide ones. Such deformation microstructures and dislocation core configurations will be discussed in relation with the brittle to ductile transition of silicon.

9:45 AM BB5.5
Nano-abrasive Wear Behavior of Silicon. Hong Liang,1 Alan F. Schwartzman2 and Christopher A. Schuh3; 1Mechanical Engineering, Texas A&M University, College Station, Texas; 2Materials Science and Engineering, MIT, Cambridge, Massachusetts.

Abrasive wear at the nanometer length scale was investigated for both amorphous and single crystalline (001) silicon, via nanoindentation and nanoscratch experiments. Characterization of the deformed surface region was also conducted using atomic force microscopy (AFM). The results illustrate the difference in behavior between crystalline and amorphous silicon under contact loading. In particular, where amorphous silicon exhibits relatively smooth and stable nano-scratch behavior, crystalline silicon shows serrated, stick-slip type abrasion at the nanoscale level. Furthermore, comparison of in situ (instrumented indentation and scratching) and ex situ (AFM) normal displacement data reveals some key differences in the deformed surface condition of the two materials. Relaxation is observed at the bottom of the amorphous Si trenches, but not for crystalline Si. Quantification of this effect will be presented.

10:30 AM *BB5.6
Cracking During Nanoindentation of Silicon and Germanium and Its Relation to Fracture Toughness. George M. Pharm and Jee-Ill Kang; 1Dept of Materials Science and Engineering, University of Tennessee; Knoxville, Tennessee; 2Materials Advances, Oak Ridge National Laboratory, Oak Ridge, Tennessee; 3Division of Materials Science and Engineering, Hanyang University, Seoul, South Korea.

Cracking during indentation has long been used to measure the fracture toughness of ceramics, most often with a microhardness tester equipped with a Vickers square-based pyramidal indenter and at loads in the range 10-10,000 g. The same techniques can be used in
nanoindentation at much smaller loads and size scales, but two significant problems are encountered. First, the square-based Vickers pyramidal indenter is known to induce a loss of geometrical similarity for indentations with characteristic dimensions on the order of a micron or so. Geometrical similarity is crucial in the interpretation of results. Second, there are threshold loads, typically in the range 10-100 g, below which no cracking occurs in many ceramic materials indentated by a Vickers indenter. These problems can be circumvented by testing with sharper indenters with the triangular pyramidal geometry, but the relevant mechanics of elastic-plastic contact is not well understood. In this study, the effect of indenter sharpness on the indentation cracking behavior of two brittle materials - silicon and germanium - was investigated for a series of triangular pyramidal indenters. The sharpness of the indenters is characterized by a single value - the contact radius - which was varied from 35 to 86 degrees. The influences of indenter angle on cracking thresholds and the relationship between the crack length and indentation load are documented and discussed in terms of prevailing theories and models. *Research supported by the National Science Foundation under grant number DMR-0203552, and by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy, SHaRE Collaborative Research Center at Oak Ridge National Laboratory, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.*

**11:00 AM BB5.7**

Contact-induced damage in ZnO epitaxial films and single crystals: implications for device manufacture.

Victoria Coleman¹, J. E. Bradby¹, C. Jagadish³, M. R. Phillips², M. V. Swain¹ and F. Munroe¹

¹Department of Electronic Materials Engineering, The University of New South Wales, Sydney, New South Wales, Australia; ²Interdisciplinary Materials Science Centre, Capital Territory, Australia; ³Microstructural Analysis Unit, The University of Technology, Sydney, Broadway, New South Wales, Australia.

The effect of contact-induced damage on both ~500 nm thick ZnO epitaxial layers grown on sapphire, and single crystal ZnO and has been studied using nanoinindentation, atomic force microscopy, cross-sectional transmission electron microscopy (XTEM), and scanning cathodoluminescence imaging. Nanoinindentation was conducted using a spherical indenter with a radius of ~4–5 μm, on both a- and c-axis oriented samples. Comparison of the indented samples revealed significant differences in the mechanical behaviour of all systems. For single crystal ZnO, a-axis material was found to be ~2.5 GPa softer than c-axis material. This is attributed to differences in dislocation propagation arising due to the orientation of the basal (primary slip) planes. For epitaxial ZnO, both orientations exhibited higher modulus and hardness values than their single crystal counterparts. XTEM suggests that the presence of as-grown dislocations and the properties of the underlying sapphire substrate confine the indentation-induced plastic deformation and inhibition of subsequent long-range defect propagation in the epitaxial layers. Indentation-induced plastic deformation can influence the properties of the metal-semiconductor interface and can affect the performance of devices. In all instances, XTEM of the spherical indent shows no evidence of cracking, delamination or phase transformation in samples loaded up to 50 mN. The results of this study have potential implications for the fabrication and processing of ZnO epitaxial layers-based (opto)electronic devices.

**11:15 AM BB6.8**

The Brittle to Ductile Transition in Undoped GaAs.

Shanling Wang¹, Ming Zhang¹, Pirouz Pirouz¹ and Jodie Bradby²

¹Department of Materials Science and Engineering, Case Western Reserve University, Cleveland, Ohio; ²The Australian National University, Canberra, Australian Capital Territory, Australia.

It is well-known that dislocations in compound semiconductors have different atomic cores (so-called α and β dislocations) giving them different velocities and possibly different nucleation activation barriers. Conventionally, below which no cracking occurs in many of the mechanical properties of compound semiconductors is significant. In this work, two techniques were employed to investigate the transition from brittle to ductile behavior in undoped GaAs single crystals. The 4-point bending test and the indentation test on precracked samples of GaAs to measure its brittle-to-ductile transition (BDT) temperature, T_BDT, at different strain rates. The BDT temperature of GaAs was found to be sharp to within ~2.5°C, to increase with increasing loading rates, and the relationship between the BDT temperature and strain rate was found to be a straight line with an activation enthalpy proportional to that for dislocation glide in GaAs. Surprisingly, it was found that the activation enthalpy obtained in this way corresponds to that for the glide of slow β dislocations rather than of the fast α dislocations. This result is consistent with a model recently proposed for the brittle-to-ductile transition temperature in tetrahydrofuran-coated materials. In addition to the BDT tests, a high-temperature displacement-sensitive indentation (DSI) machine was used to perform DSI tests from room temperature to 400°C. Over the entire temperature range, the load-displacement curves were consistent with that of a martensitic-type plastic deformation. From such plots, the temperature dependence of the energy E, spent in producing the indent was extracted and the energy density was estimated. Interestingly, while E increases continuously with temperature & increases continuously with temperature, T_BDT decreases with further increase in temperature. The BDT temperature measured by these two methods are compared and interpreted in terms of the presence of a hydrostatic component in the indentation mechanism, causing a lowering of the BDT temperature. Finally TEM of the deformed GaAs crystals in the brittle and ductile regimes will be briefly presented and discussed.

**11:30 AM BB6.9**

Indentation Response of Wall-patterned (001) GaAs Surfaces.

Eric Le Bourhis¹ and Gilles Patriarche²,³

¹Laboratoire de Metallurgie Physique, UMFR 6550 CNRS, Université Poitiers, Futuroscope-Chasseneuil Cedex, France; ²Laboratoire de Photonique et de Nanostructures, UPR 20 CNRS, Marcoussis, France.

Plasticity of III-V semiconductors has received much attention during the past two decades because of the needs of the optoelectronic industry. In this field, the indentation technique has proved to be a powerful tool to test small volume even at temperature below the brittle-ductile transition point. For so far, contact mechanics have been developed for semi-infinite half space, this assumption being not fulfilled when the size of the plastic zone becomes of the order of one of the dimensions of the object. Therefore, thin structures are expected to show a mechanical behaviour quite different from that of a bulk [2-4]. Wall-patterned GaAs surfaces have been elaborated by photolithography and dry etching. Different surfaces were produced in order to change the aspect ratio of the walls, that is the ratio of the substrates to the substrate surface. The mechanical behaviour of individual walls was investigated by nanoindentation and the responses were compared to that of a standard bulk reference (flat surface). Deviation from the bulk response was detected in a load range of 1-25 mN depending on the structures. The application of substrate patterning for optoelectronic devices is discussed in the perspective of eliminating residual dislocations appearing in mismatched structures. [1] Le Bourhis E., Patriarche G., Proc. Cryst. Growth Charac. Mater., 2003; 47, 1 [2] Choi Y., Suresh S., Scripta Mater 2003; 48: 249 [3] Largeau L, Patriarche G., Le Bourhis E, Riviere A, Riviere JP, Phil Mag 2003; 83 : 1653 [4] Uchic MD, Dimiduk DM, Florando JN, Nix WD. Science 2004; 305, 986.

**SESSION BB6**

**Environmental Influences and Direct Imaging of Deformation**

Chairs: Jodie Bradby and Robert Cook

Wednesday, November 30, 2005

Liberty (Sheraton)

**1:30 PM **BB6.1

Effect of Temperature on Response of Semiconductors to Indentation.

Yury Gogotsi and Vladimir Domnich

Materials Science and Engineering, Drexel University, Philadelphia, Pennsylvania.

The stress induced by indentation may lead to amorphization and phase transformation of hard and brittle semiconductors. Plastic deformation and fracture often occur simultaneously with phase transformations. Understanding and appreciation of phase transformations induced by contact loading is important for correct interpretation of data produced by depth-sensing indentation [1]. While the deformation and fracture mechanisms of semiconductors at room temperature have been extensively studied, limited information exists for elevated temperatures. It is particularly important to know at what temperature a transition from phase transformation to plastic deformation occurs in brittle semiconductors. A combination of depth-sensing indentation and Raman spectroscopy is a powerful tool for studying phase transformations, damage and residual stresses induced by contact loading. The paper will describe phase transformation and amorphization that occur in semiconductors, such as silicon and germanium. The effect of phase transformation and plastic deformation on the shape of the indentation load-displacement curves will be discussed. The hardness of silicon is known to be nearly
independent on temperature below a certain transition point, and to decrease steeply thereafter. Using Raman microanalysis of Vickers and Berkovich indentation-produced single-crystal silicon at 350°C, we present evidence of a transformation into a high-pressure metallic Si phase during indentation at temperatures under 350°C. We show that this transformation pressure determines silicon hardness below the transition temperature. We also report the temperature stability ranges of different metastable phases of silicon. Reference 1: High Pressure Surface Science and Engineering, Ed. Y. Gogotsi & V. Dornich, Institute of Physics Publishing, Bristol, UK, 2004

2:00 PM BB6.2
In-Situ Electron Microscopy Observations of Nanoscale Size Effects on the Deformation Pathway of Silicon. Diabn Ge1, Andrew M. Warrn2, Martin Morris3, and Eric A. Stach1; 1School of Materials Engineering, Purdue University, West Lafayette, Indiana; 2National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California; 3Materials Science and Engineering, University of California, Berkeley, California; 4Hysitron Incorporated, Minneapolis, Minnesota.

Application of the technique of nanindentation to elemental and compound semiconductors has generated renewed interest in the deformation response of these conventionally brittle materials to large loads. Complex deformation pathways have been observed in many of these systems, with the exact nature of the deformation response depending on the details of the experimental setup, including such aspects as loading rate, indenter tip shape, dwell time at maximum load and sample temperature. With this presentation, we will focus on the addition of size to sample size and discuss its effects on the deformation response of silicon. Our initial experiments focused on real time observations of deformation during indentation of wedge shaped plates of variable size within the transmission electron microscope. We find that as the sample size present under the indenter radius is altered, the movement and shape of dislocations interact, resulting in a significant increase in the force of deformation. This effect is enhanced as the sample size present under the indenter radius increases. This behavior is attributed to the kinetics and dynamics of deformation processes in the Si crystal, which is the major constituent of the two-phase alloys. Deformation of gamma-TiAl occurs under most conditions on {111} planes along close-packed {110} directions and can be provided by ordinary superdislocations. In the gamma phase of two-phase alloys, glide of superdislocations is difficult because these dislocations are liable to adopt non-planar dislocations and are subject to high Peierls stresses. Thus, there are many more restrictions upon possible dislocation modes in gamma-TiAl than for dislocations. As recognized in literature (e.g. Simmons, 1974), deformation of gamma-TiAl can also be provided by mechanical twinning. Unlike disordered face centered cubic (fcc) metals, there is only one distinct twinning shear direction per {111} slip plane that does not alter the structure. Nevertheless, the mechanism may support plastic deformation in that it provides auxiliary slip systems. Thus, mechanical twinning plays an important role in alloy design strategies for mitigating the problems associated with high-temperature, high-stress conditions. The data collected in literature indicates that our understanding of mechanical twinning of gamma-TiAl is still in many ways imperfect. To some extent this is due to the atomic scale of the processes and the difficulties associated with the development of defect structures and constraint stresses that are developed in heavily twinned material. This imbalance of information is addressed in the present study by a high-resolution transmission electron microscopy study of twin structures. The primary goals of the study are: (i) twin nucleation and propagation, (ii) effects of solutes and precipitates on the kinetics and dynamics of twin propagation, (iii) association of mechanical twinning and fracture.

3:30 PM BB6.4
Indenter Radius Influence on the Nanoindentation Response of BaTiO3. T. Scholz², G. Schneider¹ and Michael Ivanov²; ¹University of Sydney, Sydney, New South Wales, Australia; ²Ceramic Group, TUHH, Hamburg, Germany.

This investigation focuses on the nano-indentation response of a [001] oriented BaTiO3 giant grain using four different indenters, two corner cubes and two conical indenters, with tip radii ranging from 63 nm up to 2.1 μm. For the corner cube indenters two pop-in events associated with elastic-plastic and plastic-brittle response were identified whereas for the conical indenters, elastic-plastic response was evident. The elastic-plastic response of all the indenters was superimposed using the indentation stress-strain concept where the contact pressure was plotted against the contact stress or contact radius by effective indenter radius. The contact pressure after pop-in was found to be indenter radius dependent and the results were analyzed using the approach of Swadener et al (J. Mech. Phys. Solids, 50, 681 (2002)). The deformation associated with the indentation of the BaTiO3 was imaged using Pulser Free Microscopy and clearly shows twinning as well as domain motion associated with plastic deformation.

3:45 PM BB6.5
Microstructure Characterization of Shockless to Shocked Transition in Single Crystal Copper using Laser Based and High Explosive Based Platforms. Jennifer Szostak Harper¹, J. M. McNeury², N. E. Teslie³, V. A. Roevskiy³ and D. A. Remington³; ¹Lawrence Livermore National Laboratory, Livermore, California; ²Russian Federal Nuclear Center, All-Russian Institute of Experimental Physics, Sarov, Russian Federation.

Two platforms, which have been developed to produce high strain rate, high pressure quasi-isentropic loading, have been used to deform and recover single crystal copper. A quasi-isentropic pressure wave is induced in the sample by means of a laser or high explosives. As the wave propagates through the material it steepens and eventually becomes a shock. The primary difference between these two loading path is a change in strain rate. Both platforms have been used to investigate the response of <100> oriented single crystal copper loaded up to a peak pressure of 200 GPa. The deformation mechanism occurring in the shockless and shocked region has been investigated using transmission electron microscopy. The residual microstructure between the shockless (dislocation cells) and shocked (stacking faults) material are compared, as well as between the laser based and high explosive based platforms. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

4:00 PM BB6.6

PECVD organosilicate glass (OSG) is currently used as an interlayer dielectric in advanced integrated circuits. OSG has good dielectric properties, but has a low fracture toughness making it susceptible to cracking and delamination, especially in chemically active environments. In this paper, we investigate the link between the mechanical behavior of OSG films and their structure. We present the results of a systematic study of a series of PECVD OSG films deposited using octamethylcyclotetrasiloxane (OMCTS) as a precursor. We have determined the composition of the coatings as a function of the OMCTS flow rate using Rutherford backscattering spectroscopy (RBS) and forward recoil spectroscopy (FRES). The results show that the composition of the coatings is determined by varying the OMCTS precursor flow rate. The chemical bonding structure of the films was analyzed using FTIR spectrometry. Young's modulus of the films was measured as a function of OMCTS flow rate by means of the bulge test and fracture toughness using the four-point bend test. Incorporation of H and C species into the Si-O network lowers the stiffness of the material and reduces the fracture toughness through formation of terminal groups like H-Si and CH3-Si. Other bond configurations, such as Si-C2H5-Si and Si-O-C, form part of the network replacing bridging O or Si. Based on the analytical results, we have developed a detailed structural model that includes the main types of bonds found in OSG. Young's modulus and fracture toughness of the OSG2 are well correlated with the anti-terminal (including H-Si and CH3-Si) to networking bonds (including Si-O, C-O and Si-CH2). The structural model further allows determination of the correlation coefficients between FTIR absorbance and bond density, information not readily available in the literature. These
coefficients make it possible to calculate bond density directly from FTIR absorption spectra of similar materials. Fracture of these materials is greatly accelerated by the presence of chemically active species in the environment, a situation often encountered during fabrication and processing. We will present results on the effect of common species such as water and hydrogen peroxide on the fracture behavior of OSG coatings in light of the structural model.

4:15 PM BB6.7
Real-time observation of liquid condensate confined at tensile crack tips in silica glasses. Lothar Wondraczek1, Fabrice Celarie1, Anne Dittmar1, Carina Oelgart1, Matteo Cicotti2 and Christian Marlino2; 1Institute of Non-Metallic Materials, Clausthal University of Technology, Clausthal-Zellerfeld, Germany; 2University of Montpellier II, Montpellier, France.

The occurrence of hydroxyl liquid condensate between the two fracture surfaces in the vicinity the tip of tensile cracks in silica at elevated humidity is observed in real-time by means of atomic force microscopy. Sub-critical cracks are generated in double cleavage drilled compression experiments. The crack speed is controlled continuously in the regime of stress corrosion. Condensate formation is demonstrated here for macroscopic relative humidities around 40% in nitrogen atmosphere. Its shape and extent are dependent on relative humidity and humidity variations, and crack speed. The study of AFM phase-contrast data indicates an liquid hydroxyl character of the condensate. Since the crack tip chemistry is still not evident (prior studies were performed at free surfaces, rather than in very confined conditions), it is therefore of fundamental importance to provide an experimental verification of the presence of liquid water at the tip of a crack propagating in air, and of the conditions for its presence.

4:30 PM BB6.8
Contact Fatigue Loading on Glass in Water. Sanjit Bhownick1, Ija Hermann1, Jae-Won Kim1, Yu Zhang2 and Brian Lawn2; 1MSEL, National Institute of Standards and Technology, Gaithersburg, Maryland; 2College of Dentistry, New York University, New York, New York.

The appearance of Hertzian cone cracks and radial cracks in contact loading is well documented. Recently, an entirely new form of top-surface inner cone crack has been identified in cyclic contact loading experiments in monolithic brittle materials in aqueous environments. This inner cone crack nucleates inside the indentation compression zone and grows deeply downward into the material. Hydraulic pumping of water within surface flaws during loading is postulated to be the main driving force for initiating the crack. In this presentation, the evolution of inner cone cracks, along with that of potential competing modes (conventional outer cone cracks and radial cracks), will be presented for various layer configurations, with glass as the model top-layer brittle material: glass monolith, glass-poly carbonate bilayer, and glass-alumina-polycarbonate trilayer. These systems simulate essential elements of important biomechanical systems such as dental crowns and hip replacements. In the case of bilayers and trilayers, plate flexing induces tensile stresses at the bottom surface of the glass plate, which enhance the propagation of top-surface cracks towards the interface, resulting in failure. Which fracture mode ultimately fails is determined by the toughness of the materials and the distance to the interface. Top-surface cracks approaching an interface of glass and alumina may arrest or may advance by either penetrating into the alumina layer or deflecting into the interface. The implication of the failure in the context of biomedical applications will be discussed.

4:45 PM BB6.9
Cryogenic Wear of Self-Mated Alumina: A First Report. Rohit Khanna and Bikramjit Basu; Materials and Metallurgical Engineering, Indian Institute of Technology (IIT), Kanpur, Kanpur, India.

Among the potential materials used in rocket turbopumps, martensitic stainless steel (SUS 440C grade) is widely being used as bearing materials in liquid nitrogen (LN2), liquid hydrogen (LH2) or liquid oxygen (LO2) at speeds up to 50,000 rpm [1]. In recent times, some ceramics like Si3N4 are used as hybrid bearing is also tested. While ceramics are considered as candidate materials, the fundamental science behind response of perfectly brittle ceramic (like alumina) to stress under the tribomodel at sub-zero temperature (especially, cryogenic temperatures) is yet to be explored. In the above prospective, the present research is taken up to answer some specific questions: (a) whether liquid nitrogen serves as a lubricant or coolant (via frictional behavior)? (b) How does the fracture of brittle materials take place in cryogenic environment? (c) How does the wear rate depend on the load in LN2 environment? In order to obtain fundamental understanding of the friction and wear properties of ceramics, the sliding wear tests were carried out on self-mated Al2O3, a model brittle ceramic material, in liquid nitrogen (77 K) under varying load (2-10 N) and high rotational speed of 2550 rpm using a newly designed high speed cryogenic tribometer. Our experimental results reveal that self-mated alumina exhibits lower COF ~ 0.13 and suffers high wear rate (10^-5 mm²/N m) under the selected testing conditions. Detailed SEM observation (cleavage steps, grain pullout, fine debris entrainment, deep abrasive grooves and extensive intergranular cracking) of the worn surfaces indicates that severe damage of both ball and flat takes place at cryogenic test conditions by transgranular and intergranular fracture. References: [1] Nosaka, Masataka and Kikuchi, Masataka, trib. trans., 42, 1999, 1, 109-115.