SYMPOSIUM U

U: Thin Films-Stresses and Mechanical Properties X

December 1 - 5, 2003

Chairs

Sean G. Corcoran
Dept. of Materials Science & Engineering
Virginia Tech
Ste. 302
460 Turner St.
Blacksburg, VA 24061-0286
540-230-2616

Young-Chang Joo
School of Materials Science & Engineering
Seoul National University
Seoul, 151-742 KOREA
82-2-880-8986

Neville R. Moody
Sandia National Laboratories
MS 9404
P.O. Box 969
Livermore, CA 94551-0969
925-294-2622

Zhigang Suo
Division of Engineering & Applied Sciences
Harvard University
Pierce Hall 309
29 Oxford St,
Cambridge, MA 08544
617-495-3789

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* Invited paper

SESSION U1: Stress Evolution
Chairs: Neville Moody and Zhigang Suo
Monday Morning, December 1, 2003
Room 210 (Hynes)

8:30 AM U1.1
Mechanical Strain Evolution in Cu/Low K Interconnect Lines. Paul R. Besser[1] and Qing-Ting Jiang[2]; [1]Technology Development Group, Advanced Micro Devices, Sunnyvale, California; [2]Advanced Interconnect, International SEMATECH, Austin, Texas.

Stress migration is an important and challenging reliability issue. In order to better interpret stress migration results, the sources of the stress and the stress evolution in Cu interconnect lines need to be understood during thermal processing for both oxide and low-K materials. These chemical stresses in interconnects arise from thermal expansion (CTE) differences. The magnitude of the stress can be calculated based on the measured strain, using X-Ray diffraction (XRD), and the stress-temperature behavior has been published for Cu/oxide and Cu/low K for both wide and narrow lines. In the current work, the strain (and stress) state of narrow Cu lines fabricated in oxide, CVD Low K and porous organic spin-on dielectrics have been determined with XRD during annealing. It will be shown that the room temperature stress along with length (X) and width (Y) of the lines are not dramatically different while the Z component is somewhat smaller with the spin-on ILD. These small perturbations in the magnitude of the Cu stress do no reflect the dramatic differences in the Young's Modulus and the CTE of the dielectrics. It is proposed that a better metric for understanding the mechanical evolution in Cu interconnects is Cu strain, which is measured directly with XRD. The conversion factor from strain to stress is the Stiffness Coefficients, which can be considered a weighting factor. The strain-temperature data for the Cu lines illustrate the effect of the ILD clearly. Further interpretation of the strain-temperature profiles will be provided, including insight into the evolution of the strain tensor and its implication for strain relaxation mechanisms, and effects of modulus of the ILD on the strain evolution.

8:45 AM U1.2
CGS Interferometry Applied to the Study of Curvature and Stress Evolution in Thin Films/Lines Structure on Si Substrates. Ares J Rosakis[1], Tae-Soon Park[1] and Subra Suresh[2]; [1]Aeronautics, California Institute of Technology, Pasadena, California; [2]Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Coherent gradient sensing (CGS) interferometry is discussed as a means of measuring the non-uniform curvature tensor evolution over the entire wafer surfaces during semiconductor processing. Since this technique measures surface curvatures in a real-time, full-field, and vibration-insensitive manner, it is well suited for in-situ characterization of curvature and stress evolution during processing or thermal cycling. Issues presented include a) the use of CGS before and after film deposition for growth stress characterization, b) its use in conjunction with elaborate stress analysis tools for the inference of stresses in thin film and line structures during thermal cycling. Validation of the proposed methodologies is provided through detailed 3-D finite element analysis.

9:00 AM U1.3
Compressive stress generation in sputter-deposited metal films. Vidya Ramaswamy[1], William D. Nix[2] and Bruce M. Clemens[2]; [1]Division of Engineering and Applied Science, Harvard University, Cambridge, Massachusetts; [2]Department of Materials Science and Engineering, Stanford University, Stanford, California.

Large compressive stresses are often observed in sputter-deposited metal films, due to bombardment by energetic reflected neutrals and target atoms, sometimes called atomic peening. We have measured stress generated during sputter deposition of polycrystalline and epitaxial Pd/Pt multilayers by *insitu* substrate curvature measurement. Epitaxial and polycrystalline Pt layers exhibited similar stress behavior, with large compressive stresses developing at low sputtering gas pressures. In the case of Pd, polycrystalline Pd layers develop compressive stresses, while epitaxial Pd layers do not. These differences in stress behavior suggest that in some cases, grain boundaries are essential for the development of compressive stresses by the atomic peening mechanism. We discuss the differences in stress behavior in terms of the microstructure and surface morphology of Pd and Pt. A model for the incorporation of excess atoms into the growing film, consistent with experimental observations, provides insight into the mechanisms of compressive stress generation, and the importance of various deposition parameters and material properties.

9:15 AM U1.4
Bending and Extension of Copper Thin Film Single Crystals. Masao Doyama, Yoshiaki Kogure, Tadatoshi Nozaki and Yukie Kato; Materials, Teikyo University of Science and Technology, Uenohara, Yamanashi, Japan.

Copper single crystals of thin films were extended or bent. Molecular dynamics were used with embedded atom potential. It was found that old model of bending of single crystals is wrong. Macro-bending and atomic or micr-bending should be distinguished. Macro-bending without containig dislocations is possible, of course. These can be distiguished by X-ray Laue patterns. Dislocations are quite difficult to initiate on very smooth flat surface under tension. It is also difficult to initiate dislocations by pure bending, if the surface is smooth. It is easy to initiate dislocations at the winkeles of slip plane under compression. When a notch is present on a surface dislocations are created near the tip of the notch. The single crystals suddenly became polycrystals under severe pure bending, even at low temperatures. For copper crystals, partial dislocations are always created leaving a stucking fault on one side.

9:30 AM U1.5
Stress Distributions in Polycrystalline Films formed by Oxidation Ramanathan Krishnamurthy and David J. Srolovitz Princeton Materials Institute and the Department of Mechanical and Aerospace Engineering, Princeton, NJ 08540. Ramanathan Krishnamurthy and David J. Srolovitz; Princeton Materials Institute and Dept. of Mech. and Aerospace Engg., Princeton University, Princeton, New Jersey.

The generation of stresses in polycrystalline oxide films formed via the oxidation of a substrate is analyzed using a new continuum model. The model includes a description of the polycrystalline microstructure in two dimensions. The diffusion of all independent components, the rate of the oxidation reaction and the effect of stresses on these are accounted for in a thermodynamically self-consistent manner. Grain boundaries serve both as high diffusivity paths and as sites for oxide formation. Different diffusion controlled oxidation regimes(rapid oxygen/cation diffusion, comparable oxygen/cation diffusivities) and different grain boundary/bulk diffusivity ratios are examined within this framework. A homogenized 1-D treatment captures the correct signs of stresses and through-thickness stress gradients observed in experiments. Numerical solutions of the 2-D polycrystalline situation reveal large lateral stress gradients, with stresses concentrated around the grain boundaries. While the average in-plane stress is compressive and the stress at the film / substrate interface near the grain boundary highly so, large tensile stresses are observed near the grain boundary at the film surface. The grain boundary diffusivity ratio has a significant effect on the stress gradients, with larger diffusivities leading to smaller stress gradients. We also present analytical approximations for the stress distribution in the film that capture the essential features of the numerical results. The predictions are compared with experimental measurements/observations of stresses and stress gradients in oxide scales. The implications of these results for understanding the fundamental nature of self-stresses generated in oxides are discussed.

9:45 AM U1.6
Irreversible Tensile Stress Development in PECVD Silicon Nitride Films. Michael P Hughey and Robert F Cook; Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota.

The thermo-mechanical behavior of plasma-enhanced chemical vapor deposited (PECVD) silicon nitride films is studied during thermal cycling and annealing, simulating temperature excursions experienced by the films during device fabrication. It is well known that PECVD films have large amounts of incorporated hydrogen that evolve from the film on heating (typically above the deposition temperature). We show here that this removal of hydrogen is directly responsible, via constrained volume decrease, for irreversible increases in tensile stress in PECVD films. Experimental evidence shows that irreversible stress development is always tensile and its magnitude is independent of the film stress, indicating that the driving force for stress increase is dominated by a reduction in the chemical energy (the product of the chemical potential and the number of removed species), not a reduction in mechanical, or strain, energy. As such, the thermal cycling behavior of PECVD films can be modeled by chemical reaction theory, with the irreversible development of stress simply a mechanical consequence. Stress-temperature data during thermal cycling and annealing are shown for silicon nitride films and compared to simulation results based on first and higher-order reaction kinetics of Si-H bonds utilizing an activation energy and an Arrhenius temperature dependence. The activation energy is not single-valued, indicative of the strong influence that the local bonding environment has on bond and reaction energies. Silicon-rich silicon nitride is studied as a representative PECVD material that is not sensitive to moisture absorption, that contains mostly one type of impurity (Si-H), and for which stress behavior is commonly found in the literature.

10:30 AM U1.7
X-Ray Microbeam Diffraction Measurements in Polycrystalline Aluminum and Copper Thin Films.
L. E. Moyer[1], G. S. Cargill[1], Wenge Yang[2], B. C. Larson[2] and G. E. Ice[2]; [1]MS&E, Lehigh Univ., Bethlehem, Pennsylvania; [2]Oak Ridge National Lab., Oak Ridge, Tennessee.

Thermally induced residual strains in polycrystalline Al and Cu films on single crystal Si substrates have been examined on a grain-by-grain basis by x-ray microbeam diffraction. The crystallographic orientation and the deviatoric strain tensor, Thermally induced residual strains in polycrystalline Al and Cu films on single crystal Si substrates have been examined on a grain-by-grain basis by x-ray microbeam diffraction. The crystallographic orientation and the deviatoric strain tensor, $\epsilon_{ij}*$, are determined for each grain by white beam Laue diffraction. Monochromatic diffraction measurements and lattice parameters from unstrained crystals are used to determine absolute strains in single grains. The equipment and procedures used have been described by J.-S. Chung, N. Tamura, G. E. Ice, B. C. Larson, and J. D. Budai (MRS Symp. Proc. **563**, 169 (1999)). From grain orientation mapping and strain tensor measurements, information is obtained about the distributions of strains for similarly oriented grains and about the grain-to-grain correlations of strains. This type of information may be useful in developing and testing theories for intergrain effects in strain evolution in polycrystals. Information from x-ray microbeam measurements will be compared and contrasted with strain information obtained from traditional, macrobeam x-ray diffraction measurements.

10:45 AM U1.8
The Effect of Adatoms on Reversible Stress Observations at All Stages During Volmer-Weber Growth of Thin Films.
Cody A. Friesen and Carl V. Thompson; DMSE, MIT, Cambridge, Massachusetts.

In earlier work it was demonstrated that a large component of the compressive stress observed during pre-coalescence Volmer-Weber growth of Cu films reversibly relaxes during growth interruptions. In the current study, reversible stress changes have been characterized during growth interruptions at all stages of Volmer-Weber growth. These studies were conducted using capacitive measurements of cantilever displacements, giving high-sensitivity stress measurements with high temporal resolution. In the pre-coalescence, coalescence, and post-coalescence regimes, the stress changes toward a tensile state during a growth interruption, but returns to the pre-interruption stress state after growth is resumed. In all regimes, the initial rate of increase of the stress-thickness product after growth resumption is large, with initial compressive instantaneous stresses of order several GPa. This rapid initial rise is thought to be associated with the development of an excess adatom population, resulting in the observed compressive surface stress. A series of growth and growth interruption cycles were characterized during deposition of Cu films from zero thickness to 100 nm. It was found that when the films were discontinuous, the initial instantaneous stress was dependent on thickness, while it was independent of thickness after film continuity was achieved. This change in dependency relates to the difference in the adatom-surface interaction for the Cu-adatoms on the substrate (borosilicate glass) and Cu adatoms on Cu islands or grains. As expected, the initial instantaneous stress did not depend on the deposition rate. These results further support the idea that an evolving adatom population contributes to the observed reversible stress. A thermodynamic formalism has been derived to relate the effects of Cu adatoms on Cu surface stress through an interaction parameter that has been determined from experiments as well as through both semi-empirical molecular dynamics and ab-initio calculations. Experiments and calculations are in good agreement.

11:00 AM U1.9
TEM analysis of misfit stress in semiconductor epitaxial systems by the curvature method. Martiane Cabie[1], Anne Ponchet[1], Lise Durand[1], Andre Rocher[1] and Nicolas Bertru[2]; [1]CEMES, CNRS, Toulouse, France; [2]INSA, Rennes, France.

The stress induced by a lattice mismatch between an epilayer and its substrate is responsible for bending the sample. The stress and the curvature are related together through Stoney's formula which also involves the thicknesses of both the epitaxial layer and the substrate. This stress is classically determined by measuring the curvature of thick samples. In this work, this method has been transposed to TEM analysis, as described by Rocher et al (1). TEM analysis is performed under the conventional 2-beam conditions: the curvature radius is directly derived from the distance between the -g and +g bend contours observed in bright field. At 200kV, the proportionality factor is 80 for the 220 reflection. The thickness of the sample is determined from the variation of the diffracted intensity with the Bragg deviation. The investigated samples are GaInAs 30 nm layer grown by MBE on (100) InP substrates. The In concentration has been chosen equal to 43 and 71% in order to obtain respectively tensile and compressive biaxial stresses in the epilayer. Samples are prepared for TEM plane view observation. When the substrate is thinned from 300μm to 0.3μm the curvature radius induced by the misfit stress decreases from about 100m to less than 100μm. Positive and negative curvatures have been observed in good agreement with the tensile or compressive character of epitaxial stress. The variation of the curvature radius with the substrate thickness agrees well with the Stoney formula. However, its experimental value is always larger than predicted. As a consequence, the measured misfit stress is about 20% lower than the theoretical one calculated for an ideal pseudomorphic layer. Numerical calculation based on Finite Element Method has been performed to justify the use of the Stoney formula for the thin samples studied by TEM. (1) Rocher A, Cabie M, Ponchet A, Carrere H, Proceedings of the Microscopy of Semiconducting Materials Conference, Cambridge UK April 2003.

11:15 AM U1.10
Kinetic Mechanisms of Tensile and Compressive Stress Evolution in Vapor-deposited Polycrystalline Films.
Brian W. Sheldon, Ashok Rajamani, Hao Li, Abhinav Bhandari, Rod Beresford, Eric Chason and Janet Rankin; Division of Engineering, Brown University, Providence, Rhode Island.

Experimental measurements in several different polycrystalline ceramic films demonstrate that intrinsic stress evolution is not well described by existing models. Investigations were performed with AlN grown by MBE, and SiC and diamond grown by CVD. These systems were selected because grain boundary diffusion during deposition is negligible, in contrast to most of the metal films that have been the focus of previous work on intrinsic stresses. The absence of grain boundary diffusion greatly facilitates comparisons with various models of stress evolution. In comparison with previous models, the measured stresses show much weaker grain size dependence along with a strong dependence on the film growth rate and the deposition chemistry. To explain these results, a kinetic model that describes both tensile and compressive stress generation is proposed. Both of these mechanisms are believed to occur at grain boundaries, and the new model reconciles the competition between atomic-scale phenomena in a way that is consistent with a wide range of experimental observations. The experimental and theoretical results also demonstrate that processing conditions can be used to control stress gradients, which is particularly important for certain applications (e.g., MEMS).

11:30 AM U1.11
Nanoscale Stress Analysis of Semiconductor Devices Using Quantitative Electron Diffraction Contrast. Jian Li[1], Dalaver Anjum[1], Guang Rui Xia[2], Judy Hoyt[2] and Robert Hull[1]; [1]MSE dept, University of Virginia, Charlottesville 22903, Virginia; [2]Microsystems Technology Laboratories, Department of Electrical Engineering, Massachusetts Institute of Technology, Cambridge 02139, Massachusetts.

Stress is an increasingly important issue in semiconductor devices, for example because stress in the channel of a MOSFET device can significantly increase transistor operating speed. With the continuous reduction in device dimensions, nanoscale stress characterization techniques have become increasingly necessary. In this work, we present a technique that uses quantitative Electron Diffraction Contrast (EDC) to measure stresses with lateral spatial resolution on the order of 10 nm and sensitivity on the order of tens of MPa. This is accomplished by utilizing Transmission Electron Microscopy (TEM) and Focused Ion Beam (FIB) techniques in conjunction with an ensemble of computer programs that include finite element modeling (FEM), electron diffraction strain contrast simulation and image manipulation. Precisely controlled experimental EDC images of high mobility strained-Si channel MOSFETs were obtained. FEM models were then constructed for the initial stress simulations of these devices, using thin film wafer curvature measurements of the component materials of the device. The FEM output files, coupled with the precise TEM experimental conditions, were then used as the input files for EDC simulations of the entire structure. An image intensity difference map was then created, and calibrated in terms of stress differences, between the simulated image and the experimental TEM image. Stresses of the component materials (low temperature oxide, thermal oxide, TiSi2 and Poly-Si) were then varied in the FEM model until a best match between these two images is found. Analysis of various gate-length MOSFET devices shows that the stresses in the central part of the strained channel reduce by several hundred MPa when the gatelength changes from 550 nm to 100 nm, respectively. Analysis also shows that the stress distribution in the strained-Si channel is very sensitive to the stress state of the surrounding material, especially TiSi2, which can modify the stress distribution in the channel by well over 100 MPa.

11:45 AM U1.12
Strain Relaxation and Kink Nucleation During InGaAs/GaAs

Growth. Candace Lynch, Eric Chason and Rod Beresford; Division of Engineering, Brown University, Providence, Rhode Island.

Understanding strain relaxation behavior is important for growing heteroepitaxial semiconductor layers. *In − situ* stress measurement has made it possible to study the kinetics of dislocation-mediated stress relaxation during semiconductor heteroepitaxy. In this present work we have been studying relaxation behavior during growth interruptions and in compositionally graded layers to understand the process of relaxation and develop better methods for producing relaxed layers. Pausing InGaAs deposition during MBE growth of InGaAs/GaAs results in a significant decrease in the strain relaxation rate. Once growth is resumed, the strain relaxation rate returns to the earlier (high) value. We have observed this behavior over a range of temperatures and at different growth fluxes. This dependence of the strain relaxation rate on growth is explained by a model in which the process of dislocation glide by single kink nucleation is enhanced by the growth-induced surface supersaturation of adatoms. We will also present results from the growth of multilayers, including growth of GaAs layers on InGaAs thin films and growth of graded InAlAs films. The strain relaxation behavior during these growths will be discussed in terms of kink nucleation and dislocation glide. These results suggest that the effect of deposition on dislocation motion should be taken into consideration when using *ex − situ*stress measurements to analyze and optimize growth of relaxed layers.

SESSION U2: Modeling Stresses and Film Instability
Chairs: Robert Cook and Alex Volinsky
Monday Afternoon, December 1, 2003
Room 210 (Hynes)

1:30 PM *U2.1
Dynamics of stress Evolution in Thin Films. Eric Chason, Division of Engineering, Brown University, Providence, Rhode Island.

Thin films during processing are not equilibrium systems. The film structure changes dramatically as the film grows from isolated clusters into connected islands and ultimately to a continuous film. Growth conditions can change as well, as the growth flux is turned off and on or the temperature is cycled. In order to understand the development of stress in films, it is useful to be able to monitor the stress continuously during processing. We describe an optical technique that enables the stress to be measured in situ without interrupting processing. Several examples of how film stress changes during processing and its correlation with microstructure and growth conditions will be discussed.

2:00 PM U2.2
Size effects in tensile deformation evidenced by molecular dynamics and in-situ profile analysis in the Swiss Light Source. Helena Van Swygenhoven, Peter M Derlet and Zeljka Budrovic; Paul Scherrer Institute, PSI-Villigen, Switzerland.

It has been experimentally demonstrated that the mechanical properties of thin films depend on the thickness of the film, and this is very important in MEMS development. Often it is reported that this type of size effect starts playing a role when microstructural dimensions approach the physical size of the object. Large-scale Molecular Dynamics simulations are employed to investigate the influence of free surfaces during tensile deformation of nanocrystalline fcc metals. Samples representing a microbeam containing up to 750 grains with mean grain sizes of 5 nm are simulated in which periodicity along a direction normal to the tensile load is relaxed to explicitly include the role of free surfaces. Additionally the same sample without free surfaces is deformed for comparison. The second moment potential for Ni of Cleri and Rosato was used. Grain boundary relaxations, including grain reorientation and the formation of surface steps was observed during deformation. The influence of the free surfaces on the deformation processes such as GB sliding and partial dislocation activity, resulting in the formation of mesoscopic shear planes is discussed. First measurements on size effects measured during in-situ deformation and diffraction profile analysis in the Swiss Light Source are discussed.

2:15 PM U2.3
Statistical model of threading dislocation density evolution in heteroepitaxial thin film growth. Robin Selinger, Physics Dept., Catholic Univ., Washington, District of Columbia.

We present an idealized statistical model for the reduction of threading dislocation density during growth of a heteroepitaxial thin film. The model is based on a novel criterion for onset of unstable dislocation motion leading to pair annihilation, in which the annihilation radius (the threshold separation for dislocation pair annihilation) is an increasing function of film thickness. We consider a highly idealized system containing an initial density of threading dislocations of only two species, e.g. screw dislocations of opposite sign, whose positions are random in the plane of the film. As film growth progresses, the annihilation radius increases and pairs of +/- dislocations are removed sequentially from the population, resulting in the statistical emergence of clusters of like-signed dislocations. Simulation results for dislocation density vs film thickness are compared with experimental data from the literature. We discuss methods by which this highly idealized model might be further developed to provide a more realistic representation of this process. For details, see Phys. Rev. B 67, 134108 (2003).

2:30 PM U2.4
Image Stresses and Plasticity in Thin Films: a hybrid method combining dislocation dynamics simulation, finite element method and an analytical approach. Meijie Tang[1], Wei Cai[1], Vasily Bulatov[1] and Guanshui Xu[2]; [1]Lawrence Livermore National Lab., Livermore, California; [2]Mechanical Engineering, U. C. Riverside, Riverside, California.

Dislocation Dynamics (DD) simulation is an effective computational approach for studying physical mechanisms of crystal plasticity based on the collective motion of dislocations. In thin films, image stresses due to the presence of free surfaces can play a crucial role to the dislocation behavior and must be accounted for properly. This is typically treated by combining Finite Element Method (FEM) with DD. However, for dislocations intersecting the surfaces, the singularity of image stresses at the intersection points must be treated with substantial care. A brute force numerical treatment of this apparent singularity requires a very fine FEM meshes and hence is highly inefficient. We solve this problem by employing a special analytic solution that accounts for the singularity and only treat the remaining, smoothly varying stress field numerically, using a much coarser FEM mesh. This enables accurate and efficient numerical simulations of the general dislocation behavior in thin films. We will discuss a few preliminary applications of this new methodology in metallic thin films. The work of Drs. Tang, Cai and Bulatov was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

2:45 PM U2.5
Modeling of the Mechanical Behavior of Thin Films and Coatings for Microelectronics and Fiber-optics Structures: Review and Extension. Ephraim Suhir, [1]ERS Company, Los Altos, California; [2]Dept. of Civil and Materials Engineering, University of Illinois at Chicago, Chicago, Illinois.

The talk contains a brief review of the work on modeling of thermally induced stresses in thin films and coatings used in microelectronics and fiber optics structures. Thin films and coatings have plenty in common. Indeed, the silica material in a fiber optic structure plays with respect to its thin and/or low modulus coating the same role as a thick substrate plays with respect to a thin film fabricated on it. The review is based primarily on the author's research carried out during his eighteen year tenure with Bell Laboratories, Physical Sciences and Engineering Research Division. The extension part of the talk has to do with the application of a probabilistic approach. The discussed work includes, but is certainly not limited to the following publications: * E. Suhir, "Thermal Stress Modeling in Microelectronics and Photonics Packaging, and the Application of the Probabilistic Approach: Review and Extension", IMAPS Journal of Microcircuits and Electronic Packaging, vol.23, No.2, 2000 (invited paper). * S. Luryi and E. Suhir, "A New Approach to the High-Quality Epitaxial Growth of Lattice - Mismatched Materials", Applied Physics Letters, vol. 49, No. 3, July 1986. * E. Suhir, "An Approximate Analysis of Stresses in Multilayer Elastic Thin Films", ASME Journal of Applied Mechanics, vol. 55, No. 3, 1988. * E. Suhir, "Approximate Evaluation of the Elastic Interfacial Stresses in Thin Films with Application to High-Tc Superconducting Ceramics", Int. Journal of Solids and Structures, vol. 27, No. 8, 1991. * E. Suhir, "Approximate Evaluation of the Elastic Thermal Stresses in a Thin Film Fabricated on a Very Thick Circular Substrate", ASME Journal of Electronic Packaging, vol. 116, No. 3, 1994. * E. Suhir, "Predicted Stresses in a Circular Substrate/Thin-Film System Subjected to the Change in Temperature", Journal of Applied Physics, vol.88, No.5, 2000. * E. Suhir, "Stresses in Dual - Coated Optical Fibers", ASME Journal of Applied Mechanics, vol. 55, No. 10, 1988. * E. Suhir, "Thermal Stress in a Polymer Coated Optical Glass Fiber with a Low Modulus Coating at the Ends", Journal of Materials Research, vol. 16, No. 10, 2001.

3:30 PM *U2.6
Wrinkling of Thin Films on Compliant Substrates. Rui Huang, Aerospace Engineering and Engineering Mechanics, University of Texas at Austin, Austin, Texas.

Under in-plane compression, a freestanding membrane tends to buckle into an equilibrium state. For a thin film bonded to a substrate, the buckling is constrained. If the substrate is elastic and relatively compliant, the film buckles into an equilibrium state with short wavelengths, often called wrinkles. For given material properties of the film and the substrate, a critical compressive stress exists, above which wrinkle forms to reduce the total energy. By including a nonlinear term due to large deflection of the film, energy minimization predicts the equilibrium wrinkle wavelength and amplitude. For a viscous or creeping substrate, wrinkling becomes a kinetic process, with time-dependent amplitudes and wavelengths. Initially, the amplitude grows exponentially and the fastest growth mode dominates. Gradually, the amplitude saturates, approaching an equilibrium state. Subsequent evolution of the wrinkles is kinetically constrained and very slow, despite that the energetics favors wrinkles of longer wavelengths. For film of finite sizes, edge relaxation may eventually wipe out all the wrinkles. Alternatively, wrinkles may cause fracture due to large tensile stress of bending. For a film sitting on a plastic substrate, thermal cycling causes ratcheting deformation in the plastic layer. An analogy between ratcheting and creep is developed to show that wrinkles grow as the temperature cycles.

4:00 PM U2.7
Thermo-Mechanical Analysis of Polymer Materials by Means of Spherical Indentation. Zizhen Liu[1,3], Geoffrey M. Spinks[1,2], Hugh R. Brown[1], Mike Swain[3], Evan Evans[5] and Howard See[4]; [1]BHP Steel Institute, University of Wollongong, Wollongong, New South Wales, Australia; [2]School of Mechanical, Materials and Mechatronic Engineering, University of Wollongong, Wollongong, New South Wales, Australia; [3]School of Aerospace, Mechanical and Mechatronic Engineering, University of Sydney, Sydney, New South Wales, Australia; [4]Department of Chemical Engineering, University of Sydney, Sydney, New South Wales, Australia; [5]Research Laboratories, BHP Steel, Wollongong, New South Wales, Australia.

The performance of polymer coating is highly dependent on its physical and mechanical properties and their variation with temperature. It is difficult to measure thermo-mechanical properties of thin polymer coatings on metal substrate since the substrate dominates the mechanical response of the system. Indentation methods offer a possible means for measuring the coating properties in situ. The problem, however, is that the penetration can extend to the substrate with increasing temperature. This paper presents an approach to measure the thermo-mechanical properties of bulk polystyrene by using controlled force indentation tests. The experiments were carried out on a Dynamic Mechanical Analyser (DMA). A spherical indenter was employed to establish the displacement change, softening points, glass transition, depth of penetration and its elastic recovery of the material. The total penetration and residual depth of impression were obtained using the results at the maximum load in a range of 0.5N to 2N. The elastic recovery and geometry of the spherical indenter were subsequently calculated. The value of the composite elastic modulus from the recovery on fully unloading was determined by application of the Hertz equation. The effects of different temperatures for the elastic composite modulus were derived using the Hertz relationship between force and change in displacement. A preconditioning was carried out prior to the real test to prevent errors caused by any initial stress. A number of dual cantilever tests were also performed with the temperature ranging from 25C∼140C degree to verify the accuracy of the indentation tests. Both the results of the indentation and dual cantilever tests were consistent. In comparison to tensile, three-point bending or other types of measurement methods, this study indicates that indentation test can be used to measure the thermo-mechanical properties of thin polymer coating on a metal substrate effectively.

4:15 PM U2.8
Stress Evolution in Spin Coated Films of PMMA.
Anita M. Bowles[1], John W. Hutchinson[2] and Frans Spaepen[2]; [1]Harvard University, Cambridge, Massachusetts; [2]Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

Stress evolution in spin coated polymethyl methacrylate (PMMA) thin films on silicon is investigated using a coherent gradient sensing system. Two-dimensional, time- dependent measurements of the stress tensor are made over a large wafer area. The stress development accompanying solvent evaporation and stress relaxation as a function of temperature in the formed film are measured for different spin speed speeds (thicknesses) and molecular weights of PMMA. An analysis was made of how stress anisotropy (radial, tangential) would affect the curvature of the substrate.

4:30 PM U2.9
Rupture of Thin Metal Film on Compliant Substrate.
Teng Li[1], Zhenyu Huang[1], Zhichen Xi[1], Zhigang Suo[1], Stephanie P Lacour[2] and Sigurd Wagner[2]; [1]Department of Mechanical and Aerospace Engineering and Princeton Materials Institute, Princeton University, Princeton, New Jersey; [2]Department of Electrical Engineering, Princeton University, Princeton, New Jersey.

Recent experiments show that a thin metal film on a compliant substrate can sustain significant tensile strain before fatal fracture, much larger than its freestanding counterpart, whose tensile fracture strain is only about 1 percent. The major fracture mechanism of thin metal film is rupture by continued thinning around one section, so-called "necking", or by separating into two parts by slip confined to one or a few slip planes. At fracture, the local deformation near the necking section or slipping-off part of metal film is huge, usually several 100 %. A large free space must be available for thin films to accommodate this huge localized deformation. While free space is always available for the rupture of free standing films, the necking (or slipping-off) of a metal film bonded to a compliant substrate will be suppressed or delayed until debonding occurs between the film and the substrate. Theoretical analysis and finite element calculation show that a thin metal film well-bonded to a compliant substrate can be deformed stably to very high nominal strain. Finite element analysis reveals that, when the metal film is partially debonded from the substrate, the longer the debonding length, the less the substrate can suppress the film necking. The deformability of a thin metal film on a compliant substrate is also affected by the hardening of the substrate material. The higher the hardening index of the substrate, the larger the fracture strain of the system.

4:45 PM U2.10
Phase field modeling of surface instabilities induced by stress.
Dong Jin Seol, ShenYang Hu and Long-Qing Chen; Mat Sci & Eng, Penn State Univ, University Park, Pennsylvania.

Stress-induced surface instability is associated with the dislocation-free Stranski-Krastanov growth mode. During the SK growth, the growth mode transition from a layer-by-layer growth to a three-dimensional island growth occurs by the competition between the strain energy and surface energy of a film. It is essential to understand the stability and evolution dynamics of such island structures in producing controllable quantum dot arrays for optical or electronic applications. In this work, we developed a phase field model describing surface evolution dynamics of a strained solid in contact with its vapor phase. We solve elastic solutions with an iteration method for the system in which a solid film, strained by the mismatch strain between the film and substrate, has anisotropic elastic constants and the vapor phase has zero elastic constants. The phase field equation and Cahn-Hilliard equation are solved together using the semi-implicit Fourier-spectral method. The model is applied to investigating the temporal evolution of morphological instabilities on the surface leading to the islands formation and subsequent coarsening from a uniform coherent film. The effects of various factors such as the mismatch strain, the surface energy and its anisotropy, and the elastic anisotropy are studied.

SESSION U3: Deformation and Adhesion
Chairs: Paul Besser and Rui Huang
Tuesday Morning, December 2, 2003
Room 210 (Hynes)

8:30 AM *U3.1
Fracture In Thin Oxide Films. David F. Bahr, Adam L. Olson, Marian S. Kennedy, Mengzhi Pang, Diego Rodriguez-Marek and Abdulaziz Alamr; Mechanical and Materials Engineering, Washington State University, Pullman, Washington.

Oxide films can be grown in a variety of methods, ranging from electrochemical methods to vapor deposition and solution deposition. In most cases, the films are supported by more ductile underlayers and substrates. This paper will review some of the methods by which the fracture strength of thin oxide films which are subjected to both tensile and compressive loading fail. The strength of these films can be related to the microstructure in many cases, including anodic oxide films on metals and titanate based piezoelectric oxides on metallic conductors for MEMS. Through thickness fracture will be examined and demonstrated using both bulge testing and nanoindentation techniques, which allow quantification of the stresses required for fracture. Amorphous films tend to exhibit larger stresses to failure, in the 1-10 GPa regime, than crystalline films of similar compositions. As film thickness increases from 10s of nm to 1000s of nm, the applied tensile stresses for fracture decrease in solution deposited oxide films, reaching approximately 150 MPa for solution deposited lead zirconate titanate. The reduction in strength will be related to the defects observed using electron microscopy. Comparisons of nanoindentation, bulge testing, and bulk notched tensile bars will show how the length scale of testing can impact the observed strengths via a distribution of flaws in the sample volume.

9:00 AM U3.2
Fracture Mode Transitions in Ceramic Films as a Function of Thickness. Herzl Chai and Brian R Lawn; MSEL, NIST, Gaithersburg, Maryland.

The fundamentally changing nature of fracture in ceramic films on compliant substrates is examined as a function of diminishing film thickness. Attention is focussed on cracking induced by concentrated loading at the layer top surface as an illustrative case study. It is shown that the fracture mode undergoes fundamental transitions, from surface ring cracking around the contact (thick coatings) to subsurface radial cracking at the lower ceramic surface (thin coatings) and, finally, back to surface ring cracking (thin films). These transitions reflect a progressively changing stress field in the layer structures, and highlight the differences in failure mechanism that may be anticipated at the macro-scale and small-scale levels. Simple fracture relations will be presented for each mode, and data from FEA simulations and contact experiments on model ceramic/polymer bilayer systems will be used to validate the analysis. Implications of the transitional behavior in relation to the strength of brittle coatings/film systems will be discussed.

9:15 AM U3.3
Measurements of Interfacial Fracture Energy in ZnO Film/Si Substrate Systems by Indentation Tests. Bin Huang, Ming-Hao Zhao and Tong-Yi Zhang; Department of Mechanical Engineering, Hong Kong University of Science and Technology, Hong Kong, Hong Kong.

Combining the composite hardness models with the indentation-induced delamination model, we developed a method to determine the interfacial fracture energy between a film and its substrate. The novel method is particularly useful for indentation equipment without any displacement measurement devices. Experimentally, we deposited ZnO films with thicknesses ranging from 0.202 to 1.535 micrometer on 525 micrometer-thick (100) Si substrates using the magnetron sputtering method. Then, Vickers indentation tests were carried out on the ZnO/Si systems at room temperature, in which the applied load varied from 10 mN to 2.0 N. The experimental results show that only indentation-induced radial cracking occurred in the systems with film thicknesses being equal to and thinner than 0.554 micrometer, from which the residual stress in the films was extracted to be 387 MPa in compression. For the systems with film thicknesses being equal to and thicker than 0.832 micrometer, only indentation-induced delamination occurred when indentation loads were low. Using the new method, we extracted the interfacial fracture energy to be about 12.2 J m-2 and 10.5 J m-2 for the cases without and with buckling of delaminated films, respectively.

9:30 AM U3.4
Thin Film Herringbone Buckling Patterns. Xi Chen[1] and John W. Hutchinson[2]; [1]Civil Engineering and Engineering Mechanics, Columbia University, New York, New York; [2]Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

Self-assembly involving thin films has become the cornerstone for fabricating micro and nanostructures. In this study, spontaneous pattern formation by biaxial elastic buckling is analyzed. A thin metal film vapor deposited on thick elastomer substrate develops an equi-biaxial compressive stress state when the system is cooled due to the large thermal expansion mismatch between the elastomer and the metal. At a critical stress, the film undergoes buckling into a family of modes with short wavelengths characteristic of a thin plate on a compliant elastic foundation. As the system is further cooled, a highly ordered herringbone pattern has been observed to develop. Here it is shown that the herringbone mode constitutes a minimum energy configuration among a limited set of competing modes. Closed form solutions are obtained for the competing one-dimensional and square checkerboard buckling modes. Numerical analysis has been used to obtain the strain energy of the herringbone mode. It is shown that the herringbone mode can relieve 50% more energy than other competing modes, which makes it to become the favorite buckling pattern in the thin film systems.

9:45 AM U3.5
Measuring Adhesion Energies between Metal Films and Polymers by Hydrogen Induced Buckeling. Reiner Kirchheim, Astrid Pundt and Eugen Nikitin; Institut fuer Materialphysik, University of Goettingen, Goettingen, Germany.

Hydrogen absorption in niobium films on polycarbonate (PC) gives rise to large compressive in plane stresses which finally lead to a delamination of the metal from the substrate. Delamination occurs via straight and wrinkled buckles and can be observed with an optical microscope. By doping the Nb-film electrochemically with hydrogen the increasing stresses are measured by determining the curvature of the PC-substrate. The initial linear increase of the stress is in accordance with the known volume expansion caused by H-atoms in Nb. Deviations from the linear response start at the same time when buckles are formed. The corresponding critical stress for buckling is measured for 50, 100 and 200 nm thick Nb-films which were covered on both sides with a 10 nm thick Palladium film. It can be shown that the critical stresses for buckling are much larger than the ones needed to exceed Euler's instability criterion. They are required to overcome the adhesion between Pd and PC. From a simple energy balance between the adhesion energy and the released elastic energy a value of about 1 J/m/m for the adhesion energy is calculated. The precision of the evaluated adhesion energy can be improved by measuring initial stresses and/or the shape of the buckles. It will be shown that the new concept of measuring adhesion energies is rather general and can be applied to all combinations of two materials, if the dissolution of substances in one of them causes compressive stresses.

10:30 AM U3.6
Effects of Roughness and Interface Chemistry on Adhesion of Metal Films To Dielectric Substrates. Mengzhi Pang, Christopher C. Umbach and Shefford P. Baker; Department of Materials Science and Engineering, Cornell University, Ithaca, New York.

Metal film/dielectric interfaces are of major technological relevance in microelectronics, flat panel displays, catalysts, sensors, and many other applications. The lack of strong chemical affinity between a metal and dielectric often leads to a weakly bonded interface, which may undergo slow delamination due to the combined effects of film residual stress and corrosion. In this report, effects of roughness and interface chemistry on adhesion are studied in detail. Interfaces with well-controlled roughness were prepared by using ion beam bombardment to generate silicon oxide surfaces with periodic corrugations having wavelengths less than 200 nm and amplitudes up to 2 nm. Oxygen content was controlled by precisely regulating the amount of oxygen in the ultra high vacuum chamber during film deposition. Interfacial fracture resistance was measured using a driver film method. A fracture mechanics based mechanism is proposed in terms of the stress state and localized chemistry at the crack tip.

10:45 AM U3.7
Nanoindentation induced delamination and telephone cord induced delamination methods for the study of Pt adhesion . Alan Lee, Bruce M Clemens and Paul C McIntyre; mse, stanford, Stanford, California.

Adhesion of Pt films to Si substrates with a native oxide has been investigated using two methods of quantitative adhesion characterization. The nanoindentation induced delamination method uses an impression to store compressive strain energy in an overlayer film in order to induce delamination at the Pt/SiO2 interface. Likewise, the telephone cord induced delamination method involves sputtering on a thick compressively stressed overlayer onto the Pt/SiO2 films to induce telephone cord delamination patterns in the Pt film. Crack extension forces and interface toughnesses can be calculated from the dimensions of the circular blister or the telephone cords using currently available models. Our experiments, coupled with focused ion beam (FIB) observations, show that the nanoindentation method is difficult to implement due to extensive crack formation in the substrate beneath the indentation impression. As a result, interface toughnesses from this test are grossly overestimated. The telephone cord measurements on the other hand give realistic interface toughnesses, allowing us to show that the adhesion of Pt to oxidized Si can be controlled by changing the Argon gas pressure during Pt sputtering. However, this method is currently limited by the maximum amount of compressive stress that can be grown into the overlayer. As a result, the upper limit of crack extension force that can be measured by the telephone method is on the order of 6 N/m for the sputtered molybdenum overlayers and 10 N/m for the sputtered tungsten overlayers used in this work. As a result we were able to show that decreasing the argon pressure during Pt sputtering significantly increases the adhesion of the films to the substrate, suggesting that the highly energetic neutral argon atoms striking the growing surface improve interface adhesion.

11:00 AM U3.8
Stress-induced fracture pattern formations is thin films. Alex A Volinsky[1], Dirk Meyer[2], Tilmann Leisegang[2] and Peter Paufler[2]; [1]PMCL, Motorola, Tempe, Arizona; [2]Institut fur Strukturphysik, Fachrichtung Physik der Technischen Universitat, Dresden, Germany.

While there are many stress relief mechanisms observed in thin films, excessive residual and external stresses cause film fracture. In the case of tensile residual stress a network of through-thickness cracks forms in the film. In the case of compressive residual stress thin film buckling is observed in the form of blisters. Thin film delamination is an inseparable phenomenon of buckling. The buckling delamination blisters can be either circular, straight, or form periodic buckling patterns commonly known as telephone cord delamination morphology. While biaxial residual stress is the key for causing thin film fracture, either in tension, or compression, it is the influence of the external stress that can controls the final fracture pattern. In this paper we consider phone cord buckling delamination observed in compressed W/Si and TiWN/GaAs thin film systems, as well as spiral and sinusoidal though-thickness cracks observed in Mo/Si multilayers on bent substrates in tension.

11:15 AM U3.9
Crack Tip Plasticity Effects on the Interfacial Failure of Thin Gold Films. Neville Reid Moody[1], David Adams[2], Megan Cordill[3], Alex Volinsky[4], David Bahr[3] and William Gerberich[5]; [1]Sandia National Laboratories, Livermore, California; [2]Sandia National Laboratories, Albuquerque, California; [3]Washington State University, Pullman, Washington; [4]Motorola, Mesa, Arizona; [5]University of Minnesota, Minneapolis, Minnesota.

Gold films are the material of choice for MEMS mirrors. They must be thin to minimize mirror curvature and well-adhered to resist delamination and spalling. However, the relationship between film thickness and adhesion is not well-defined especially at the nanoscale. As a result, we have begun a study of the relationship between film thickness and gold film adhesion. The films were sputter deposited onto sapphire substrates to thicknesses ranging from 10 nm to 200 nm. Nanoindentation was used to measure properties and combined with stressed overlayers to trigger delamination and buckling. Fracture energies and interfacial toughness values were then obtained from the buckles using mechanics-based models. The results showed that fracture energies decreased with decreasing film thickness to a lower limiting value near 1.5 J/m2 for films less than 100 nm thick. The corresponding mode I component was equal to 0.6 J/m2 for all films studied. In this presentation it will be shown that the opening mode component equals the true work of adhesion and governs delamination in all tests. The shear component is comprised of contributions from the test technique and crack tip plasticity with a direct correlation between the increase in fracture energy and the emission of crack tip dislocations. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

11:30 AM U3.10
Energy Dissipation Processes in Thin Film.
Jeffrey William Kysar, Department of Mechanical Engineering, Columbia University, New York, New York.

A ductile material is generally understood to be able to dissipate energy through the activation of various irreversible energy dissipation mechanisms. Often it is of interest to know the ductile response of a material which contains a crack, so it is important to predict the conditions under which different energy dissipations are activated at or near the crack tip. For instance, in a pure crystalline material of macroscopic extent, the material can dissipate energy via cleavage, crack tip dislocation nucleation, and activation of a Frank-Read source near the crack tip. It is notable that the lengths involved with these dissipation mechanisms span from the atomic to the micron length scales. The activation conditions for cleavage and crack tip dislocation nucleation have been studied extensively. A recent analysis by Kysar (J. Mech. Phys. Solids, 2003, 51, 795-824) quantified the approximate energy release rate at which Frank-Read sources are activated. Therefore it is possible to set up energetic competitions between the three possible energy dissipation mechanisms across the pertinent length scales. The result of the analysis was a set of criteria which determine the conditions under which each of the three dissipation mechanisms is the first to be activated. If cleavage or crack tip dislocation nucleation is the initial energy dissipation mechanism, the material is, respectively, intrinsically brittle or intrinsically ductile. On the other hand, if Frank-Read sources are the initial energy dissipation mechanism, the material is said to be extrinsically ductile. It should be emphasized that as a material becomes very small, certain of the possible energy dissipation mechanisms are restricted from occurring, and it is possible that other dissipation mechanisms not previously considered can be activated. The present talk will explore the ways in which energy dissipation mechanisms are different in thin films than in macroscopic materials, and how these changes will affect the criteria for initial energy dissipation mechanism.

11:45 AM U3.11
DDD Simulations of Thin-Film Plasticity. Klaus W. Schwarz, IBM Research, Yorktown Heights, New York.

Discrete Dislocation Dynamics (DDD) simulation of the actual dislocation motions during plastic behavior can not only provide quantitative predictions about specific film systems, but can also help to identify the important concepts which need to enter theoretical models. We report simulations of the dislocation motion in a thin film that is suddenly strained. The simulations show good agreement with experiment, while yielding some unexpected new insights. Specifically, we find that hardening interactions such as jogging and junction formation play an important role in thin-film plasticity; that local over-relaxation is more significant than 'blocking' in limiting the plastic response of a thin film; and that the density of sources strongly affects the degree to which a film can relax.

SESSION U4: Film Fracture and Fatigue
Chairs: David Bahr and Maarten de Boer
Tuesday Afternoon, December 2, 2003
Room 210 (Hynes)

1:30 PM *U4.1
Toughness and Contact Behavior of Conventional and Low-k Dielectric Thin Films. Robert F. Cook[1], Dylan J. Morris[1] and Jeremy Thurn[2]; [1]Materials Science and Engineering, University of Minnesota, Minneapolis, Minnesota; [2]Advanced Mechanical Technology, Seagate Technology, Bloomington, Minnesota.

Dielectric films are used in a wide variety of advanced technologies: microelectronic, photonic, magnetic storage and micro-electromechanical systems (MEMS). In all cases, the dielectric performs a (frequently primary) role as a structural element, in addition to providing appropriate insulating, optical or permeabilitiy characteristics. As most dielectrics are brittle, knowledge of film toughness (controlling all aspects of film fracture) and sharp contact behavior (the dominant mode of crack initiation) is then of crucial importance for advanced device design. This talk will describe a variety of controlled-flaw, indentation-based, techniques used to determine the toughness of two sets of dielectric films. The first will consider PVD aluminum oxide films of order 5-50 micrometers thick and use macroscopic methods, including macroscopic depth-sensing indentation and optical observation of indentation-induced film and interface cracks, to estimate film toughness and interfacial fracture resistance. The second will consider spin-on low-k films of order 0.5-2 micrometers thick and use ultra-microscopic methods, including nanoindentation and low voltage SEM of cracks, to estimate film toughness. New models are presented that describe the variation of crack length with indentation load and permit film toughness to be estimated. Key features of these models are: the alteration of contact and fracture geometry, as the elastic and plastic indentation fields, and cracks, interact with a substrate; the effects of film stress to drive or resist fracture; and, for the low-k materials, the introduction of a new indentation fracture term associated with the wedging action of indenters of greater acuity (cube-corner, for example) than the conventionally used Vickers and Berkovich.

2:00 PM U4.2
The Effect of Varying Length Scales on the Interfacial Adhesion of Thin-Films and Patterned Lines.
Christopher S. Litteken[1], Guanghai Xu[2], Tracey Scherban[2], Jihperng Leu[2] and Reinhold Dauskardt[1]; [1]Materials Science and Engineering, Stanford University, Stanford, California; [2]Logic Technology Development Quality & Reliability, Intel Corporation, Hillsboro, Oregon.

The dimensions of materials utilized in emerging device technologies play an important role in determining their mechanical behavior, particularly when confined to the nanometer length scale. The intent of the present study was to investigate the role of varying length scales and multi-dimensional elastic constraint on the adhesive characteristics of thin-films and patterned lines commonly used in microelectronic devices. Thin-film structures containing metal, organic, and inorganic films were fabricated that systematically varied, on the nanometer to micron scale, film thickness, aspect ratio of patterned features and pore size. Fracture mechanics techniques were employed to measure the critical interfacial adhesion energy (G_c). Results from these studies will be presented that indicate interfacial adhesion is dominated by energy dissipation mechanisms such as plastic deformation at the debond tip and frictional contact of surface asperities behind the debond tip. It will be demonstrated that the extent to which these energy dissipation mechanisms contribute to the macroscopic adhesion of the structure is related to the pertinent length scale of the adjacent features. Mechanics models detailing the role of varying length scales on the relevant energy dissipation process and high-resolution surface characterization will be presented to explain the observed trends in adhesion.

2:15 PM U4.3
Understanding Adhesion Failure in Low-K Dielectric Stacks During Chemical Mechanical Polishing. Francesca Iacopi[1,2], Dominiek Degryse[1,2], Ingrid Vos[1], Matthias Patz[3] and Karen Maex[1,2]; [1]IMEC, Leuven; [2]E.E.Dept., Katholieke Universiteit Leuven, Leuven, Belgium; [3]JSR Corp., Tsukuba, Japan.

Low-k dielectrics are currently being considered for replacing SiO2 in advanced on-chip interconnects. Being low surface energy materials, a major concern for the integration of such dielectrics in Cu damascene processes is given by adhesion failure at the interfaces of the low-k films with capping or liner layers during Chemical Mechanical Polishing (CMP). In recent literature it was shown how occurrence of interface failure can be reduced by enhancing the fundamental work of adhesion (i.e. increasing surface energy of the dielectric films and favouring cross-layer bonds). In this study we discuss also the importance of the practical work of adhesion in determining failure upon CMP load. In particular, we report on the critical role of CMP -induced shear stress on the onset of interface debonding, and the way the shear load is transmitted to the critical interfaces. A cross-comparison of interface fracture energies as measured through four-point bending, CMP experiments and stress fields computed by Finite Element Modeling, shows that it is not possible to predict failures based on adhesion energies alone. For a given CMP load, the minimal interfacial fracture energy needed to avoid failure appears strongly linked to the position of the weak interfaces, and to the thickness, residual stresses, and mechanical properties of the materials present in the stack.

2:30 PM U4.4
The Propagation Mechanism of Non-symmetric Telephone Cord Buckle in Compressive Thin Films. Myoung-Woon Moon[1], Anette Karlsson[2], Kyang-Ryel Lee[3] and Kyu Hwan Oh[1]; [1]School of Material Science and Engineering, Seoul National University, Seoul, South Korea; [2]Department of Mechanical Engineering, Univ. Delaware, Newark, Delaware; [3]KIST, Seoul, South Korea.

Delamination buckles are commonly observed in films with residually compressive stresses. Three common classes are: (i) circular blister; (ii) symmetric, straight sided blister; and (iii) anti symmetric, wavy blister. The mechanics of the two first types is fairly well understood. However, even though the last type, frequently referred to as telephone cord buckles, has received significantly attention, the true cause of the unique shape is not established. In this paper we will discuss real-time, in-situ observations of a propagating telephone cord buckle. The observations where made from diamond like carbon coating deposited on soda lime glass and silicon wafers. This study reveals that the smooth zigzag shape of the telephone cord buckle is derived from an intricate combination of local growth of circular buckles in combination of bifurcation theory at the leading front of telephone cord buckles : A symmetric leading front in circular shape of telephone cord buckle along certain direction is observed to be bifurcated at critical buckle width, then suddenly lose its symmetry perturbed by new bifurcated branch and become the non-symmetric side. At the same time, the new spreading branch is leading the propagation of telephone cord buckle as a new buckle advance. Therefore the instability at the symmetry circular front perturbed by the bifurcation branch results in the non-symmetric side of telephone cord buckle.

2:45 PM U4.5
Solutions and Discussions of Thin Film Undergoing the Nonlinear Peeling. Yueguang Wei, Siqi Shu and Ying Du; LNM, Institute of Mechanics, Chinese Academy of Sciences, Beijing, China.

The thin film, with its unique characteristics, is closely connected with most advanced materials, and has been widely applied to the surface- and interfacial-engineering areas. Its fundamental roles in modern materials are not only as protection layer or connection layer between device with device, but also as the strengthening or the toughening of material, etc. The main failure form of the thin film/substrate systems is decohesion along interface. In order to check the adhesion property (strength) between the thin film and substrate, a simple test method, peel test, was designed and presented in year fifties of last century. Due to advantage of simply operating, the peel test method has been widely applying in many research regions for measuring the adhesion behaviors between thin film and substrate or one material with another until now. In the present research, based on a nonlinear bending model for thin film, three double-parameter criteria characterizing peeling process are introduced. These double-parameter characterizations include: (1) the interfacial fracture toughness and the interfacial separation strength, (2) the interfacial fracture toughness and the slope angle of thin film at the crack tip, and (3) the interfacial fracture toughness and the critical Mises effective strain of thin film at crack tip. Based on the three double-parameter criteria, the thin film nonlinear peeling problems are solved exactly for each case. Through analyses and comparisons to different cases and different solutions, the thin film mechanical behavior and the interfacial adhesion behavior between thin film and substrate are assessed. Furthermore, the solutions based on the bending model and based on the plane strain elastic-plastic analysis are connected. The above theoretical analyses will be connected to an experimental measurement in the peel test for Ti/Si thin film/substrate system.

3:30 PM *U4.6
Fatigue in Thin Polycrystalline Films: Experiments and Modelling. Oliver Kraft, [1]Institut fuer Materialforschung II, Forschungszentrum Karlsruhe, Karlsruhe, Germany; [2]Institut fuer Zuverlaessigkeit von Bauteilen und Systemen, Universitaet Karlsruhe, Karlsruhe, Germany.

Mechanical properties of thin films are well known to depend on the film thickness. Several recent experimental studies have shown that the fatigue behavior of thin polycrystalline metal films on substrates depends strongly on film thickness and grain size. Based on experiments on Cu, Ag and Al films with thickness and grain size ranging from 50 nm to 3 μm stressed either by mechanical, thermo-mechanical or acoustical cyclic loading, the following general trends have been observed: Failure of thick films with large grains is associated with the formation of self-organized dislocation structures, extrusions at the surface and voids near the film/substrate interface. In thin films with fine grains, damage appears as intergranular grooving, voiding and possibly cracking with a trend of decreasing dislocation density with decreasing thickness and grain size. These findings indicate that fatigue in metal films with thickness and/or grain size of less than approximately 1 μm appears to be more and more controlled by diffusive mechanisms and interface properties rather than by just dislocation glide. It is argued that this transition is related to the formation of point defects, in particular vacancies, under cyclic loading. Based on a phenomenological description of the experimental observations, a strategy will be outlined how to predict the lifetime of thin metal films on substrates.

4:00 PM U4.7
Mechanical Fatigue of Polysilicon: Effects of Mean Stress and Stress Amplitude. H Kahn[1], R Ballarini[2] and A H Heuer[1]; [1]Materials Science and Engineering, Case Western Reserve University, Cleveland, Ohio; [2]Civil Engineering, Case Western Reserve University, Cleveland, Ohio.

A micromachined device fabricated from polysilicon has been developed that consists of a single edge-notched specimen integrated with an electrostatic actuator. By varying the DC and AC voltages applied to the actuator, this device allows fatigue testing of polysilicon with any desired mean stress or stress amplitude. Several surprising results have been obtained for the fatigue behavior of this brittle material. For compressive mean stresses, high fatigue stress amplitudes lead to decreased bend strength, due to crack extension. However, for low fatigue stress amplitudes, bend strength is increased for both highly tensile mean stresses and highly compressive mean stresses, while bend strength is unaffected for small mean stresses. The physical origins of the observed behavior will be discussed.

4:15 PM U4.8
Frequency Effects on AC - Induced Damage in Cu Interconnects. Young-Bae Park[1], Reiner Moenig[1], Eduard Arzt[1] and Cynthia A. Volkert[2]; [1]Max Planck Institute for Metals Research, Stuttgart, Germany; [2]Institut fuer Materialforschung II, Forschungszentrum Karlsruhe, Karlsruhe, Germany.

It has recently been observed that severe fatigue damage is formed in Al and Cu interconnects due to the cyclic temperatures generated by Joule heating of the metal lines by the passage of alternating currents (AC). However, the effect of the AC frequency on the damage evolution characteristics is not known so far. In this talk, we will summarize our in-situ SEM observations of damage formation during fatigue of polycrystalline sputtered Cu lines (100 to 300 nm thick and 8 um wide) with Ta underlayer on Si. Temperature cycles with amplitudes from 100 to 300°C and frequencies between 100 Hz and 10 kHz were applied. Damage appears as surface extrusions and intrusions and initially grows within single grains. As testing continues, the damage spreads to a larger area and eventually leads to crack formation and electrical failure of the line by local melting. The exact nature of the damage evolution depends on the grain size and orientation, which will be discussed in detail. We have previously observed that growth of damaged grains often occurs during testing, although the films had been annealed before testing to stabilize the grain structure. Our frequency studies, comparing 100 Hz and 10 kHz, show that this is a general phenomenon but is strongly enhanced by high frequencies. The higher loading frequency not only accelerated grain growth of damaged grains, but also led to earlier failure. Generally, smaller grained films exhibited longer lifetimes. Finally, results from the effect of a soft overlayer on damage formation and fatigue lifetime will be presented. These results imply that thermal mechanical fatigue may be a serious reliability threat to Cu interconnects with soft low-k interlevel dielectrics.

4:30 PM U4.9
Fracture Behavior of Micro-Sized Specimens Prepared from a TiAl Thin Foil. Kazuki Takashima[1], Discar Rudinal[1], Yakichi Higo[1], Timothy P. Halford[2] and Paul Bowen[2]; [1]P&I Laboratory,

Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; [2]Department of Metallurgy and Materials, University of Birmingham, Birmingham, United Kingdom.

γ-TiAl based alloys are expected to be applied to high temperature MEMS devices because of their excellent mechanical properties at high temperature up to 800 K. For practical applications of TiAl alloys to MEMS devices, the evaluation of mechanical properties on the micro-meter scale is important, as the effect of size reduction on mechanical properties becomes prominent at such small sizes. In particular, fracture properties including fracture toughness are essential for designing reliable MEMS devices. In this investigation, fracture tests have been carried out for micro-sized specimens prepared from a fully lamellar γ-TiAl based alloy thin foil. Micro cantilever beam type specimens with dimensions of 10 x 10 x 50 μm^3 were prepared from one lamellar colony of the thin foil by focused ion beam machining. Notches with a width of 0.5 μm and a depth of 5 μm were also introduced into the micro-sized specimens by focused ion beam machining. Notch directions were set to be trans- and inter-lamellar directions, respectively. Fracture tests were carried out using a mechanical testing machine for micro-sized specimens (MFT-2000). Fracture tests for the micro-sized specimens were performed successfully, and the fracture behavior is dependent on the notch direction. The fracture toughness of the specimen with a notch direction being perpendicular to the lamellar direction was 2-3 $MPam^{1/2}$, while that with a notch direction being the inter-lamellar direction was 0.2-0.6 $MPm^{1/2}$. This indicates that orientation of the lamellar microstructure greatly affects the fracture properties of micro-sized components prepared from fully lamellar γ-TiAl based alloy thin foils. It is required to consider the results obtained in this investigation when designing actual small scale structures using TiAl thin foils.

4:45 PM U4.10
A New Methodology to Investigate Fracture Toughness of Freestanding Thin Solid Films. Horacio Dante Espinosa and Bei Peng; Northwestern University, Evanston, Illinois.

A novel membrane deflection technique (MDT) was designed to investigate the fracture toughness of MEMS materials. The specimens were notched using a focused ion beam (FIB) milling method to produce pre-existing cracks with a tip radius as small as 50 nm. The fracture behavior of ultrananocrystalline diamond (UNCD) developed at Argonne National Laboratory has been investigated using this methodology. 15 UNCD thin film membranes with the gauge size of 200 μm in length, 20 μm in width, and 800 nm in thickness were tested under tensile stress until failure. Measured fracture toughness (Kc) values were found to be 6.8-7.3 MPa$sqrt$m with the crack size varying from 1 —*mu*m to 5 μm, indicating that the fracture toughness is independent of the geometry.

SESSION U5: Poster Session
Chairs: Sean Corcoran and Neville Moody
Tuesday Evening, December 2, 2003
8:00 PM
Exhibition Hall D (Hynes)

U5.1
Evolution of Stress-Induced Surface Damage at Elevated Temperatures. Soo-Jung Hwang[1,2], Young-Chang Joo[1] and Junichi Koike[2]; [1]School of Materials Science & Engineering, Seoul National University, Seoul 151-742, South Korea; [2]Dept. of Materials Science & Engineering, Tohoku University, Sendai 980-8579, Japan.

During fabrication processes of integrated circuits, interconnect materials are subject to many thermal cycles. Difference in thermal expansion among the various materials in the integrated circuits gives rise to a large thermal stress, which induces stress-induced damage in metal films. Thus, understanding and controlling the stress-induced damage is important for the reliability of interconnect material. In this work, we investigated surface grooving and its relation to the relaxation of thermal stress. Cu films were electroplated to a thickness of 1 μm on TaN barrier (450 Å)/oxidized Si wafer. Some films were detached from the substrate and annealed as free-standing films in order to investigate the effect of substrate. Stress of Cu films with substrate was measured in a temperature range from R. T. to 435 oC. Isothermal annealing for Cu films with substrate was carried out at given temperature of heating and cooling stage. Stress change was measured by a wafer-curvature machine. Surface morphology was observed by a scanning electron microscope (SEM). SEM observation showed that deep grooves were formed in Cu with the substrate, whereas shallow grooves were formed in Cu without the substrate. The results indicate that grooving can be enhanced by thermal stress. The stress change with time was found to be fit well by an exponential decay function. Two relaxation processes were observed in the heating stage with the characteristic time constants of $\tau1$=0.01~1 h and $\tau2$=1~10 h. In contrast, only a single relaxation process with τ=1~10 h was observed in the cooling stage. The obtained results suggest that the evolution of stress-induced grooving can be ascribed to the rapid stress relaxation of compressive stress observed only in the heating stage. Detailed mechanisms will be discussed at presentation.

U5.2
Thickness and Temperature Dependence of Stress Relaxation in Nanoscale Aluminum Films. Seungmin Hyun, Walter Browm and Richard Vinci; MS&E, Lehigh University, Bethlehem, Pennsylvania.

We have found that the stress relaxation behavior of nanoscale Al thin films is strongly dependent on both film thickness and temperature. Films of 33, 107 and 205nm prepared by evaporation onto a silicon nitride membrane substrate were studied using a unique resonance system. A single thermal cycle to 300°C was used to establish a stress, after which the time dependence of the stress relaxation was measured for the three film thicknesses at 50, 75 and 100°C. The relaxation rate is highest for the highest temperature and the thinnest film. The time dependence in all cases is well described by a power law expression and is attributed to dislocation motion. A dislocation locking mechanism is suggested as a possible explanation for the observed thickness dependence.

U5.3
Abstract Withdrawn

U5.4
Non-destructive recognition of defects in semiconductor wafers. Petra Feichtinger[1], Matthew Wormington[1], Christine H Russell[1], Frank Hofmann[1], David E Joyce[2] and Keith D Bowen[1]; [1]Bede Scientific Inc, Englewood, Colorado; [2]Bede Scientific Instruments Ltd, Durham, England, United Kingdom.

Defect recognition in early stages of processing allows wafers to be sorted based on their potential to meet final product quality requirements. Identification of residual mechanical edge damage, tilted domains, misfit dislocations and similar bulk or interfacial defects could potentially provide significant cost savings in wafer manufacturing lines. These defects may act as sufficient stress concentrators during subsequent device processing steps to introduce strain relief, causing device failure or even wafer breakage. A standard non-destructive characterization method for such defects involves mapping the X-ray diffraction (XRD) full width at half maximum (FWHM) across the wafer surface. Scans producing larger FWHM values indicate regions of higher defect density. X-ray topography (XRT) qualifies as an interesting alternative metrology technique. In XRT, defects are directly imaged by measuring changes in the diffracted X-ray intensity across a wafer due to lattice parameter changes (strain) and/or tilt introduced into the crystal lattice by the defects. Rapid, convenient defect identification via XRT in a manufacturing environment has so far been aggravated by the complicated operation of conventional topography tools. We present a fully digital high-speed topography system that can examine up to 300mm diameter single crystals, such as semiconductor substrate wafers and epitaxial thin film stacks. The defect contrast is similar to white beam Synchrotron topography, although a laboratory X-ray source is employed. Either reflection or transmission geometry can be chosen. The method offers substantial advantages over film methods in convenience and the application of digital image processing techniques. We are presenting extraction of additional information, such as orientation maps to quantify the plastic wafer deformation.

U5.5
Thickness dependence of tensile stress in PVD chromium films. S.Yu. Grachev[1] and G.C.A.M. Janssen[2]; [1]NIMR, Delft, Netherlands; [2]Materials Science, Delft University of Technology, Delft, Netherlands.

Intrinsic stress in polycrystalline films can either be tensile or compressive. The compressive part is due to ion peening. The tensile part is due to the columnar microstructure which induces grain boundary shrinkage leading to lateral strain and thus to stress. We investigated tensile stress in PVD chromium films as a function of film thickness. The stress was measured by wafer curvature. We correlate the stress evolution to the grain growth in the polycrystalline films. Both grain growth and stress evolution obey the same power law dependence on thickness. From this observation we conclude that the tensile stress is generated at the grain boundaries. We performed experiments in an industrial PVD reactor, laid out to coat 3-D objects as well as in a parallel plate reactor designed to coat Si-wafers. For both pieces of equipment a power law dependence of the stress on the film thickness was obtained. However for the industrial reactor the power of the exponent varied from 0.25 to 0.8 up on varying the gas pressure during sputtering from $5 \cdot 10^{-3}$ to $2 \cdot 10^{-2}$ mbar. In the parallel plate reactor varying of the pressure over this range had no effect whatsoever. Here the power of the exponent remained 0.57. The results are discussed with respect to the average number of collisions an atom makes between target and substrate, the extension of the plasma in front of the target into the deposition chamber and the resulting angular distribution of arrival angles of the Cr atoms on the substrate.

U5.6
The Formation of Nano-Structures on the Stranski-Krastanow Systems By Surface Diffusion. Cheng-hsin Chiu and Zhijun Huang; Materials Science, National University of Singapore, Singapore, Singapore.

In this talk we present our theoretical investigation of the formation of nano-crystalline structures on the surfaces of Stranski-Krastanow (SK) film-substrate systems during the self-assembly process. Of particular interest here is a mechanism that can explain why similar film-substrate systems can follow the various kinetic pathways and develop distinct nano-structures. For example, the SiGe film in experiments can form separate pyramid islands, a network of ridges, and structures growing into the film such as the pits and the trenches. The study was carried out by three-dimensional numerical simulation for the morphological evolution of the islands driven by surface diffusion. The simulation was based on a continuum model, which allows us to examine the effects of the film thickness, the mismatch strains in the film, and the anisotropic surface energy density. Our results demonstrate that though there are apparent differences in the formation process, the formation follows a general sequence: the growth of an undulating film surface, the shape transition from a smooth surface profile to facet structures, and the development of a wetting layer. The development of the wetting layer is the critical process leading to the different kinetic pathways and the various nano-structures of the system. The results explain the contrasting findings of the SiGe film reported in the literature. The results also shed new light on the relation between the nano-structures formation and the energetic forces of the morphological evolution.

U5.7
The Nanostructure Evolution during and after Magnetron Deposition of Au Films. Norbert Schell[1,2], T. Jensen[3], J.H. Petersen[3], K.P. Andreasen[3], J. Bottiger[3] and J. Chevallier[3]; [1]ROBL-CRG, Forschungszentrum Rossendorf, Grenoble, France; [2]Institute of Ion Beam Physics and Materials Research, Forschungszentrum Rossendorf, Dresden, Germany; [3]Department of Physics and Astronomy, University of Aarhus, Aarhus, Denmark.

The evolution of the nanostructure of magnetron sputtered Au films has been experimentally studied. At a synchrotron-radiation beam line, during growth and subsequent annealing, in-situ x-ray diffraction has been carried out to follow the texture, the grain size, the microstrain and lattice-plane distances. With Bragg-Brentano geometry, only (111) grains, having a (111) plane parallel to the film surface, have been observed, while, with glancing incidence and exit x-ray diffraction, (111)* grains, having one of their (111) planes perpendicular to the film surface, have also been observed. Both during growth and subsequent annealing, the (111) texture changed, and some (111)* grains recrystallized and/or the orientation of the grains changed. The microstrain decreased drastically during the first few minutes of growth (30 nm) while, simultaneously, the size of the coherently diffracting domains increased. Subsequently, the microstrain became constant, and the rate of increase of the size of the coherently diffracting domains levelled off. Initially, during the first few minutes of annealing, a large decrease in the microstrain was observed simultaneously with a dramatic rise of the size of the coherently diffracting domains. After this initial annealing period, as during film growth, the microstrain became constant, and the rate of increase of the size of the coherently diffracting domains levelled off. The activation energy for the initial growth of the coherent diffracting domains was found to be $Q = 0.25\pm0.03$ eV, and the activation energy for normal grain growth was found to be $Q = 0.99\pm0.04$ eV. Finally, the thin-film stress was followed during growth and subsequent annealing. A tensile contribution to the stress was observed during island coalescence.

U5.8
Stress Relaxation Thickness Dependencies with the Onset of $L1_0$ Ordering in FePt Thin Films. Kurt W Wierman, Christopher L Platt and James K Howard; Materials, Seagate Research, Pittsburgh, Pennsylvania.

During the chemical ordering of the high anisotropy FePt $L1_0$ phase an fcc$\rightarrow$ fct distortion in the unit cell occurs. The strains associated with this transformation are $-$ 1.5% in the <101> and $-$ 1.0% in the <111> directions, which significantly modify the internal stress state of the film. Monitoring the evolution of stress during the onset of the $L1_0$ phase transformation may lead to a better understanding of the thickness dependencies of the $L1_0$ordering kinetics. The stress in the sputter deposited $Fe_{51}Pt_{49}$ (50, 25, 10nm) films were measured using a laser-based wafer curvature technique during 6 hour isothermal anneals at various temperatures ranging from 267^{0}C to 330^{0}C. As-deposited films were fcc polycrystalline with a strong (111) fiber texture with an initial compressive stress of approximately 930, 840, 615 MPa, respectively, for each targeted thickness/. During the 10^{0}C/min substrate heating and cooling temperature transitions the stress varied nearly linearly due to thermal expansion effects. During the isothermal portion of the anneal cycle all films showed an exponential decaying stress relaxation with time to lower compressive stress states for all temperatures and thickness/. The stress relaxation rate increased significantly with an increase in FePt film thickness and annealing temperature. This correlated well with increases in post annealed Hc film measurements, the increasing presence of the (001) forbidden $L1_0$ peak, and the shifting of the (111) peak position, all indications of $L1_0$ chemical ordering transformation.

U5.9
Single-Crystal and Nano-Columnar Growth of Gadolinium-Doped Ceria Thin Films on Oxide Substrates Studied Using Electron Microscopy. Daxiang Huang, Chonglin Chen, Ling Chen and Allan Jacobson; Chemistry, University of Houston, Houston, Texas.

Gadolinium-doped ceria (GDC) is one of the most promising electrolyte materials for replacing the commonly-used yttrium stabilized zirconia (YSZ) in solid oxide fuel cell (SOFC) applications at low operating temperatures due to its high ionic conductivity. Thin film electrolytes have been considered because of the advantage associated with lowering ohmic losses. Therefore, recently we have systematically investigated the epitaxial growth of GDC thin films on various oxide single crystal substrates including LAO, STO, MgO, YSZ, and NGO. Due the different lattice mismatch levels for the GDC films grown on different substrates, the thin film microstructural features are much different. Single crystal GDC thin films have been obtained for the first time on LAO substrates. A series of nano-columnar GDC epitaxial thin films are formed on other oxide substrates. In this study, we report the details of the thin film microstructures determined by using transmission electron microscopy. The induced strain fields in the thin films due to the film/substrate lattice mismatch have been analyzed in all samples. The strain field relaxation mechanisms during the thin film growth and their influences on the final thin film microstructures will be discussed.

U5.10
Strain control in $SrRuO_3$ thin films by using a lattice constant tunable buffer. Kota Terai[1], Tsuyoshi Ohnishi[2], Mikk Lippmaa[2], Hideomi Koinuma[3] and Masashi Kawasaki[4]; [1]Synchrotron Radiation Research Center, Japan Atomic Energy Research Institute, Sayo-gun, Hyogo, Japan; [2]Institute for Solid State Physics, University of Tokyo, Kashiwa, Chiba, Japan; [3]Materials and Structures Laboratory, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; [4]Institute for Materials Research, Tohoku University, Sendai, Miyagi, Japan.

The physical properties of transition metal oxide thin films can change dramatically due to the in-plane strain imposed by a substrate crystal. We have developed a process to fabricate buffers with a tunable lattice constants from 3.9 to 4.0$\sim\AA$, which can be used for growing strain-controlled films. In this report, we demonstrate the use of tunable buffers by growing strain-tuned $SrRuO_3$ (SRO) thin films. We start by growing a $Ba_{1-x}Sr_xTiO_3$ (BSTO : x = 0.75, 0.5) / $BaTiO_3$ (BTO) bilayer buffer on $SrTiO_3$ (STO) by pulsed laser deposition at a temperature of 650°C, followed by annealing at 1200°C. The thicknesses of the two layers in the buffer are 900$\sim\AA$ and 120$\sim\AA$. We deposited 360$\sim\AA$-thick SRO films on these buffers at 650°C and, for comparison, a SRO film was also grown directly on a STO single crystal substrate under identical growth conditions. Reciprocal-space mapping showed that the SRO thin film on STO was compressively strained, while a SRO film on a BSTO(x=0.5)/BTO buffer was nearly strain free. The in-plane lattice constant of the SRO film changed from 3.91$\AA$ to 3.94$\AA$ when a BSTO(x=0.5)/BTO buffer was used. The magnetic properties of the films were measured with a SQUID magnetometer. The magnetic hysteresis curves indicated that the magnetic easy axis of a SRO film on STO was in the out-of-plane direction. The easy axis of a strain-free SRO film on a BSTO/BTO buffer was oriented along the in-plane direction. In addition, a Tc shift to higher temperature was observed in unstrained films. The in-plane lattice constant of SRO could be changed by using suitable buffers and, as the magnetic measurements show, buffers with tunable lattice constants are an effective tool for the modification of strain-related physical properties of thin films.

U5.11
Thermal Stability and Internal Stress for Strongly Oriented (111) Cu Films. Shinji Takayama and Tokuji Himuro; Systems and Control Engineering, Hosei University, Tokyo, Japan.

Resistivities, internal stresses and thermal stability of strongly (111) oriented Cu thin films, which are one of promissing interconnect materials in advanced ULSI devices, have been studied comparing with those of non-oriented Cu films. Their internal stresses within a film plain were measured by a conventional X-ray diffraction technique, while the strain distribution with depth by a grazing incidence X-ray scattering (GIXS) methods. The resistivity and X-ray diffraction measurements revealed that present strongly (111) oriented Cu films showed high thermal stability, compared with non-oriented Cu films. The internal stresses of both highly oriented and non-oriented Cu films rapidly increased at first with annealing temperatures due to the difference of thermal expnasion between Si substrates and Cu films. Then, they started to decrease above 200°C and 250°C, respectively associated with a large stress relaxation. It was revealed that some extrusions like hillocks of Al films and also wedge-shaped voids started to appear at the non-oriented Cu film surfaces at the temperatures where a large stress relaxation takes place. Both internal stress and hardness for the strongly (111) oriented Cu films increased with decrease of film thickness, indicating that their yield stresses increased with decrease of thickness. It was found that the residual internal stresses of highly oriented Cu films increased almost linearly throughout the thickness up to the oxidized Si substrate. The change of the stress distribution with depth was measured as a function of annealing temperatures for strongly oriented Cu films. It was revealed that the change of the internal stresses with depth was nearly the same as that of plain stresses within the film plain on aneealing.

U5.12
Stress and Defect Generation in Si Epitaxy. Tien Yang Wang and Douglas Carlson; Microwave Solutions Business Unit, M/A-COM, Inc., Burlington, Massachusetts.

A quasi-static model is proposed to describe wafer deformation due to stress build up in the Si epitaxy process. The analysis takes into account temporal stress variations as the wafer sags into the dish-shaped pocket of the susceptor at a deposition temperature above 1100 oC. It is shown that the magnitude of the negative bending moment at the wafer supporting edge decreases over time. As a result, the maximum deflection at the wafer center and the tensile stress at the wafer supporting edge also decreases over time. The generation of slip defects due to shear stress build up near the wafer edge as the wafer responds the thermal, gravitational and contact stresses is explored. A new test methodology is developed to track the time-varying wafer deflection. It allows for the measurement of quality of the susceptor and its fitness for use in the reactor system.

U5.13
Stress measurements during GaN heteroepitaxy.
Armin Dadgar, Gunther Strassburger, Till Riemann, Juergen Blaesing, Juergen Christen, Rainer Clos and Alois Krost; FNW/IEP, Otto-von-Guericke-Universitaet Magdeburg, Magdeburg, Germany.

We present an in-situ study of stress evolution during different stages of GaN MOVPE growth on Silicon. For this we apply a self-made optical stress sensor with vertical incidence on the sample surface suited for MOVPE reactors with small windows. After the inital stages of GaN heteroepitaxy on Si using a low-temperature deposited AlN seed layer we observe tensile stress likely originating from island coalescence. this tensile stress can be reduced by an in-situ deposited SiN mask which leads to increased grain size and thus less coalescence boundaries. By this method stress is indeed not only reduced but even gets slightly compressive which we attribute to compressive stress originating in the freestanding islands before coalescence. Silicon doping of GaN is another source of tensile stress which can be also reduced by SiN in-situ masks. A procedure is given how the stress is determined from the curvature measurements. We further report on the impact of low-temperature deposited AlN layers for strain compensation.

U5.14
The Effects of Biaxial Misfit Stress On Interdiffusion Rates in Si/SiGe Epitaxial Thin Films. Daniel B Aubertine, Nevran Ozguven and Paul C McIntyre; Materials Science and Engineering, Stanford University, Stanford, California.

It is expected that biaxial film stress should influence the kinetics of interdiffusion at Si/SiGe interfaces because, in general, stress affects both the driving force for interdiffusion and the magnitude of the interdiffusivity (through the activation strain tensor). However, no consensus on the magnitude and nature of these effects can be drawn from reported experimental data for interdiffusion in this technologically important semiconductor material system. We present systematic measurements of the effects of biaxial misfit stress on interdiffusion rates based on a novel strategy utilizing symmetric and non-symmetric x-ray diffraction from fully coherent and strain-relaxed $Si_{0.94}Ge_{0.06}/Si_{0.92}Ge_{0.08}$ superlattices. The SiGe interdiffusivity in these structures can be extracted directly from x-ray measurements by monitoring the decay rate of superlattice satellite peaks about the 004 Bragg reflection as a function of annealing time. Comparing the interdiffusivity values for a range of temperatures between 795°C and 870°C, we find that the activation enthalpy varies by at most 0.1 eV for a change in strain of 0.0022. This places an upper bound of 0.26 eV/GPa on the stress dependence for the activation enthalpy of SiGe interdiffusivity.

U5.15
Stress and Strain in CeO_2 Thin Films on Si. Anna Kossoy[1], Yishay Feldman[1], Ellen Wachtel Ellen Wachtel[2] and Igor Lubomirsky[1]; [1]Materials & Interfaces, Weizmann Institute, Rehovot, Israel; [2]Research Support, Weizmann Institute, Rehovot, Israel.

Cerium oxide is a promising material for a number of applications ranging from high dielectric constant layers to fuel cells. The slow chemical reaction between CeO_2 and Si may also permit applications of CeO_{2-x} based membranes integrated in Si-based microelectromechanical systems (MEMS). However, CeO_2 is known to form a number of marginally stable oxygen deficient phases. The stress and strain in these films may undergo spontaneous changes due to phase transformation between the polymorphs. We have investigated the phase transformation between triclinic $Ce_{11}O_{20-y}$ and cubic CeO_{2-x} phases. Films were deposited on Si by RF sputtering of a pure CeO_2 target in Ar. Depending on the sputtering conditions the as-deposited films contained one or both of the phases. It was found that the lattice parameter decreases with time during the course of days, followed by an abrupt increase. The change in the lattice parameter is accompanied by changes in the in-plane compressive stress. Changes of the lattice parameter were observed not only in films exposed to oxygen but also in encapsulated films. This indicates that, in practice, CeO_{2-x} and $Ce_{11}O_{20-y}$ phases cannot coexist. All experimental observations agree with the hypothesis of an order-disorder transition of oxygen vacancies and demonstrate that this transformation can be driven by oxygen diffusion from CeO_{2-x} to $Ce_{11}O_{20-y}$ at room temperature. Since phase transformations in cerium oxide films are accompanied by large changes in the in-plane stress, they must be taken into account for practical applications.

U5.16
Residual Tensile Stresses in Homoepitaxial Electron-Beam Deposited Yttria-Stabilized Zirconia (YSZ) Thin Films.
Jason Draut and Frans Spaepen; Harvard University, Cambridge, Massachusetts.

We deposited yttria-stabilized zirconia (9.5 mol% yttria) films onto heated single crystal YSZ (100) substrates of the same composition using electron beam evaporation. The stress in the film was measured *insitu* from the curvature of the cantilevered substrate. The substrate temperatures ranged from 400°C to 800°C. Tensile stresses were observed which decreased with increasing substrate temperature. All films were perfectly epitaxial single crystals. Cross-sectional and plan view transmission electron microscopy showed a columnar structure defined by periodic void arrays (period of approximately 100 Å). We attribute the formation of the columns to the faceting observed at the surface and propose a mechanism that relates the tensile stress to this void structure.

U5.17
Evaluation on Stress and Optical Property of Thin Films Used in Optical MEMS Device. Lianchao Sun[1] and Ping Hou[2,3]; [1]SOPRA Inc, Acton, Massachusetts; [2]Nortel Networks Inc., Wilmington, Massachusetts; [3]SuperPower Inc., Schenectady, New York.

Control of film stress and optical property has long been considered as an issue in tunable optical MEMS devices. In this paper, the atmospheric evolution of TiO2 and SiO2 thin films for optical MEMS devices were studied. These films were prepared with ion-assisted e-beam evaporation. It is found that as-deposited SiO2 films exhibit compressive stress; whereas, TiO2 film stress could be tuned from compressive to tensile, depending on processing parameters. When annealed at 150 oC, both SiO2 and TiO2 films show little change of stress with annealing time. However, increasing the annealing temperature to 250 oC caused an obvious change of film stresses with time, in which SiO2 film turns into less compressive and TiO2 film appears to be more tensile. The relationship between the film stress and optical properties was investigated through the technique of spectroscopic ellipsometry analyses. At both experimental temperatures, the film thickness increases slightly and the refractive index at 1550 nm decreases a little at the initial annealing phase for SiO2 film.The optical homogeneity of TiO2 films was also briefly addressed in this paper.

U5.18
In situ growth of gold films on amorphous Al_2O_3- and crystalline TiO_2-substrates. Lukas Lauter and Reinhard Abermann; Institute of Physical Chemistry, University of Innsbruck, Innsbruck, Austria.

The growth of thin gold films on amorphous Al_2O_3- and highly crystalline TiO_2-substrates and its dependence on substrate temperature was investigated under UHV-conditions by in situ internal stress measurement based on the cantilever beam principle. When depositing gold on amorphous Al_2O_3 a tensile stress is built up during the initial growth stage which reaches a maximum at a mean thickness at which the film becomes continuous. As the film deposition proceeds the recorded stress is compressive. After the end of deposition a tensile stress change is observed with time. This typical stress vs. thickness curve (type II) is characteristic of the growth of polycrystalline films with pronounced island growth during the initial growth stage (Volmer-Weber growth). Raising the substrate temperature from 300K to 573K increases the thickness at which the gold film becomes completely continuous from 9 nm to 112 nm. All the concurrent changes in the stress vs. thickness curve fully confirm the assumptions made in a model for the origin of film growth stress published earlier. On crystalline TiO_2-substrates two different growth regimes are indicated by the respective stress vs. thickness curves of gold. At substrate temperatures below 473K the growth of gold on this crystalline substrate is more or less identical with that on amorphous Al_2O_3. At temperatures above 473K a large incremental tensile stress is built up during deposition of the first few monolayers, which subsequently becomes constant at a mean film thickness of 2 nm, probably due to the insertion of dislocations and/or vacancies in this fairly thin interface layer. As the film thickness is further increased a type II stress curve is superimposed. This novel stress vs. thickness curve is interpreted to indicate Stranski-Krastanov growth of gold on crystalline TiO_2 at elevated substrate temperatures. The microstructure deduced from the the in situ internal stress measurement is fully confirmed by that seen in the electron microscope.

U5.19
Mechanical properties of organic/inorganic hybrid coatings. Alan Atkinson and Malcolm R Robertson; Materials, Imperial College, London, SW7 2AZ, United Kingdom.

Organic/inorganic hybrid polymers have application as thermally resistant and abrasion resistant coatings since they have better performance than organic coatings yet can be processed at relatively low temperatures. We have studied a range of such hybrids produced by the sol-gel route from tri-functional organo-silanes $R\text{-}Si(OR\prime)_3$, where R is methyl, methacryloxy-propyl or glycidoxy-propyl. The coatings were applied on glass, stainless steel and polyester-coated steel substrates. The coatings were cured either thermally, with amine (for the epoxy) or with UV radiation (for the acrylate). The residual stress during processing was deduced by measuring the deformation of thin glass substrates to which the coating was applied and their elastic and plastic deformation studied using nano-indentation. TEOS or colloidal silica (nano-particles) was added to increase the inorganic content and di-functional organo-silanes to reduce the cross-linking of the inorganic network. The coatings display a wide range of mechanical properties, and the nano-indentation response varies from mainly elastic to elasto-plastic. All show time-dependent visco-plasticity to some degree and the apparent properties vary with indentation depth. This is partly due to perturbation from the underlying substrate. Elastic moduli are in the range 0.4 to 15 GPa and hardness 0.02 to 1.6 GPa. Tensile residual stress in high inorganic content films cured at high temperature reaches approximately 110 MPa with a fracture energy of approximately 1.2 J m^{-2}. The dependence of mechanical properties on composition and processing is interpreted in terms of the cross link density in the hybrid and the volume fraction of colloidal nano-particles.

U5.20
Tailoring Properties of Polyimide by Ion Irradiation.
Thomas E. Felter, M. L. Anthamatten and Sergei O. Kucheyev; Lawrence Livermore National Laboratory, Livermore, California.

Polyimides are high performance plastic materials with an attractive combination of mechanical, electrical, chemical, and thermal properties. For example, the most well studied polyimide, Kapton^{TM}$, is noted for its wide operating temperature range (4 – 675 K) and a high tensile strength of 230 MPa. Moreover, polyimides can be conveniently cast from one or two components into flat films or vapor deposited into complicated shapes. Due to these properties, polyimides have been used in the fabrication of complex spherical targets for inertial confinement fusion (ICF) at LLNL. However, for the next generation of ICF targets, polyimides with improved mechanical properties are highly desirable. In this presentation, we show how irradiation with MeV light ions can change mechanical, optical, and thermal properties of polyimide foils. We use a combination of the following characterization techniques: Instron^{TM} tensile test, nanoindentation, Fourier transform infrared (FTIR) spectroscopy, UV-visible absorption spectroscopy, differential scanning calorimetry, thermogravimetric analysis, and scanning near-field optical microscopy. Changes to polyimides by ion irradiation are attributed to increased cross-linking and sp^{2} to sp^{3} bond conversion. This work was performed under the auspices of the U. S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

U5.21
Adhesion Induced Instability in Thin Elastic Film A. Ghatak, L. Mahadevan, M. K. Chaudhury. Animangsu Ghatak[1], L Mahadevan[1] and M K Chaudhury[2]; [1]CMS, Cambridge University, Cambridge, United Kingdom; [2]Chemical engineering, Lehigh University, Bethlehem, PA, Pennsylvania.

Surface of a confined thin elastic film develops undulations almost instantaneously when subjected to forces of adhesion. The instabilities develop in the form of fingers when a flexible plate is brought in contact with a layer of elastic adhesive bonded to a rigid substrate or is peeled from it. The characteristic wavelengths of the patterns remain independent of all material and geometrical properties of the system except the thickness of the film. This instability contrast sharply the Saffman-Taylor type phenomena in which the length scale of instability patterns depends on the viscous and surface tension forces in addition to the thickness of the liquid film. The instability reported here is rather akin to the Rayleigh-Kelvin instability with curved liquid meniscus, as in both cases, surface force triggers the instability while not deciding its wavelength. The instability also throws light on cavitation phenomena in confined adhesive layers. Patterns of instability suggest that cavitation in elastic film is guided by the same physics that determines also the fingering instability. A theoretical model predicts qualitatively the important morphological features of the instability phenomenon

U5.22
Abstract Withdrawn

U5.23
Formability of ultra-thin plasma-polymer films deposited on metal sheet: mesoscopic and nanoscopic aspects of defect formation. Michael Rohwerder, Birgit Baumert and Martin Stratmann; Max-Planck-Institute for Iron Research, Dusseldorf, Germany.

Forming of pre-coated metal sheet becomes of increasing importance in various fields of industrial application. For instance, the trend in automotive industry goes for ready coated steel sheet that can be formed and cut without loss of performance. As a first step into this direction, in the recent years ultra-thin plasma-polymer films have emerged as candidates for substituting phosphatation and the environmentally critical chromatation as pre-treatments for steel sheets. This paper deals with the mesoscopic and nanoscopic defect formation in such ultra-thin plasma-polymer films induced by tensile stretching, as observed with in-situ AFM and FEG-SEM. Fundamental studies on ultra-thin plasma-polymer films have been performed on both, technical galvanised steel substrates and model-substrates. The in situ AFM experiments have been performed with a special linear stretching device which combines with an Atomic Force Microscope (AFM). In this paper a classification of diverse micro- and nano-defects will be presented, which occur on both model and technical samples, when the linear stress is applied. Of special interest is the influence of the forming behaviour of the different substrates on the defect formation in the plasma-polymer films. The plasma-polymer-coatings have been varied, in thickness and chemical composition. The influence of these parameters on localized de-adhesion, as well as on the crack and fracture behaviour of these ultra-thin plasma-polymer films will be discussed. Furthermore first results of the electro-chemical relevance of these nano-sized defects will be presented.

U5.24
Surface Hydrogenation of KD_2PO_4 Nonlinear Crystals.
Sergei O. Kucheyev, T. E. Felter, W. J. Siekhaus, A. J. Nelson, P. K. Whitman and T. A. Land; Lawrence Livermore National Laboratory, Livermore, California.

Normal and deuterated potassium dihydrogen phosphate single crystals (KH_2PO_4 and $KD_{2x}H_{2(1-x)}PO_4$, also called KDP and DKDP) are perhaps the best studied hydrogen bonded ferroelectrics. Recent significant interest in the growth and processing of large KDP and DKDP has been stimulated by their use as optical switches and for frequency conversion in large-aperture laser systems. One severe problem of DKDP crystals is related to their aging at ambient

conditions, resulting in the development of surface cracks. Such surface cracking has been attributed to deleterious effects of diffusion-limited deuterium–hydrogen (D/H) exchange and an associated buildup of tensile stress in near-surface layers. Here, we present a systematic study of the D/H exchange and lattice stress and their effects on the mechanical properties of DKDP. We use a combination of elastic recoil detection analysis (ERDA), Rutherford backscattering/channeling (RBS/C), particle-induced x-ray emission/channeling (PIXE/C), and spherical nanoindentation techniques to study how sample aging and the parameters of chemo-mechanical polishing affect the composition, stress, and mechanical properties (with a depth resolution of several nanometers) of near-surface layers of DKDP crystals. This study may have important technological implications for the estimation of the effects of surface preparation parameters and aging on the near-surface properties of DKDP. This work was performed under the auspices of the U.S. Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

U5.25

Optimization-Based Characterization Method for Visco-Elastic/Plastic Materials Using Rheological Constitutive Models and the Finite Element Method in the Hemispherical Nanondentation Geometry. Timothy C. Ovaert[1] and Jianjun Wang[2]; [1]Aerospace and Mechanical Engineering, University of Notre Dame, Notre Dame, Indiana; [2]ABB Robotics, Inc., Windsor, Connecticut.

To meet the need for identifying the mechanical properties of visco-elastic/plastic materials in the nanoindentation test, a numerical optimization-based approach is proposed. This approach essentially utilizes inverse engineering and formulates the material characterization problem as an optimization problem. The objective function of the formulated optimization is the least-square difference between the finite element derived indentation simulation results and the experimental indentation data. It is well known that this optimization problem is difficult to solve due to the nonlinear nature of the load vs. displacement relation, as well as the heavy computation load from the finite element simulation. This work addresses these difficulties from four practical aspects: sensitivity analysis to provide gradient information, heuristic or neural network based estimation method to obtain the initial guess, a gradient-based optimization algorithm for fast convergence, and smart experimental design to extract some of the material properties directly from the test data. The proposed optimization-based method has been successfully applied viscous-elastic/plastic materials. The rheological model for this class of materials is a linear dashpot in parallel with a series combination of linear spring and slider elements. With the help of a heuristic estimation method and an optimization algorithm, the four unknown material constants may be identified with speed and efficiency.

U5.26

Investigation of mechanical properties of thin films mesoporous Methylsilesquioxane Materials.

Sylvain Maitrejean[1], Marc Verdier[2] and Gerard Passemard[3]; [1]CEA-LETI, Grenoble, France; [2]LTPCM-CNRS, Grenoble, France; [3]STMicroelectronics, Grenoble, France.

In ULSI circuit, interlayer thin film with low dielectric constant shall be used in order to reduce the interconnection RC delay. One promising material is methylsilesquioxane (MSQ) deposited by spin coating with controlled porosity. High porosity combined with matrix weakness result in low mechanical properties that could be detrimental to circuit reliability. In this study we have investigated deformation of mesoporous MSQ thin films by nanoindentation and by AFM observations of residual indents. At first, proper measurements conditions were explored by modulating the film thickness from 100 nm up to 600nm: the very low elastic modulus linked with an intrinsic brittleness of the material requires great care in the raw data analysis. Indeed a careful surface contact determination shall be made. An attempt to extract the low elastic modulus from the composite film-substrate behaviour was made. Based on this, the effect of film porosity on the modulus and hardness of the film were obtained. To rationalize the results, Ashby type law for foams were used.

U5.27

The Mechanical Properties Of Borazine-Siloxane Polymer Thin Films. Masami Inoue, Hiroshi Yanazawa, ASET, Yokohama, Kanagawa, JAPAN Yuko Uchimaru, AIST, Tsukuba, Ibaraki, JAPAN. Inoue Masami, Yokohama research center, ASET, Yokohama, Kanagawa, Japan.

Application of new materials as low-k interlayer dielectrics makes it necessary to understand the mechanical behavior of these thin films. We have succeeded in the development of the organic/inorganic hybrid polymer containing borazine and siloxane units alternatively. Borazine-siloxane polymer can be synthesized by hydrosililation polymerization of borazine compounds such as B,B',B"-triethynyl-N,N',N"-trimethyl-borazine with silicon compounds such as tetramethylcyclosiloxane (1:1mol/mol). Polymerization under diluted condition gave a homogeneous solution of the linear polymer at room temperature or 40C. From the polymer solution, a thin film of linear polymer can be made on a silicon wafer by spin-coating. Upon continued annealing at 200 400C, the thin film became to be network structure caused by intermolecular crosslinking. The dielectric constants of these thin films were evaluated to be 2.8 2.2. Howeve, these mechanical properties were not so good as well as low-k porous silica and organic polymer thin films. We tried to improve the mechanical properties by increasing crosslinking density, which depends on annealing temperature, molecular weight of prepolymer and functional group of borazine compound for hydrosililation. The mechanical properties were appreciated by hardness and elastic modulus detarmined by nanoindentation mehtod. Hardness and elastic modulus increased slightly with increasing molecular weight and annealing temperature. The introduction of more reactive functional group into borazine ring was most effective, therefore, the resulting thin films had good mechanical properties, that is, hardness is 1GPa and elastic modulus is 15GPa.

U5.28

Mechanical Properties of Partially Carbonized Polyimide Films. Hiromi Abe, Takayuki Hirai, Yoshiki Takagi, Yoshiaki Kogure and Masao Doyama; Environmental Materials, Teikyo University of Science & Technology, Uenohara, Yamanashi, Japan.

Aromatic polyimide is a good temperature resistant insulator and completely carbonized aromatic polyimide is the graphite, which is a semi-metal. Aromatic polyimide films were partially carbonized between 600 and 1000 centigrade. The temperature dependence of the electric conductivity, and the Hall coefficient of these materials have been measured, which showed semiconductor-like properties. The derived carrier density and the carrier mobility of the material showed Arrhenius-type temperature dependence and activation energies for those properties increased with the carbonization temperatures. The mechanical properties of these materials are important for practical application of these materials. The Young's modulus and Raman spectra of these materials are measured to characterize the structural and mechanical properties. The Young's modulus is determined by measuring the displacement of bending and fitting the stress-strain relation to the cubic equation of the elasticity formula of finite displacement. We studied in detail by Raman spectroscopy through the evolution of D- and G-bands. The shapes of Raman spectra vary depending on carbonization temperatures, and were separated with the individual G- and D-band components of the fit. The detailed analysis of the relationship between microstructures of these materials and these components will be discussed.

U5.29

Engineering and Characterization of the Surface Properties of Ultrananocrystalline Diamond Thin Films.

Anirudha V. Sumant[1], David S. Grierson[1], Jennifer E. Gerbi[2], James P. Birrell[2], Orlando Auciello[2], John A. Carlisle[2] and Robert W Carpick[1]; [1]Engineering Physics, University of Wisconsin - Madison, Madison, Wisconsin; [2]Materials Science Division, Argonne National Laboratory, Argonne, Wisconsin.

Ultrananocrystalline diamond (UNCD) is a material that has many exceptional physical, electrical, chemical and mechanical/tribological properties that are nearly equivalent to or exceed those of natural diamond. These properties render it of great interest for applications ranging from microelectromechanical systems (MEMS) to biological implants to tribological coatings. However, little is known about the surface chemistry of this material, and how it will affect its performance, particularly in case of mechanical contacts at both the micro- and nano-scale. We have carried out detailed, systematic studies of the morphology, chemistry, and tribology of UNCD thin film surfaces. We have applied Auger electron spectroscopy (AES), near-edge x-ray absorption fine structure (NEXAFS), x-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and atomic force microscopy (AFM) to understand the chemical nature, phase, and micro/nanostructure of UNCD surfaces. We have found significant differences in the structural and chemical properties between the as-grown UNCD top surface and the underside of the film as revealed after etching away the substrate. Characterization of the underside of the film is particularly important in MEMS applications, since in many dynamic MEMS applications it is this side of the film that is the contacting surface. These studies also reveal detailed information on the initial nucleation and growth properties of UNCD, which can be tuned to control film mechanical properties such as surface roughness and adhesion. We will discuss how these properties are influenced by various aspects of the UNCD plasma-based growth process, including the initial nucleation pre-treatment and the gas chemistry used during growth, and how one can engineer the surface by tuning these growth parameters. Finally, we will discuss the consequences of such changes for key applications including UNCD-based MEMS devices. This work was supported by the US Department of Energy, BES-Materials Sciences, under Contracts DE-FG02-02ER46016 and W-13-109-ENG-38.

U5.30
Interaction Between Ferroelectric Phase Transition And Interfacial Dislocations In Thin Films. Yulan Li, Shenyang Hu and Long-Qing Chen; Department of Materials Science and Engineering, The Pennsylvania State University, University Park, Pennsylvania.

Interactions between interfacial dislocations and ferroelectric phase transition as well as ferroelectric domain evolution in thin ferroelectric films are studied. The interfacial dislocations are introduced through stress-free strains or eigenstrains, and a ferroeletric domain structure is described by an inhomogeneous distribution of spontaneous polarization. The phase-field approach is employed to simulate the movement of interfacial dislocations and domain structural evolution. Phase-field approach does not need to explicitly track the positions of the dislocations and the domain walls during evolution. The phase transition from a cubic paraelectric phase to a tetragonal ferroelectric phase is considered. Preliminary results showed that the paraelectric phase becomes unstable near dislocations close but above the bulk ferroelectric phase transition temperature, and the types of the simultaneous formed ferroelectric tetragonal variants depend on the directions of both the dislocation lines and their Burgers vectors. When the interfacial dislocations are immobile, the ferroelectric domains prefer to surround the existed dislocations. The effect of mobile interfacial dislocations on the domain structure evolution and the equilibrium locations of dislocations will be discussed in the present work.

U5.31
***InSitu*, Real-Time Curvature Imaging During Chemical Vapor Deposition.** David A. Boyd, Ashok B . Tripathi, Mohamed Y. El-Naggar and David G. Goodwin; Division of Engineering and Applied Science, California Institute of Technology, Pasadena, California.

Coherent Gradient Sensing (CGS) is a full-field optical technique that produces real-time images of macroscopic wafer curvature, which, for thin films, can be related to stress through Stoney's equation. Here we describe the use of CGS as an *insitu* diagnostic to observe film stress distributions during chemical vapor deposition. The application of this method to measure oxygen diffusion rates in thin-film $YBa_2Cu_3O_{6\delta}$ (YBCO) and stresses in thin film $Pb_xBa_{1-x}TiO_3$ (PBT) under chemical vapor deposition (CVD) conditions will be discussed.

SESSION U6: Processing and Structure
Chairs: Adrian Mann and Tom Shaw
Wednesday Morning, December 3, 2003
Room 210 (Hynes)

8:30 AM U6.1
Stress-driven growth of nanowires. Yang-Tse Cheng, Anita M. Weiner, Curtis A. Wong, Michael P Balogh and Michael J. Lukitsch; General Motors Research and Development Center, Warren, Michigan.

We recently discovered that compressive stresses in thin films can be exploited to grow nanowires [1]. Nanowires of bismuth (Bi) with diameters ranging from 30 to 200 nm and lengths up to several millimeters were formed spontaneously at the rate of a few micrometers per second at room temperature on surfaces of freshly grown composite thin films consisting of Bi and chrome-nitride (CrN). The high compressive stress in the composite thin films was shown to be the driving force responsible for the nanowire formation. In this presentation, the effects of stress, composition, and temperature on the growth and structure of nanowires of bismuth and other materials will be presented. [1] Yang-Tse Cheng, Anita M. Weiner, Curtis A. Wong, Michael P. Balogh, and Michael J. Lukitsch, Applied Physics Letters 81, 3248 (2002)

8:45 AM U6.2
Line Width Dependence of Stress and Microstructure in Damascene Cu Lines. Jong-Min Paik[1], Hyun Park[3], Young-Chang Joo[1] and Ki-Chul Park[2]; [1]School of Material Science & Engineering, Seoul National University, Seoul, South Korea; [2]Advanced Process Development Project Team, System LSI Division, Samsung Electronics Co., Ltd., Young-In, South Korea; [3]Center for Microstructure Science of Materials, Seoul National University, Seoul, South Korea.

Damascene Cu lines are usually deposited at room temperature by electroplating. During subsequent annealing and dielectric deposition process, thermal stress builds up in the lines due to thermal mismatches between different materials. In addition to thermal stress, stress caused by grain growth and defect annihilation, so called growth stress, can be developed since the microstructure of damascene Cu lines evolves under the confinement by dielectric materials. We analyzed the stress of damascene Cu lines varied in width from 0.13 to 2 μm using X-ray diffraction. 3-dimensional grain structures of the lines were quantified by the TEM cross-section images along and across the trench center. Three principle stresses showed the monotonous variation according to the line width. However abrupt change was observed in narrow lines. It is believed that the grain growth through the trench thickness is confined by the line width in the narrow lines and it affects the line width dependence of the growth stress. The contribution of thermal and growth stress to total stress was calculated using finite element method based on the measured average grain size and thermal history. By comparing measured stress and calculated stress obtained by finite element method, the line width dependence of stress in damascene Cu can be predicted.

9:00 AM U6.3
Stress Creation During Non-stochastic Island Coalescence. Sean J. Hearne[1], Jerrold A. Floro[1], Chris Dyck[1], Todd R. Christenson[1], Wenjun Fan[2] and S. R. J. Brueck[2]; [1]Sandia National Labs., Albuquerque, New Mexico; [2]University of New Mexico, Albuquerque, New Mexico.

It is generally accepted that island coalescence during thin film growth results in a tensile stress. However, prior to this work there had been no systematic demonstration of the functional stress generation behavior associated solely with the coalescence process. Using selective lateral growth of Ni films on monosized patterned substrates during electrodeposition, we have obtained direct comparisons of the experimentally-measured tensile stress due to island coalescence with theoretical predictions. This technique allowed for the systematic variation of island size and geometry while avoiding the complications associated with stochastic island coalescence that have plagued previous measurements obtained over the last 30 years. In the presentation, we will compare our experimentally measured results with those predicted by a Hertzian contact model recently proposed by Freund and Chason[1], and with 2-D and 3-D finite element models. This work was partially supported by the DOE Office of Basic Energy Sciences and by the ARO/MURI in Deep Subwavelength Optical Nanolithography. Sandia is a multiprogram laboratory of the United States Department of Energy operated by Sandia Corporation, a Lockheed Martin Company, under contract DE-AC04-94AL85000. 1 - L.B. Freund, E. Chason, JAP 89, 4866 (2001).

9:15 AM U6.4
Contact Mechanics of Crystallite Coalescence during Film Growth. Harley T. Johnson, Allison Suh, Ning Yu, Ki-Myung Lee and Andreas Polycarpou; Mechanical & Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois.

Various models appear in the literature to explain the intrinsic tensile stress due to coalescence in growing polycrystalline thin films. A model described by Freund and Suresh (2003), for example, successfully addresses the coalescence or zipping-up problem using adhesion concepts from the JKR theory of contact mechanics. In this talk, various other contact mechanics formulations for adhesion are compared analytically as candidate theories for predicting the residual stress developed in a simple 2D coalescing grain model. Two JKR-based approaches are compared to cases that incorporate the DMT model, the Maugis-Dugdale model, and the Double-Hertz model. A numerical evaluation of these models shows that JKR and Maugis-Dugdale based approaches yield residual stresses in reasonable agreement with experimental data. Finally, a finite element analysis incorporating special surface contact elements is used to predict residual stress in the 2D coalescing grain model for comparison. The contact elements incorporate either van der Waals or Lennard-Jones type interatomic potentials. Results of the finite element analysis are in good agreement with the analytical models and experimental data and are useful for illustrating full-field stress distributions in the coalescing grains.

9:30 AM U6.5
Stress Relaxation by Excimer Laser Annealing in Amorphous Carbon Thin Films.‡. Thomas A Friedmann and John P Sullivan; Nanostructure Physics, Sandia National Laboratories, Albuquerque, New Mexico.

Thin films of tetrahedral amorphous-carbon (ta-C) are grown at room temperature and typically have high stress (2-10 GPa) as grown. These stresses are intrinsic and are a result of the non-equilibrium growth process that produces metastable carbon bonding configurations that are frozen in at the low growth temperature.

Thermal annealing near 600 °C for short times (minutes) has been shown to completely relieve this stress, enabling ta-C thick film growth (through repeated deposition and annealing steps), membrane based sensors, and micromachines. The stress relief mechanism is through first-order strain-relieving bonding changes that are thermally activated. Unfortunately, there are two practical problems to using thermal annealing for stress relief. First heating and cooling cycles interrupt the growth and can significantly slow the practical growth rate, and second the 600 °C processing temperature and time is higher than the thermal budget allowed for deposition on existing CMOS electronics. We have found that pulsed laser annealing offers a solution to these two problems. A pulsed excimer laser operating at 248 nm was employed to irradiate stressed ta-C samples in air, while simultaneously monitoring the wafer curvature with an optical technique. It was found that stress relaxation was possible and that the relaxation was critically dependent on the laser fluence. Interestingly, one ∼20 ns laser pulse of the proper fluence could produce complete stress relief with no visible damage to the sample and no significant heating of the substrate. Raman spectra of the irradiated films reveal only subtle changes that are consistent with the spectra from films that are furnace annealed, indicating a similar stress relief mechanism. Thus, it should be possible to stress relieve a sample in situ without excess heating and without interrupting the growth. ‡This work was supported by the U.S. DOE under contract DE-AC04-94AL85000 through the LDRD Program, Sandia National Laboratories.

9:45 AM U6.6
¡111> Columns of Copper: Strain-Driven Nucleation and Kinetics-Enforced Stability. Helin Wei[1], Hanchen Huang[2], ChungHo Woo[1] and X.X. Zhang[3]; [1]Department of Mechanical Engineering, Hong Kong Polytechnic University, Kowloon, Hong Kong; [2]Department of Mechanical, Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, New York; [3]Department of Physics, Hong Kong University of Science and Technology, Kowloon, Hong Kong.

Columns nucleate and grow during thin film deposition, when atomic diffusion is lacking and/or geometrical shadowing dominates. These columns generally are along thermodynamically non-preferred orientations; such as <110> in copper. In this work, we report the nucleation and growth of copper <111> columns. The results may appear anomalous or intriguing since these columns should have merged to form <111> uniform films, which are thermodynamically more preferred and commonly observed. Combining experimental observation and simple theoretical analysis, we present the mechanisms of the <111> column nucleation and stabilization. The columns are found to nucleate from <111> uniform films, when the internal strain reaches a critical value. Each grain in the uniform film leads to the formation of multiple columns. Once formed, these columns are stabilized by nothing other than the facet-facet kinetic barrier, as we proposed and calculated recently.

10:30 AM U6.7
Real time in situ stress evolution investigation during electrochemical deposition. Donglei Fan[1], Frank Qing Zhu[2], Ingrid Xiaoyan Shao[1], Peter C Searson[1] and Robert C Cammarata[1]; [1]Materials Science and Engineering, Johns Hopkins Univ, Baltimore, Maryland; [2]Physics and Astronomy, Johns Hopkins University, Baltimore, Maryland.

Thin solid films on a solid substrate are generally deposited in a state of stress. The generation of stress in nonepitaxial films produced by physical vapor deposition (PVD) has been heavily investigated recently, both experimentally and theoretically. We will present real time in situ measurements of stress evolution during electrochemical deposition. These measurements were obtained using a wafer curvature system designed and optimized for use in an electrochemical system. The intrinsic stress generation in Cu and Ni metallic films, and in semi-metallic Bi films, will be discussed, as well as the influence of deposition conditions on the resulting film stress, microstructure, and morphology. The real time stress measurements will also be compared to the predictions of theoretical models that have been used with some success to explain stress generation in PVD films.

10:45 AM U6.8
Strain Engineering on Compliant Substrates. Haizhou Yin[1], Karl D Hobart[2], Rebecca L. Peterson[1], Sean R Shieh[3], Thomas S. Duffy[3] and James C. Sturm[1]; [1]POEM & Dept. of Electrical Engineering, Princeton Univ., Princeton, New Jersey; [2]Naval Research Laboratory, Washington, District of Columbia; [3]Dept. of Geosciences, Princeton Univ, Princeton, New Jersey.

Strain engineering plays a critical role in many applications involving heterogeneous films. Depending on individual cases, compressive, tensile or relaxed films are needed for optimal electrical properties. Here, we demonstrate strain engineering based on compliant substrates In our experiments, 200 nm borophosphorosilicate glass (BPSG) on silicon is used as a compliant substrate to allow for strain manipulation of elastic films on top. Fully-strained SiGe (∼30nm) and unstrained Si (∼25nm) films, initially grown pseudomorphically on a Si(100) substrate and then transferred onto BPSG by wafer bonding and Smart-cut processes [1], have been studied. SiGe and Si films on BPSG are patterned into islands and the strain in islands is adjusted through their lateral expansion and contraction on BPSG softened by high-temperature anneals. As a result of the strong island size dependence of the lateral expansion and contraction rates of SiGe and Si islands on BPSG (linearly proportional to square of the island dimension [2]), strain in the islands on BPSG varies with island size after annealing. Strain from 1.1% to 0.6% in SiGe has been achieved on islands of different sizes on the same wafer. This can be used to integrate strained-SiGe PMOSFETs and strained-Si NMOSFETs. The direction of strain relaxation in strained islands on BPSG can be controlled by island geometry. By adjusting the relative size of different planar dimensions of the island, the island can be fully relaxed in one direction while nearly maintaining full strain in the other direction. Uniaxially-strained Si films have been achieved on SiGe/Si strips on BPSG. The outdiffusion of dopants in BPSG during high-temperature anneals can result in heavy doping in the SiGe/Si on top, which renders the films unsuitable for electronic devices. As a dopant diffusion barrier, a nitride film can be inserted between SiGe/Si and BPSG by depositing a nitride film on SiGe and Si films before the layer transfer. The diffusion suppression by the nitride film and its effect on relaxation of SiGe/Si on BPSG will be reported. This work is supported by DARPA(N66001-00-1-8957). Reference: 1. K.D. Hobart et al., J. Electron. Mater. 29, 897 (2000) 2. H. Yin et al., J. Appl. Phys. 91 (2002)

11:00 AM U6.9
Electrical resistance and morphology of elastic gold film/silicone substrate structures. Stephanie P Lacour[1], Joyelle Jones[1], Sigurd Wagner[1], Teng Li[2] and Z. Suo[2]; [1]Electrical Engineering, Princeton University, Princeton, New Jersey; [2]Mechanical and Aerospace Engineering, Princeton University, Princeton, New Jersey.

Thin stripes of gold deposited onto elastomeric substrates can be stretched reversibly by more than 20 % while remaining electrically conducting. We are developing such stripes to serve as electrical interconnects on stretchable electronic skins. The stripes are gold layers evaporated on top of 5-nm thick chromium film on 1-mm thick polydimethylsiloxane (PDMS) membrane. The chromium acts as an adhesive layer between the PDMS and the gold. The gold layers are 25-nm to 500-nm thick. We observe two different film morphologies: the film is either smooth and continuous, or contains micrometer-long cracks. Stretchability depends on the thickness of the gold layer. Layers thicker than 100-nm fail electrically at tensile strain of 1 % to 2 %. Thinner films remain conducting up to much larger strain. Upon stretching smooth films break into islands on the scale of 1 to 100 micrometers, while initially microcracked films retain their structure on the 1-micrometer scale. The electrical resistance of initially microcracked films rises linearly with strain, starting from values that can lie 10 times above the resistance of films made on glass substrates (the ideal value). Initially smooth films can have electrical resistance close to the ideal value. With elongation their resistance is observed to fluctuate. In both cases the electrical resistance cycles reversibly when the films are cycled mechanically. We present our understanding of the electro-mechanical behavior of these stretchable conductors, discuss the dependence of their electrical resistance on the applied strain and interpret the influence of the gold film morphology on the resistance.

11:15 AM U6.10
Stress-Assisted Copper-Induced Lateral Growth of Polycrystalline Germanium. Bahman Hekmatshoar[1], Davood Shahrjerdi[1], Shamsoddin Mohajerzadeh[1], Ali Afzali Kusha[1] and Ali Khakifirooz[2]; [1]Electrical and Computer Eng. Dept., University of Tehran, Tehran, Iran; [2]Microsystems Technology Laboratories, Massachusetts Institute of Technology, Cambridge, Massachusetts.

Lateral growth of poly-Ge at temperatures as low as 160°C is reported. External mechanical stress has been properly manipulated to drive the low-temperature Cu-induced crystallization of poly-Ge wherever Cu is deposited to form the crystallization seed, as well as providing the chance of the lateral growth. Uniaxial compressive stress has been externally applied to the Ge layer by bending the flexible PET substrate inward. Sample preparation has been performed by e-beam evaporation of a 1000Å a-Ge layer at a base pressure of 10^{-6} torr and at a temperature of 100°C. A 2000Å-thick SiO_2 layer has been then e-beam evaporated to serve as the passivation layer against the diffusion of Cu into the a-Ge layer. After patterning the SiO_2 layer by conventional photolithography, 10Å of Cu is thermally evaporated, which will form the seeding islands after the sacrificial SiO_2 layer is etched away. A growth rate of 2.5 μm/hour in the direction of the applied stress and 1.8 μm/hour perpendicular to it is observed by applying 0.05% equivalent compressive strain, as confirmed by SEM analysis. The SEM micrographs also demonstrate a granular surface morphology in the lateral growth region, with grains in the direction of the uniaxial stress. XRD analysis indicates the partial growth of poly-Ge in the form of a tetragonal structure, only in the presence of compressive stress and this is believed to be the main mechanism, which leads to the otherwise impossible lateral growth of poly-Ge. No lateral growth was observed without applying the compressive stress even at temperatures as high as 400°C (on glass substrate). This approach is an exquisite candidate for ultra-low temperature fabrication of poly-Ge TFTs, having the unique privilege of offering a minimal metal contamination in the lateral growth region, which would be used as the channel for fabricating thin film transistors on flexible substrate.

11:30 AM U6.11
Atomic-scale mechanisms of ion-irradiation induced stress modifications in thin metal films. S. G. Mayr[1] and R.S. Averback[2]; [1]I. Physikalisches Institut, Georg-August-Universitat Gottingen, Gottingen, Germany; [2]Department of Materials Science and Engineering, 1304 W. Green St, Urbana, Idaho.

Ion beam irradiation induced changes in the stresses operating in thin films are correlated with the thermodynamic phases of the films and the evolution in the films microstructure and morphology. We investigate using a combination of experiments and molecular dynamics computer simulations the mechanisms which lead to residual stress changes in amorphous, nanocrystalline, columnar polycrystalline and single crystal thin films, as well as the atomic-scale mechanism of radiation induced viscous flow. While local viscous relaxation within the collision cascade underlies all stress changes, the initial microstructure controls the final state of stress. Financially supported by the US DOE (DEFG02-91-ER45439) and the German DFG - SFB 602, TP B3.

11:45 AM U6.12
The Effect of Deposition Rate on Compressive Stress in Polycrystalline Cu and Ag Thin Films. Andrea Del Vecchio and Frans Spaepen; Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

Although in situ stress measurements have demonstrated that a compressive stress develops in high mobility, polycrystalline metallic thin films after island coalescence, the mechanism that underlies this stress evolution is not clear. Variation of the deposition rate is a tool for testing the various proposed mechanisms. In this study we investigate the effect of a change in deposition rate on the post-coalescence asymptotic stress and the film microstructure in polycrystalline Cu and Ag thin films. The changes in the microstructure with deposition rate are correlated with the measured stresses.

SESSION U7: Indentation Testing
Chairs: Ron Gibala and Jim Knapp
Wednesday Afternoon, December 3, 2003
Room 210 (Hynes)

1:30 PM *U7.1
Indentation Testing of Freestanding Sub-micron Films.
Matthew Begley, Structural and Solid Mechanics, University of Virginia, Charlottesville, Virginia.

It is often challenging to extract thin film mechanical properties from indentation tests on films attached to substrates, particularly for films with thickness less than a micron. Substrate effects and tip calibration errors lead to ambiguity in material properties, particularly for soft materials. Indentation of freestanding films offers considerable advantages for determining the properties of compliant (e.g. polymeric or porous) materials. This talk will describe two important components of testing freestanding films: (*i*) a novel and inexpensive approach to fabricating perfectly circular freestanding films (i.e. windows) with radii in the 20 micron to 2 millimeter range, (*ii*) a general theoretical framework that allows one to identify *apriori* appropriate mechanics solutions (e.g. plate, linear membrane and non-linear membrane). Freestanding spans are created by depositing (via spin-casting, sputtering, etc.) a film on a glass plate with embedded etchable fibers. After film deposition, the fibers (whose composition is propriety) are removed by one-sided etching in a relatively weak concentration of HCl. The etching can take place on any wet-bench, and monitored either optically or using sacrificial coupons that replicate the plate configuration. The freestanding spans can be indented using a conventional nano- or micro-indenter with the desired tip shape. The results of this test approach can be interpreted in terms of a single mechanics framework that relates load-deflection response to the film's elastic modulus, Poisson's ratio and residual stress. This framework will be outlined and used to identify critical values of film thickness, freestanding span and indenter load, which indicate when analytical solutions lead to accurate material properties. The effects of finite-contact sizes (as opposed to point-loads) will also be described in the context of analytical solutions for spherical indenters indenting freestanding films. These two components of testing freestanding films will be illustrated using experiments on poly(methylmethacrylate) (PMMA) and poly(phenylene oxide) PPO with thickness in the 300-750 nm range. Experimental results will be presented which demonstrate that: (*i*) the etching process can be monitored such exposure of the back surface of the film to the HCl solution is minimal, and (*ii*) the micro-fabrication procedure does not affect the films' mechanical response. A comparison of theory and experiment illustrates that the elastic modulus and pre-strain can be extracted from a single test conducted in regimes that include non-linear kinematic effects. Finally, f time permits, a preliminary study of plasticity in metals will be described which uses spherical indenters to induce bi-axial stretching in the film.

2:00 PM U7.2
Combining and comparing acoustic and indentation measurements to determine thin-film mechanical properties.
Donna C. Hurley[1], Douglas T. Smith[2] and Nigel M. Jennett[3]; [1]Materials Reliability Division, NIST, Boulder, Colorado; [2]Ceramics Division, NIST, Gaithersburg, Maryland; [3]Materials Centre, NPL, Teddington, Middlesex, United Kingdom.

Nanoindentation is rapidly becoming the most commercially widespread technique to investigate the mechanical behavior of thin films. Quantitative methods to obtain the indentation (plane strain) modulus of a film have been published. However, absolute validation, for example by acoustic methods, necessitates knowledge of other film parameters such as Poisson ratio or thickness. A procedure is therefore required that will measure all of the necessary thin-film properties (Young's modulus, Poisson ratio, thickness, and density) and validate the nanoindentation results by independent methods. To address this requirement, we compare the results of different measurement techniques obtained on the same thin-film samples. Samples included a variety of films (oxides such as TiO_2 and SiO_2 and metals such as Nb or Ni), substrates (isotropic crown glass and anisotropic silicon), and film thickness (a few hundred nanometers to a few micrometers). We describe experiments with surface acoustic wave spectroscopy (SAWS), a noncontacting method that interrogates a sample area of a few square centimeters. Information from nanoindentation and SAWS measurements were combined so as to exploit the different sensitivity of each method to different parameters. In this way, we obtained a more consistent and complete set of film values than could be achieved by either method separately. For instance, using the nanoindentation modulus as input to the SAWS analysis yielded very precise thickness values. We also discuss how the accuracy of our results is affected by factors like measurement uncertainty, inter- and intra-laboratory reproducibility, and the presence of a thin topcoat. Finally, results from other methods like atomic force acoustic microscopy and microtensile testing are reported to demonstrate how they can contribute supplemental information.

2:15 PM U7.3
Probing strain fields about thin film structures using x-ray microdiffraction. Conal E Murray[1], I C Noyan[1], Patricia M Mooney[1], Barry Lai[2] and Zhonghou Cai[2]; [1]IBM T.J. Watson Research Center, Yorktown Heights, New York; [2]Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois.

The transfer of strain between thin film features and the underlying substrate represents an important factor in the performance and reliability of semiconductor devices, particularly as the distances between these structures decrease. In order to characterize the interaction regions produced in the substrate due to strained thin film structures, we employed synchrotron-based x-ray diffraction techniques to map the enhanced diffracted intensity of the single-crystal Si substrate at a sub-micron resolution. The dynamic-to-kinematic transition observed in the scattering of x-rays from deformed crystals makes this technique extremely sensitive to the amount of substrate deformation as a function of position. Measurements were conducted on 1 μm thick Ni dots evaporated onto Si (111) substrates and 0.1 μm thick, heteroepitaxial SiGe strips of various widths grown on Si (001). The interaction field resolved by the enhanced Si diffracted intensity in the substrate extended up to 100 times the thickness of these features. Although the boundary of the interaction field varied as a function of feature width, a characteristic curve was generated to describe the decay rate of enhanced Si diffracted intensity when the distance from the feature edge is normalized by a mean interaction distance (MID). The rate of decay of the strain fields predicted by traditional treatments of the mechanical interaction between the thin film and substrate, such as those based on[1], did not correspond to the measured decay rates.

Reasons for the discrepancy between the existing elastic models and the observations will be discussed. 1. Blech, I.A. and Meireian, E.S., J. Appl. Phys. **38**, 2913 (1967)

2:30 PM U7.4
The role of grain boundaries during nanoindentation: A Molecular Dynamics simulations. Abdellatif Hasnaoui, Peter M Derlet and Helena Van Swygenhoven; Paul Scherrer Institute, PSI-Villigen, Switzerland.

Large-scale Molecular Dynamics simulations are employed to investigate nanoindentation deformation properties (mechanisms) of model nanocrystalline (nc)-Au containing up to 750 grains. The second moment potential for Au of Cleri and Rosato was used. Loading and unloading curves are simulated for the nanocrystalline structures and for comparison on a single fcc structure. The size effects coming from the ratio between the indenter size and the grain size on the elastic behaviour as well as on the plastic behaviour are discussed. Furthermore the interaction between dislocation activity under the indenter and the grain boundary (GB) network is discussed. In the present work, we have found that when a dislocation is absorbed at a GB, the GB region experiences a local increase in hydrostatic stress. In the case when the GB region is already under a high local stress, the arriving dislocations are repelled to the plastic zone. This high stress localization is situated in regions that have a favorable geometrical orientation, with respect to the projection of the indentation contact area along [110] directions. Furthermore, these local high stress regions are capable of emission of dislocations after which they show a net local release of their stress.

2:45 PM U7.5
Substrate effects on the determination of hardness of thin films by the nanoscratch and nanoindentation techniques. Noureddine Tayebi[1,2,3], Andreas A. Polycarpou[1] and Thomas F. Conry[2]; [1]Mechanical and Industrial Engineering, The University of Illinois at Urbana-Champaign, Urbana, Illinois; [2]General Engineering, The University of Illinois at Urbana-Champaign, Urbana, Illinois; [3]National Center for Super Computing Applications, The University of Illinois at Urbana-Champaign, Urbana, Illinois.

Mechanical testing of thin films on a supporting substrate is most often performed directly on the film layer using the nanoindentation technique. However, the interaction of a thin film with a substrate will typically influence the measurement of its intrinsic mechanical properties. As a rule-of-thumb the indentation depth should not exceed 10-20% of the total film thickness in order to minimize or eliminate substrate effects. Accurate estimates of hardness are almost impossible to attain for thin films with a thickness less than 20 nm since extremely shallow indentations, on the order of a few nanometers, provide inconsistent results due to instrument limitations. In the current study, the nanoscratch technique is applied to thin films to overcome some of the measurement difficulties encountered with nanoindentation. Scratches are generated using a finite-radius diamond tip that traverses along the thin-film surface for a distance of several microns with a prescribed vertical load profile. This technique utilizes measurements of the normal force, the lateral force, and the residual cross-sectional (plowing) area of the resulting scratch, which minimizes the substrate effect. This allows for the examination of much thinner films than is possible with nanoindentation. Furthermore, since data are obtained over several to a hundred of micrometers of lateral range, the nanoscratch technique has more of an averaging effect among grains, grain boundaries, and surface roughness. For this purpose, an analytical model that relates the hardness and interfacial shear stress to the measured normal and lateral forces in a nanoscratch experiment, as well as to the groove cross-sectional area, has been developed. This model also takes into account the effects of the elastic recovery of the plastically deformed surfaces. The model was verified by performing scratch experiments on fused quartz and aluminum samples. The hardness calculated using this model at very shallow scratch depths (~2-4 nm) was in very close agreement with hardness values obtained from the nanoindentation technique at much larger indentation depths. In this paper, the study is extended to the cases of both soft films on hard substrate (gold on fused quartz) and hard films on soft substrate (SiO2 on aluminum) for different film thicknesses (50-300 nm). It is found that the effect of substrate interaction on the measurement of hardness using the nanoscratch technique is delayed to 50-60% of the total film thickness as compared to that of the nanoindentation technique (10-20%). Moreover, experiments with scratch depths as shallow as 3 nm are used to estimate hardness of the thin films.

3:30 PM U7.6
Multivariable nanoindentation study of metal thin films prepared by combinatorial methodologies. Oden L. Warren[1], S.A. Syed Asif[1], Antanas Daugela[1], Dehua Yang[1], Thomas J. Wyrobek[1], C. Eric Ramberg[2] and Qun Fan[2]; [1]Hysitron, Inc., Minneapolis, Minnesota; [2]Symyx Technologies, Inc., Santa Clara, California.

Prior examinations of factors influencing thin film nanoindentation results utilized limited specimen sets involving only a few combinations of thin film material, substrate material, and thin film thickness. Consequently, a comprehensive understanding of factors that affect the determination of nanoindentation modulus and hardness of thin films has yet to emerge. With the recent advent of efficient combinatorial methodologies for sample creation, it now is possible to produce material libraries spanning a substantial portion of the parameter space relevant to thin film systems. In this study, we have undertaken a multivariable, high-throughput approach to ascertaining the influence of a number of nanoindentation and material parameters on the determined nanomechanical properties of a series of metals deposited onto various substrates. Both Hertzian and Oliver-Pharr contact mechanics are used to analyze the nanoindentation data.

3:45 PM U7.7
Mechanical Properties of Fe-Pd Shape Memory Thin Films. Yuki Sugimura and Joost J. Vlassak; DEAS, Harvard University, Cambridge, Massachusetts.

Thermally activated shape memory materials such as Ni-Ti alloys are widely used as functional materials in sensors and actuators. However, heat transfer issues in these materials may limit their application in devices that require rapid response. Magnetically driven shape memory effect in select ferromagnetic alloys offers potential for faster response time, expanding the range of application of these materials. Ferromagnetic shape memory alloys such as the Fe-Pd system have already been studied in detail in bulk form by a number of researchers. While thin films are more suitable for miniature devices research activities in Fe-Pd thin films have begun only recently. The thermoelastic austenite-to-martensite transformation takes place over a composition range of approximately 28 to 32 atomic percent Pd in bulk Fe-Pd alloys. In this study we examine the mechanical properties and shape memory effect of thin Fe-Pd alloys in this composition range deposited by dc magnetron sputtering. Development of film stresses during deposition and subsequent thermal treatment is investigated by the substrate curvature technique. Effect of these stresses on phase transformation temperature and martensite variant reorientation in the presence of an external magnetic field is also studied. Variant maps are developed as a function of film residual stress based on the constrained theory of magnetoelasticity by DeSimone and James (2002).

4:00 PM U7.8
Combinatorial Studies of Mechanical Properties of Ti-Al Thin Films using Nanoindentation. Seung Min J. Han[1], Ranjana Shah[1], Rajarshi Banerjee[2], Babu Viswanathan[2], Bruce M. Clemens[1] and William D. Nix[1]; [1]Materials Science and Engineering, Stanford University, Stanford, California; [2]Materials Science and Engineering, Ohio State University, Columbus, Ohio.

Combinatorial chemistry has developed as an efficient method for rapidly identifying, promising compositions for pharmaceuticals, phosphors and catalysts. This method has been extended recently to bulk structural materials through the creation of libraries of compositions formed in diffusion couples and subsequently mechanically tested by nanoindentation. In the present work, we further extend the combinatorial method by using thin film methods to identify alloy compositions of potential interest as structural materials. To investigate this idea, compositionally graded Ti-Al thin films are sputter deposited onto Si substrates, and the mechanical properties of the various compositions are then probed using nanoindentation. This combinatorial method offers the ability to rapidly vary the compositions of alloys and quickly determine the alloy compositions of greatest interest for further development into bulk structural alloys. The objective of the nanoindentation experiments is to extract the true properties of the film so that the bulk properties of the material may be estimated. But for a thin film on a substrate, nanoindentation senses the properties of both the film and the substrate, and the interactions between the two (pile-up). Thus the standard Oliver & Pharr method requires modification. We have developed a new method for extracting the true hardness of films on substrates and applied it to compositionally graded Ti-Al thin films. Together with the corrected nanoindentation data and a compositional analysis, we are able to establish a relationship between the true hardness of the film and the composition. We discuss the results of these combinatorial experiments and how these results can be used to estimate the mechanical properties of corresponding bulk materials.

4:15 PM U7.9
Dislocation Nucleation and Segregation In Nano-Scale Contact of Stepped Surfaces. HongHui Yu[2], Pranav Shrotriya[1], Junlan Wang[1] and Kyung-Suk Kim[1]; [1]Division of Engineering, Box

D, Brown University, Providence, Rhode Island; [2]Mechanical Engineering, The city college of the city university of New York, New York, New York.

A myriad of engineering applications involve contact between two surfaces, which induces localized plastic deformation near the surface asperities. A unit process model consisting of a surface with one step in contact with a flat rigid surface is considered in the first stage of studying plastic deformation of the surface. Under contact load, a dislocation nucleates and grows out from the surface step. The driving force on the dislocation is calculated using conservation integrals. The effects of surface adhesion, step size, and lattice resistance on the dislocation driving force are analyzed in a continuum dislocation model. The driving force along with a driving force based nucleation criterion determines nucleation and the equilibrium distance traveled by the dislocation away from the surface step. Results of the unit process model show that under a contact load, dislocations in certain slip plane can be easily nucleated but only stay in a thin layer near the surface, while dislocations nucleated along other slip planes easily move away from the surface into the bulk material. The former dislocation is named anti-load dislocation and the latter dislocation is called pro-load dislocation. Embedded atom method (EAM) is also utilized to perform atomistic simulation of the unit-process model. As predicted by the continuum dislocation model, the atomistic simulations also indicate that surface adhesion plays significant role in dislocation nucleation process. Varying the surface adhesion leads to three different regimes of load-deflection instabilities, namely, just dislocation-nucleation instability for no adhesive interaction, two distinct surface-adhesion and dislocation-nucleation instabilities for weak adhesive interaction and a single combined surface-adhesion and dislocation-nucleation instability for strong adhesive interaction. The atomistic simulations provide additional information on dislocation nucleation and growth near the surface steps. The dislocations nucleated from the surface steps interact to form dislocation junctions and their motions away from the steps are strongly influenced by the junction formation.

4:30 PM U7.10
Determination of Residual Stress-Free State and Mapping of Residual Stress Fields Using Speckle Interferometry and Thermal Relaxation. Dong-Won Kim[1], Jong-jin Kim[1], Dongil Son[1], Nak-Kyu Lee[2], Kyoung-Hoan Na[3] and Dongil Kwon[1]; [1]School of Materials Science & Engineering, Seoul National University, Seoul, South Korea; [2]Industrial Technology Center, Korea Institute of Industrial Technology, Incheon City, South Korea; [3]Korea Institute of Industrial Technology, ChonAnsi, South Korea.

We applied in-situ analysis of the relationship between annealing time and thermal displacement to determining the residual stress-free state, using modified electronic speckle pattern interferometry (ESPI) applicable to very small measuring areas ($\sim 500\mu m \times 500\mu m$) with high spatial resolution (10nm). The relaxed stresses in annealing, the thermal strains/stresses and the residual stress fields in the general case were modeled from the relationship between relaxed stresses and thermal strains based on thermoelastic theory. Our experiments mapped the surface residual stress fields on indented bulk Cu, 0.5μm Au film, thermally treated Fe-42%Ni lead frame and welded steel pipe. In indented Cu, the normal and shear residual stress range from -800 MPa to 700 MPa and -600 MPa to 600 MPa respectively around the indented point. In the Au film deposited on Si wafers, the tensile residual stress is uniformly distributed over 650$\pm$100 MPa; this result was consistent with the residual stress measured by x-ray diffractometer (XRD). In the Fe-42%Ni lead frame we prepared specimens thermally pretreated in various conditions at 690°C to 750°C for 1 to 3 min, and also studied specimens without thermal pretreatment. The residual stresses in Fe-42%Ni evaluated by ESPI were verified by theoretical calibration from beam-bending theory. The residual stresses relaxed by various thermal pretreatments were derived from the resultant residual stresses measured by ESPI: a totally relaxed residual stress state was obtained by annealing at 750°C - 2min. In addition, magnification by long-working-distance microscope enabled us to map the residual stress fields in the bending area with a width of 110μm.

4:45 PM U7.11
Influence of Defects on the Nanoindentation Behavior of Gold Single Crystals. Martha M. McCann and Sean G. Corcoran; Materials Science and Engineering, Virginia Tech, Blacksburg, Virginia.

Nanoindentation is an increasingly used tool to investigate the mechanical properties of very small volumes of material. It is usually a very consistent measure of a material's elastic and plastic response to point contact loading. Nanoindentation experiments on gold single crystals display tremendous variation in the residual deformation. The depth of penetration, which is vital in hardness determination, can differ by as much as 100%. This is the first time such a wide spectrum of results has been recorded for a single well prepared surface. The inconsistency of the results is of the order that is usually explained by surface differences like crystal orientation, oxide thickness and contamination layers. None of the previously researched reasons for variation hold true for this surface. The onset of plastic deformation is observed at a stress level on the order of the theoretical yield strength. The difference in depth of penetration is independent of this elastic−plastic transition. Attempts to correlate the depth of penetration to surface feature size have been inconclusive. At this scale, in this system, there are a limited number of defects that are capable of causing this type of discontinuity. It is our goal to try and change the density of some of these defects to investigate their role in single crystal deformation behavior.

SESSION U8: Poster Session
Chairs: Young-Chang Joo and Zhigang Suo
Wednesday Evening, December 3, 2003
8:00 PM
Exhibition Hall D (Hynes)

U8.1
Subcritical Delamination of Dielectric and Metal Films from Low-k Organosilicate Glass (OSG) Thin Films. Youbo Lin and Joost Vlassak; DEAS, Harvard University, Cambridge, Massachusetts.

Organosilicate glass (OSG) thin films are leading candidates among new low-k dielectric materials for use as interlayer dielectric (ILD) in high-performance interconnects. The mechanical properties of OSG are inferior to those of silica. When subject to aggressive chemical environments and loads during the integrated circuit fabrication process, OSG films may be vulnerable to stress-corrosion, leading to subcritical delamination of the film stack. Previous subcritical crack growth experiments in aqueous environments have shown a dependence of the threshold energy release rate and slope of the crack growth rate curves on pH for the OSG/SiO2 system. In this report, we will present subcritical delamination data in aqueous environments for the OSG/SiO2, OSG/TaN, and OSG/SiNx systems. Crack velocity results will be discussed in light of a model of reaction-controlled cracking in these materials systems.

U8.2
Cracking in an Elastic Film on a Power-law Creep Underlayer. Jim Liang[3], Zhen Zhang[1,4], Jean H Prevost[2] and Zhigang Suo[1,4]; [1]Mech & Aero Engr, Princeton University, Princeton, New Jersey; [2]Civil & Environmental Engr, Princeton University, Princeton, New Jersey; [3]Intel Corp., Hillsboro, Oregon; [4]Division of Engr & Appl Sci, Harvard University, Cambridge, Massachusetts.

Devices in modern technologies are structures of complex architectures, diverse materials, and small features. Their reliability depends on inelastic and time-dependent mechanical behavior in such integrated structures. This paper analyzes a three-layer structure consisting of, from top to bottom, an elastic film, a power-law creep underlayer, and a rigid substrate. The layers are well bonded. A channel crack is in the elastic film. Initially, the film is subject to a uniform biaxial tensile stress. As the underlayer creeps, the stress field in the film relaxes in the crack wake but intensifies around the crack tip. We study the time-dependent crack behavior using a two-dimensional shear lag model. When the crack does not grow, the region in which the stress field relaxes increases with time. We identify the length scale of the region from a nonlinear diffusion-like equation. The stress intensity factor is proportional to the square-root of the length scale. When the crack advances, its velocity can reach a steady state. We identify the scaling law for the steady velocity. The extended finite element method (X-FEM) is used to simulate the crack behavior. Numerical results are presented for the stress intensity factors of stationary cracks, and the steady velocities of advancing cracks.

U8.3
Bonding Integrity of Oxidized Polydimethylsiloxane (PDMS) Using Four Point Bending. J. Jay McMahon, Y Kwon, J -Q Lu, T S Cale and R J Gutmann; Focus Center - New York, Rensselaer: Interconnections for Gigascale Integration, Center for Integrated Electronics, Rensselaer Polytechnic Institute, Troy, New York.

PDMS has been used as a material in microfluidic systems because of its low cost, advantageous surface properties, and simple bonding procedure through surface modification using oxygen plasma. This research focuses on evaluation of the bonding integrity of bonded pairs of glass slides, borosilicate glass, and silicon substrates using PDMS thin films. PDMS films were spin cast and cured, surface modified using a low power oxygen plasma, bonded to various materials, then destructively tested using a four point bending technique. Evaluation of permutations of the films and substrates provides the dependence of adhesion energy on both materials and surface preparation. The critical adhesion energy, G_c, using surface modified PDMS to bond glass slides is 3.0 J/m^2, and G_c using unmodified PDMS is 2.8, 1.8, and 1.6 J/m^2 for bonded silicon, glass slides, and borosilicate glass respectively. Correlation of these results to material surface and interface properties will be presented. PDMS will be compared to other dielectric bonding glues for wafer level three dimensional (3D) heterogeneous integration technology platforms.

U8.4
Fracture in Drying Nanoparticle Suspensions.
Eric Robert Dufresne, Daniel J. Stark, Jacqueline Ashmore, John W. Hutchinson and David A. Weitz; Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

Drying nanoparticle suspensions can fracture as they transform from a fluid to a solid. While these cracks can present serious technological problems, they also pose a number of intriguing scientific questions. We study the dynamics of fracture in drying aqueous suspensions of monodisperse silica nanoparticles. In our experiments, the suspension fills a rectangular cross-section glass capillary tube and dries from one edge. As water evaporates, evenly-spaced cracks invade from the drying edge. We show that the average motion of crack-tips is determined by a delicate balance of interfacial and hydrodynamic forces. Additionally, plastic deformation and material heterogeneities lead to intermittent motion.

U8.5
Effects of Process Paramerters on the Adhesion of Copper Film on Polyethylene Terephthalate (Pet) Substrate Prepared By ECR - MOCVD Coupled With Periodic DC Bias.
Joong-Kee Lee[1], Jin Hyun[2], Dongjin Byun[2] and Byung Won Cho[1]; [1]Eco-Nano Research Center, Korea Institute of Science and Technology, Seoul, South Korea; [2]Dept of Materials Science, Korea University, Seoul, South Korea.

The ECR (Electron Cyclotron Resonance) plasma has attracted much attention for its high electron density of 10^{27}-10^{18} m^{-3} at gas pressure range of 1x10^{-4}- 5x10^{-3} torr. The function of ECR is to increase the path of electrons in the plasma by applying a magnetic field normal to the electron trajectory. The charged electron or ions from plasma generate circulation motion normal to the direction of the magnetic field. Resonance is achieved when the frequency at which microwave energy is fed to an electron circulating in a magnetic field is equal to the characteristic frequency at which electron circulates. This circulating motion increases the ratio of ionized to non-ionized species in ECR plasma by three orders of magnitude over that in conventional radio frequency (RF) plasma. Gases fed to the ECR system can be activated with high efficiency by action of resonance. Preparing metallized polymer by metal organic chemical vapor deposition (MOCVD) is of considerable interest since it enables the production of metal film of good adhesion with polymer substrate for microelectronic packaging, organic LCD and electromagnetic interference (EMI) shielding. In the present study, the structural and chemical analyses of the Cu films were carried out and their adhesion force between copper and PET substrate was determined as a function of H_2/Ar mole ratio, microwave power, periodic negative voltage and current of magnets. Adhesion properties of the prepared films were measured in accordance with modified ASTM D5179-91. The coated side of the substrate of 6mm diameter was attached on the surface of a homemade jig with a cyanoacrylate adhesive and tested with Instron®(Model 1127). Measurement was conducted at room temperature and humidity at cross-head speed of 2mm/min. and nano-scratch tester was also employed to investigate the interfacial structure of the copper film prepared by ECR-MOCVD.

U8.6
Adhesion Strength of Polymer Coatings studied by Laser Induced Delamination. Alexander Fedorov[1,2], Tom van Veen[1,2], Redmer van Tijum[2] and Jeff Th.M. de Hosson[2]; [1]Defects in Materials, Interfaculty Reactor Institute, TU Delft, Delft, Netherlands; [2]Dept. Applied Physics, University of Groningen, Groningen, Netherlands.

Laser Induced Delamination is a novel technique aimed at quantification of adhesion of thin polymer films to a metal substrate. In this technique a high power IR pulsed laser beam (maximum 500 mJ in 5 ns) is used to create the primary delaminated area in the form of a blister. The blister is formed due to heating of the metal substrate and because of partial evaporation of the polymer material adjacent to the interface. By varying the power of the laser pulse the gas pressure inside the blister is increased until further delamination takes place. This critical pressure is related to the strength of adhesion of the polymer to the substrate. The shape of the blister (radius and height) is controlled with a stylus profiler and with an optical confocal microscope. In the present work 30 um thick PET and PP films on steel substrate were studied. From the shape of the obtained blisters the strength of adhesion of the polymer is estimated. Various models describing blister formation and growth are discussed.

U8.7
Effect of Aqueous Solution Chemistry on Subcritical Crack Growth in Nanoporous Glass. Eric P. Guyer and Reinhold H. Dauskardt; Materials Science & Engineering, Stanford University, Stanford, California.

Nanoporous organosilicate glasses are being considered for a number of emerging technologies ranging from biosensors to ultra low dielectric constant materials in microelectronic devices. The integration of these materials into such devices has, however, been limited due to their extremely brittle nature and susceptibility to stress corrosion cracking in moist environments. While the effect of moisture and temperature on crack growth has recently received some attention, virtually nothing is known about the effect of more aggressive aqueous solutions, which might be encountered during device fabrication or in service. This research demonstrates for the first time anomalously high subcritical crack growth rates of nanoporous methylsilsesquioxane (MSSQ) thin-films in weakly acidic hydrogen peroxide solutions. Results vary markedly from those predicted by solution pH, as acidic environments are generally considered to inhibit cracking. In this presentation we elucidate the fundamental chemical interactions and molecular mechanisms responsible for the anomalous crack growth behavior. Implications for the integration of nanoporous thin-films in emerging device technologies are considered.

U8.8
An Atomistic View of Dislocation Plasticity in Thin Metal Films. E. S. Ege and Y.-L. Shen; Mechanical Engineering, University of New Mexico, Albuquerque, New Mexico.

Polycrystalline metallic films attached to substrates display constrained plasticity because of the dimensional confinement. Typically a passivated film can carry a higher stress than its unpassivated counterpart, which in turn can carry a higher stress than the free-standing film of the same thickness and grain size. Theories centered around the concept of misfit dislocations at the interface have been developed to explain this effect, although general experimental evidences are lacking due in part to the fact that the metal films are adjacent to amorphous layers in most cases. In this work experimental results on the strength of aluminum and copper films are first presented. Atomistic simulations using molecular statics are carried out to explore the nature of dislocation plasticity in these films. The existence of an interface between the film and the substrate is shown to delay plastic yielding and lead to film strengthening. Some dislocations need to travel a longer distance (by being reflected from the interface), or it will be until new mobile dislocations are generated, to achieve gross plastic deformation of the film. The capability of atoms to slide along the interface plays a crucial role in determining the macroscopic stress-strain response and the microscopic dislocation activities. Representative cases of voiding induced damage in the film will also be presented.

U8.9
Dislocation Dynamics in Semiconductor Thin Films. Lizhi Sun and E.H. Tan; University of Iowa, Iowa City, Iowa.

Microstructural dislocation defects play a central role in the fabrication, performance and reliability of microelectronic devices. A discrete dislocation dynamics model is developed to establish the equations of motion for three-dimensional interacting dislocation loops in the semiconductor thin film - substrate system. The film is assumed to be an elastic thin layer and is perfectly bonded with another elastic substrate. The stress fields of threading and misfit dislocation loops are first calculated as an essential ingredient in the dislocation dynamics method. Dislocation loops are discretized into segments, each of which is represented by a parametric space curve of specific shape functions and associated degree of freedom. The simulation of dislocation activities is applied to several issues related to threading dislocation growth, interaction of dislocation loops with surface and interface. Comparisons between the present prediction and experimental observation are also presented.

U8.10
Microstructural aspect of fracture in nanolayered TiAlCrN thin films. Ayat Karimi[1], Antonio Santana[1], Andreas Schuetze[2] and Volker Derflinger[2]; [1]Faculty of Basic Science, EPFL, Lausanne, Switzerland; [2]Thin Films Technology, Balzers Ltd., Balzers, Liechtenstein.

This paper deals with the effects of bilayer thickness and chromium content on the microstructure and mechanical properties of nanolayered TiAlN/TiAlCrN thin films deposited onto the WC-Co substrates using cathodic arc PVD. Conventional and HR-TEM showed that aluminum contributes to refinement of structure in TiAlN, while chromium favors the formation of columnar structures in TiAlCrN layers. In thick bilayer films, TiAlN periodically interrupts the formation of columns in TiAlCrN, while for thinner bilayer films (e<10 nm) the columns are not interrupted leading thus to the formation of perfectly columnar films. Both Cr content and bilayer thickness contribute to hardness enhancement. Effect of Cr arises from the formation of hard fcc-(Cr,Al)N phase and vanish of softer wurtzite-like hcp-AlN. The hardness enhancement due to multilayering is explained by two factors; a grain refinement based on Hall-Petch effect and the formation of columnar structures with (111) preferred orientation. Such structural modifications strongly influence crack modes and morphologies observed using AFM and FIB cross-section of indents. Thin bilayer films exhibit well-organized straight cracks parallel to contact edge between indenter and film, while large bilayer films show a network of discontinuous irregular mud cracks attributed to grain boundary sliding. In contrast, columnar films yields the formation of radial cracks emanating from the corner of indenter. FIB micrographs confirmed that nanolayering results in crack meandering and branching, which prevents the propagation of large cracks with more dramatic effects in accordance with fracture toughness of different crack modes. In this paper the relationships between microstructure, mechanical properties and fracture modes and mechanisms of nanolayered hard thin films are disdcussed.

U8.11
Crack Propagation Instability in Four-Point Bend Test Specimen Under Displacement Controlled Loading.
Zhenyu Huang[1], Zhigang Suo[1], Jean Prevost[2], N Sukumar[3], Jessica Xu[4] and Jun He[4]; [1]Dpt.MAE, Princeton University, Princeton, New Jersey; [2]Civil and Environmental Engineering Department, Princeton University, Princeton, New Jersey; [3]Department of Civil and Environmental Engineering, University of California, Davis, Davis, California; [4]Intel Corporation, Santa Clara, California.

Numerical results are presented for the four-point bend structure test specimen, which is commonly used in industry to measure the interfacial toughness. We studied the unstable propagation distance issues relating to the initial interfacial flaw size and testing machine compliance. According to the results, for the sandwiched structure with a compliant layer, when the crack kinks onto the upper interface, it propagates stably; however, when the crack deflects onto the lower interface, it propagates unstably, the shorter the initial crack length, the longer the unstable propagation distance. The unstable propagation distance is independent of the interfacial toughness once it is initiated to grow. The unstable propagation distance can be reduced by introduction a relatively large flaw size or by reduction of the machine compliance. For the crack at the lower interface, the initial crack length has more significant influence on unstable propagation distance; for homogeneous cases, machine compliance has larger influence on the unstable propagation distance than the initial flaw size does. When an initial crack length is a few sandwiched layer thickness, the unstable propagation distance is the same as the homogeneous case.

U8.12
Strain relaxation during in-situ growth of SrTiO3 thin films.
Luke Peng[1], Xiaoxing Xi[2] and Brian Moeckly[1]; [1]Superconductor Technologies, Sunnyvale, California; [2]Materials Science and Engineering, Pennsylvania State University, University Park, Pennsylvania.

We have used in-situ reflection high-energy electron diffraction (RHEED) to study the real time strain relaxation of SrTiO3 films as they are grown on substrates of different lattice and thermal expansion mismatches. The initial misfit strain in the SrTiO3 film is tensile when grown on MgO and compressive on LaAlO3, as expected from the lattice mismatches between the film and the substrates. Strain relaxation begins immediately after the deposition starts, but it is not complete until the film thickness reaches 500 - 2500A depending on the substrate and the deposition temperature. However, x-ray measurements show that the room-temperature film strain is compressive for thin (<800A) SrTiO3 films on both substrates. We will discuss an interpretation of these results in terms of the formation of misfit dislocations. This work was partially supported by DARPA as part of the FAME program, Contract No. N00014-98-C-0287.

U8.13
Mechanical Behavior of Pt-Ir Alloy Thin Films as a Function of Oxidation Conditions. Richard Chromik, Thirumalesh Bannuru, Walter Brown and Richard P. Vinci; Materials Science and Engineering, Lehigh University, Bethlehem, Pennsylvania.

Novel electrode materials for MEMS applications require both robust electrical and mechanical properties. For elevated temperature applications, stress relaxation and morphological stability often present problems. In bulk materials, these are usually addressed by alloying, but the unique conditions that pertain to sub-micron metal thin films may make alloying less effective. We study Pt-Ir films (oxidized and unoxidized) that should provide both solid solution and oxide dispersion strengthening while retaining reasonable electrical properties. Alloy films were fabricated by co-deposition in the composition range of 0 to 25at% Ir, as determined by x-ray photoelectron spectroscopy (XPS). Both nanoindentation and wafer curvature measurements were used to determine the stress state and mechanical properties of the films. For as-prepared films, solid solution strengthening was observed, where films with higher Ir content exhibited higher hardness. Oxidation studies were carried out to form Ir oxide particles and examine the dispersion strengthening mechanisms as well.

U8.14
Nanohardness and Crack Resistance of HTS YBCO Thin Films. Yakov M Soifer[1], A Verdyan[1], J. Azoulay[1], E. Rabkin[2] and M. Kazakevich[2]; [1]Department of Sciences, Holon Academic Institute of Technology, Holon, Israel; [2]Department of Materials Engineering, Technion, Israel Institute of Technology, Haifa, Israel.

In this study the nanoindenting atomic force microscope (AFM) was employed for studying of local mechanical properties of commercially available high TC superconducting YBCO thin films of high critical current density (TC of 90K, JC (H=0) of 3∙107 A/cm2 at 4.2K and 2∙106 A/cm2 at 77K). YBCO films on two different substrates with very different hardness and Young modules (sapphire and SrTiO3) were studied. The relationship between mechanical properties and microstructure of these films was investigated. The morphology and microstructure of the films were determined by scanning electron microscopy (SEM), AFM and X-ray diffraction. Hardness, Young modulus and scratch parameters were measured for different indentation loads from 1 to 9 mN. The standard analysis of load-displacement curves was complemented by the quantitative analyses of AFM images of the indentation imprints (corresponding indent parameters can be measured with the nanometric accuracy). The determined intrinsic hardness and Young modulus of YBCO thin films are H $\sim$ 8.5 GPa, and E $\sim$210 GPa, respectively. The critical parameters of crack nucleation are determined and the results obtained are discussed in the terms of the microscopic deformation mechanisms.

U8.15
Cracking and Phase Transformation in Silicon and Germanium During Nanoindentation. Jae-il Jang[1,2], Songqing Wen[1], I. M. Anderson[1,2] and G. M. Pharr[1,2]; [1]Department of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee; [2]Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Nanoindentation has been used to examine two phenomena important in semiconductor substrate materials: pressure-induced phase transformations and small-scale cracking. Nanoindentation experiments were performed Si and Ge using a series of triangular pyramidal indenters with centerline-to-face angles in the range 35 degree to 85 degree. The influences of indenter geometry, maximum load, and loading/unloading rate were systematically examined to establish cracking thresholds and provide evidence for phase transformations. Field emission scanning electron microscopy was used to image indentations as small as 50 nm in depth in order to characterize the contact damage. Results are presented and discussed in terms of prevailing descriptions and models for cracking and indentation-induced phase transformation.

U8.16
Scratch test measurements of the adhesion of CrN coatings.
P. van Essen[1,2], R. Hoy[2], J.-D. Kamminga[2], S.Yu. Grachev[2], G.C.A.M. Janssen[1] and A.P. Ehiasarian[3]; [1]Materials Science, Delft University of Technology, Delft, Netherlands; [2]NIMR, Delft, Netherlands; [3]Materials Research Institute, Sheffield Hallam University, Sheffield, United Kingdom.

Hard CrN coatings of 3.6 micron thickness were deposited by means of reactive sputter deposition in an industrial PVD reactor. The dependence of the scratch test performance on the composition of the coatings was studied. The composition was varied from understoichiometric CrN to CrN deposited in a surplus of nitrogen. The nitrogen gasflow was varied from 50 to 80 sccm during deposition. The hardness of all coatings, irrespective of composition, was about 30 GPa, the reduced modulus of all coatings was about 230 GPa. All coatings were under compressive stress in the range of 0.7 to 1.9 GPa. The coatings were deposited both on high speed steel and hot work tool steel. The adhesion was evaluated by means of scratch testing with a diamond stylus. The load at which chipping occured was taken as a measure for adhesion. Chipping started in the load range 8-65 N depending on coating composition and substrate For all compositions chipping occured at lower loads on high speed steel than on hot work tool steel. For the same load the track width after scratch testing was wider on the hot work tool steel than on the high speed steel. Consequently the hot work tool steel is softer and deforms more. The difference in the load needed for chipping is attributed not to the adhesion but to a larger deformation of the softer hot work tool steel leading to a larger deformation of the coating. For coatings on the same substrate the scratch test performance depends strongly on composition. Stoichiomeric CrN coatings outperform understoichiometric CrN coatings in scratch tests both on high speed steel and on hot work tool steel.

U8.17
A New Method to Evaluate the Fracture Toughness of Thin Films. Xia Zhenhai, Abhinav Bhandari, Hao Li, William A. Curtin and Brian W. Sheldon; Division of Engineering, Brown University, Providence, Rhode Island.

A new method based on microindentation is developed to determine the fracture toughness of a thin film bonded to a brittle substrate. The sample consists of a partially-coated substrate, and indentation on the uncoated portion of the substrate is used to generate a radial crack that propagates into the coated region. This test method avoids the complicating effects of delamination that often occur when the coating is indented directly. Comparison of the lengths of the indentation cracks into and away from the coated region can indicate toughening imparted by the coating. To extract quantitative results, a three dimensional finite element model of the system geometry is generated and a cohesive zone model is used to predict the complex equilibrium crack front. The model predicts the length of the crack penetration into the coating versus the length of the crack growth away from the coating as a function of the elastic and toughness properties of the coating and the substrate. Residual stresses make an important contribution to the detailed crack growth and are included in the numerical calculations. An approximate analysis using energy balances is developed to calculate the coating toughness from experimental data and this agrees well with the numerical results over a wide range of coating and substrate property values. The experimental method and numerical analysis are applied to assess the toughness of a variety of CVD diamond coatings. Relationships between the measured fracture toughness and the grain size, residual stress, and stress gradients will be presented.

U8.18
The effect of interface morphology on the delamination of thin film. Honghui Yu, Mechanical Engineering, The City College of New York, New York, New York.

When a thin film is under residual stress, surface roughness could introduce normal tensile stress on the interface and thus initiates film delamination. On the other hand, people roughen the road surface before re-paving a highway, and sand a glossy furniture surface before re-painting. By changing the mode mixity of an interface crack and increasing the interface area, the interface roughness may increase the effective interface toughness, the resistance to crack propagation along the interface. In this paper, the driving force for crack propagation, the energy release rate, is calculated along the curved interface. The corresponding interface toughness will be obtained from the calculation of mode mixity. The competition of the energy release rate and interface toughness determines the stability of the crack. The effects of residual stress, the work of adhesion of the interface, and the interface morphology on the film delamination will be discussed.

U8.19
Comparison of propagation velocity for repeated cracking of semiconductor glass films. Sheng Liu[1], Hee C Lim[1], John F Federici[1], Helena Gleskova[2], Sigurd Wagner[2], Zhigang Suo[3] and Gordon A Thomas[1]; [1]Physics, NJIT, Newark, New Jersey; [2]Electrical Engineering, Princeton, Princeton, New Jersey; [3]Mechanical and Aerospace Engineering, Princeton, Princeton, New Jersey.

We have determined the propagation velocity of successive cracks in a single glass sample as a function of energy release rate. We have obtained the conditions for controlled, repetitive crack formation by using a substrate of compliant plastic that survives the cracking of a thin film thermally bonded to it. We have studied cracks in amorphous SiN_x, chosen because it does not delaminate under any of our experimental conditions and because it has electronic applications. We have also studied a bilayer of a-SiN_x and a-Si, chosen because it is widely used to make flexible electronic circuits. The cracking studies delineate the region of applied stress under which the flexible circuits can operate without damage due to mechanical challenges including tensile strain and bending. We have recorded the crack velocity curves using high-speed micro-photography with dark field illumination. Under uniform, uniaxial tensile strain, the films crack in an array of essentially straight, parallel lines, if the increase of the strain density is slow. The crack morphology shows a transition to disorder as the strain growth rate increases. As shown previously, after a crack forms, its velocity increases rapidly with strain, slows down and then increases rapidly before completion: the acceleration is maximun at the start and end. Our data provide the unusual information that the successive cracks show velocity curves that scale with each other.

U8.20
Transformation Properties and Microstructure of Ti-rich NiTi Shape Memory Thin Films. Xi Wang[1], Ann Lai[1], Joost J. Vlassak[1] and Yves Bellouard[2]; [1]Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts; [2]Center for Automation Technologies, Rensselaer Polytechnic Institute, Troy, New York.

Thin films of NiTi shape memory alloys find application as actuators in numerous MEMS devices. When deposited at room temperature, NiTi thin films are amorphous and need to be crystallized before they can be used as a functional material. We will present the results of an annealing study on NiTi shape memory thin films in which films have been crystallized using both conventional vacuum annealing and laser annealing. This last technique has the advantage that the shape memory properties of the film can be spatially distributed as required by the application. Amorphous films of a Ti-rich NiTi shape memory alloy were deposited by means of UHV sputtering. The films were grown on (100) Si wafers both with and without an LPCVD SiNx barrier film and the thickness of the films varied between 0.5 and 1.5 mm. The as-deposited films were annealed in vacuum at temperatures ranging from 500oC to 800oC. The stress evolution of the film during the crystallization process was measured using the wafer curvature technique. Composition measurements indicate that there is a small shift in the Ti content of the films on the Si substrate before and after annealing. After the anneal, the shape memory behavior was investigated using differential scanning calorimetry and wafer curvature measurements. The microstructure of annealed films was characterized using electron backscattered diffraction (EBSD) and transmission electron microscopy (TEM). The microstructure obtained after annealing in vacuum is compared to that obtained by selectively annealing freestanding NiTi films using a laser beam and the effect of laser annealing parameters such as laser power, pulse duration, and spot size on the microstructure of the films is investigated.

U8.21
Reliability Improvement Challenges of Advanced Low-k Dielectric for 65-nm Technology Node. Ichiro Kato[1], Sumio Sekiyama[1], Shinichi Takeshiro[1], Yoko Uchida[1], Takuya Fukuda[1] and Hiroshi Yanazawa[1]; [1]ASET, Atsugi, Japan; [2]Association of Super-Advanced Electronics Technologies (ASET), Atsugi, Japan.

To establish a PFC (Perfluorocarbon)-free-process, ASET has been developing a new inorganic low-k dielectric polymer film with a dielectric constant of about 2.4. An interlayer dielectric consisting of a combination of this film and an appropriate organic film should provide an effective dielectric constant below 2.3-2.7, which is required for the 65-nm technology node. Furthermore, the combination of these films is an important step toward a PFC-free-process. Mechanical stress during chemical mechanical polishing process (CMP) often degrades the electrical characteristics of Cu damascene interconnects made with the new low-k dielectric. Finding ways to improve the characteristics is also a subject for study regarding our organic/inorganic film combination. A cleaning treatment of comb-shaped Cu interconnects with CO2 supercritical fluid (SCF) appears to be an effective way to improve the breakdown voltage and TDDB lifetime of organic dielectrics. However, treating bulk organic and inorganic dielectrics with CO2-SCF was found to degrade their CV and IV characteristics. So, the effect of this treatment seems to be different before electrode evaporation and after CMP. The improvement observed after CMP may be due to the removal of moisture. In this study, the relation between an effect of CO2-SCF treatment and a mechanical adhesion property was examined separately for the organic and the inorganic films. Acknowledgements: This work was performed under the management of ASET in an MITI R&D program supported by NEDO.

U8.22
The Mechanisms Controlling the Hardness of Si, Ge and Diamond. Luc J. Vandeperre, Finn Giuliani, Stephen J. Lloyd and William J. Clegg; Department of Materials Science and Metallurgy, University of Cambridge, Cambridge, United Kingdom.

It is well known that phase transformations occur during low temperature indentation of materials with the diamond structure such as Si, Ge and diamond itself. Moreover, the hardness of these materials is similar to the pressure where the diamond structure becomes unstable in the respective materials, suggesting that the resistance to indentation is controlled by the phase transformation. It is shown that a phase transformation alone cannot give rise to the hardness values observed, and that plastic flow must occur in the transformed material. Moreover, the hardness can only be similar to the pressure, which induces the phase transformation, provided the yield strength of the transformed material is small compared to the transformation pressure, and this is consistent with both observations in the TEM and estimates of the Peierls stress in the transformed β-Sn structure. Using these estimates for the yield behaviour of the transformed material together with experimental values for the material in the diamond structure, predictions for the variation of the hardness with temperature give good agreement with experiments.

U8.23

Computational and Experimental Characterization of Indentation Creep. Ming Dao[1], Hidenari Takagi[2], Masami Fujiwara[3] and Masahisa Otsuka[4]; [1]Dept of Materials Science & Engineering, MIT, Cambridge, Massachusetts; [2]Dept of Mechanical Engineering, Nihon University, Tamuramachi, Koriyama, Fukushima, Japan; [3]Dept of Applied Physics, Nihon University, Tamuramachi, Koriyama, Fukushima, Japan; [4]Dept of Materials Science & Engineering, Shibaura Institute of Technology, Tokyo, Japan.

Detailed finite-element computations and carefully designed indentation creep experiments were carried out in order to establish a robust and systematic method to accurately extract creep properties during indentation creep tests. Samples made from an Al-5.3mol%Mg solid solution alloy were tested at temperatures ranging from 573 K to 773 K. Finite-element simulations confirmed that, for a power law creep material, the indentation creep strain field is indeed self-similar in a constant-load indentation creep test, except during short transient periods at the initial loading stage and when there is a deformation mechanism change. Self-similar indentation creep leads to a constitutive equation from which the power-law creep exponent, n, the activation energy for creep, Q_c, the back/internal stress and so on can be evaluated robustly. The creep stress exponent, n, was found to change distinctively from 4.8 to 3.2 below a critical stress level, while this critical stress decreases rapidly with increasing temperature. The activation energy for creep in the stress range of n=3.2 was evaluated to be 123 kJ/mol, close to the activation energy for mutual diffusion of this alloy, 130 kJ/mol. These results are in good agreement with those obtained from conventional uniaxial creep tests in the dislocation creep regime.

U8.24

On the Expanded Range of Silicon Film Susceptibility to Reaction-Layer Fatigue. Olivier N. Pierron and Christopher L Muhlstein; Department of Materials Science and Engineering and The Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania.

In contrast to their bulk forms, micron-scale single-crystal and polycrystalline silicon structural films are known to undergo fatigue failure in room temperature air. Published experiments and numerical models by the authors have attributed the failures to a "reaction-layer fatigue" mechanism whereby cracks can initiate and grow in materials that are immune to environmentally-assisted and fatigue crack growth in their bulk form. Reaction-layer fatigue of silicon films involves a sequential, coupled process of mechanically-induced oxidation of the silicon and environmentally-assisted cracking of the oxide layer. While previous developments have focused on the stability of cracks *within* the surface oxide layer, this work utilizes finite element models and laboratory experiments to establish that a significantly larger range of geometries are susceptible to reaction-layer fatigue. Finite element models of a crack growing within the surface layer of a bimaterial system of silicon dioxide on top of a silicon substrate were developed and were used to construct a "failure map" that establishes the vulnerability of the system to failure due to cracks in the surface layer. The results of these calculations suggest that the reaction-layer fatigue mechanism is a viable failure mode for silicon thin films when surface oxide layers thicker than ~10-20 nm, a two-to three fold reduction from the previous estimates of the required layer thicknesses for the mechanism to be active. Such oxide thicknesses are commensurate with the oxide thickness than can form on silicon thin films within MEMS structures. A crucial aspect of this theoretical development is the consideration of the stability of a crack within the surface layer when crack tip is located on the bimaterial interface. The ability of a previously-derived interfacial stress intensity factor to describe the critical conditions for failure were investigated using macro-scale, composite beams of a glass layer bonded to single-crystal silicon, loaded in four-point bending. These experiments are a unique attempt to establish the validity of a modified stress intensity factor failure criterion for a crack perpendicular to the interface of a bimaterial. The results of these numerical and experimental results provide important insight into the evolution of fatigue damage in silicon films and constitute a useful framework for the design of microelectromechanical systems.

U8.25

Tuning the mechanical properties of SiO2 thin film for MEMS applications. Wang-Shen Su[1], Weileun Fang[1] and Ming-Shih Tsai[2]; [1]Institute of MicroElectroMechanical System, National Tsing Hua University, Taiwan, Hsinchu, Taiwan; [2]National Nano Device Laboratories, Taiwan, Hsinchu, Taiwan.

Thin film mechanical properties are very critical for the performance of MEMS devices. In this study, a convenient approach to tune the thin film mechanical properties using plasma was realized. To demonstrate the feasibility of this approach, various plasma treatments, including O2, H2, NH3 atmospheres, were implemented to tune the Young's modulus and residual stress of SiO2 film. The mechanical properties were determined using the micromachined SiO2 cantilevers. The residual stress of the SiO2 film was determined by the static tip deflection of the SiO2 cantilever. Without plasma treatment, the tip deformation of a 200μm long cantilever beam was 9.01μm. After treatment with H2, O2, and NH3 plasma, the tip deformation of a 200μm long cantilever became 10.22, 8.28, and -6.84μm respectively. It is evident that the residual stress was significantly changed by the NH3 plasma. The Young's modulus of the SiO2 film was determined by the natural frequency of the SiO2 cantilever. The Young's modulus of the SiO2 film without plasma treatment was 78.5GPa. However, the Young's modulus of the film after treated with H2, O2, NH3 plasma became 70.8, 74.7, and 71.4 GPa, respectively. Hence, the H2 and NH3 plasma can reduce the elastic modulus of SiO2 film for about 10%. In addition, the surface bonding and depth profile were also characterized to investigate the mechanism leaded to the change of mechanical properties. Regarding to electron spectroscopy for chemical analysis (ESCA) analyses, there was significant Si-NH2/Si-N bonds formed after NH3 plasma treatment, but no obvious change of Si-O bonding was observed after O2 and H2 treatments. The secondary ion mass spectrometer (SIMS) depth profile of N atom analysis with various plasma treatment, the results showed that the high concentration between 5-20 nm observed after NH3 treatment, but no obvious change of N atom in surface was observed after O2, H2 and without plasma treatments. In conclusion, plasma pre-treatment on the surface provided a convenient way to tune the mechanical characteristics of MEMS structures.

U8.26

Optimization of Film Stresses Utilized in Composite Piezoelectric Membrane Microgenerators. Marian Kennedy[1], M Zosel[1], C D Richards[1], R F Richards[1], D F Bahr[1], K W Hipps[2] and N R Moody[3]; [1]School of Mechanical and Materials Engineering, Washington State University, Pullman, Washington; [2]Department of Chemistry, Washington State University, Pullman, Washington; [3]Sandia National Laboratories, Livermore, California.

Micro-actuators and -generators use the piezoelectric effect of lead-zirconate- titanate (PZT) films to provide relatively high power density in flexing systems. To optimize these systems to achieve the highest electrical output, the relationships between processing, microstructure and mechanical properties of layers need to be examined. The focus of this study is to control the PZT composition and processing and the resulting contributions to the modulus, microstructure and residual stress. PZT films were deposited using solution deposition onto platinized silicon and silicon oxide membranes. The thin films were characterized using continuous stiffness nanoindentation, AFM, Raman Spectroscopy, and ferroelectric testing. The membrane residual stresses were extracted with pneumatic bulge testing and PZT films were analyzed with x-ray diffraction and Raman spectroscopy. By varying sol-gel PZT processing to produce both rhombohedral 52:48 and tetragonal 40:60 PZT with acetic acid and 2-MOE solvents, tensile stresses were varied between 400 and 200 MPa. Variations in Raman spectra were tracked for both processing and crystal structure to correlate peak shifts in the spectra with the tensile stress in the PZT. Raman spectroscopy was utilized to show the effect of PZT stress on membranes that underwent conventional and rapid thermal annealing. A tungsten compressive layer minimized the effective residual stress in the composite membrane and increased the mechanical compliance by 10%. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

U8.27

Stress and Strength of free standing 2-dimensional tetrahedral amorphous carbon bridge arrays. Daniel H.C. Chua[1], William I. Milne[1], B K Tay[2] and D. Sheeja[2]; [1]Engineering Dept, Cambridge University, Cambridge, United Kingdom; [2]School of EEE, Nanyang Technological University, Singapore, Singapore.

The stress and strength of ultrathin (25nm) 2-dimensional free-standing arrays of tetrahedral amorphous Carbon (ta-C) microbridges is reported. The ta-C films were deposited by a Filtered Cathodic Vacuum Arc (FCVA) deposition system where the sp3 content in the film was measured to be in excess of 90% by high resolution XPS. Continuous arrays of free standing ta-C bridges whose aspect ratios ranged from 1:1 to 12:1 were successfully

fabricated while maintaining the same thickness. Due to the naturally high compressive stress of ta-C films, the buckling of films was perpendicular to the length of the beam. We attempt to approximate the curvature of the bend with the intrinsic stress of the films. The displacement of curvature obtained was further compared with FEM simulation results. Moreover, the curvature or arch of these ultrathin films, coupled with a high Youngs modulus (750GPa) and Hardness (60GPa), meant they could withstand a vertical force in excess of 8000mN without breaking.

U8.28
A Microtensile Set Up for Characterising the Mechanical Properties of Thin Films. Brigita Cyziute[2], Liudvikas Augulis[2], Joel Bonneville[1], Philippe Goudeau[1], Sigitas Tamulevicius[3,2] and Claude Templier[1]; [1]Physics, University of Poitiers, Chasseneuil Futuroscope, France; [2]Physics, University of Technology, Kaunas, Lithuania; [3]Physical Electronics, University of Technology, Kaunas, Lithuania.

The design and performance of an original computer control deformation set-up, which has been specifically developed for measuring the elastic and plastic properties of thin films, will be presented. It combines a piezo-actuated microtensile testing set-up with a speckle interferometer stage for the measurement of specimen deformation. The microtensile deformation set up is synchronised with the speckle interferometer to measure specimen elongation up to 70 mm on specimen having a gauge length of 4mm with an accuracy of nearly 50 nm. A main advantage of using speckle interferometry technique is that no patterning or marking of the specimen surface is needed. Displacements can be measured at various spots of the film surface, which in practice allows for the evaluation of the Young's modulus and Poisson's ratio. Tensile forces are directly measured with a miniature load cell. Deformation experiments are performed at constant strain-rate with a precise monitoring of the voltage ramp applied to the piezo-actuator. The paper will be mainly focused on the experimental set up and the method of speckle interferometry technique. Preliminary tensile tests have been carried out on 10mm thick aluminium sheets of commercial purity and on polymer foils with thickness of 50 and 75 mm. The Young's moduli, which are deduced from the stress-strain curves, are in fair agreement with reported bulk average values. The results confirmed the ability of the equipment for the measurements of very small load and displacement levels, which are a prerequisite for such type of investigations. Work is in progress to use this microtensile set up for studying the mechanical properties of free standing thin films of pure copper, as well as aluminium coated polymer foils. A particular attention will be paid to actually characterise the transition between micro- and macro-plastic domain by performing transient tests in order to evaluate the corresponding activation parameters.

U8.29
Mechanical Characterization of Thin Films Based on Membranes Pressure-Deflection Method. Mircea Capanu, Andrew Cervin-Lawry and Atin Patel; Technology R&D, Gennum Corporation, Burlington, Ontario, Canada.

Thin film properties like residual stresses, biaxial modulus, Young modulus, Poisson ratio are important to know for the modeling, design, manufacturing and testing of micromachined devices, especially for MEMS. The pressure-deflection measurements of thin film membranes reveal the residual stresses and biaxial modulus. In order to calculate the Young modulus and Poisson ratio, correlation between different membranes is needed: square and rectangular, circular and rectangular, etc. In the present work we have investigated the validity of the formula used by this method by considering a membranes made of single crystal Si. LPCVD SixNy and PolySi thin film membranes have been considered. Squares and rectangular membranes were made using KOH and circular, squares and rectangular membranes were made using DRIE.

U8.30
Comparison of Micro-bending Experiments and the Indentation Size Effect. J Gregory Swadener and Amit Misra; MST-8, Los Alamos National Laboatory, Los Alamos, New Mexico.

When tested in small volumes, crystalline materials have often shown dramatic increases in strength compared to their macroscopic strength. Generally, the test geometry imposes non-uniform deformation and leads to length scale effects, such as the indentation size effect. Length scales on the order of 1 micrometer have been observed in micro-indentation, micro-torsion and micro-bend experiments. The length scales determined previously have been composed of both material and geometric parameters, but tend to indicate the critical dimension at which the increase of strength becomes significant. Our earlier studies have shown that the relevant length scale can be $>$ 100 micrometers in crystalline materials with initially low dislocation densities (10e9 cm-2). In both metals and ionic salt crystals, we have found that the predicted effects of geometrically necessary dislocations and statistically stored dislocations must be treated separately in order to reach agreement with experimental results. One difficulty in the analysis of indentation results is that numerous interacting slip systems are involved. We are presently conducting micro-bend experiments in free standing copper films. The micro-bend geometry produces a more simplified dislocation structure, which allows a straight forward analysis of the relation between dislocation density and strength. The analysis will then be applied to previous results that show the indentation size effect using spherical indenters. Research sponsored by the Office of Basic Energy Sciences, U. S. Department of Energy.

U8.31
New Methods to Examine Formation and Properties of Interfacial Films in Sliding Contacts. Gun Y. Lee, Irwin L. Singer and Kathryn J Wahl; U.S. Naval Research Laboratory, Washington, District of Columbia.

Mechanical properties of surfaces and interfaces are important for understanding the behavior of tribological contacts, where changes in interfacial properties can result from surface treatments, contaminant films, or sliding processes. At all scales - from atomically thin surface films to chunks of wear particles - interfacial mechanics and dynamics play an important role in friction and wear. To address these problems, we are combining two approaches: 1) in situ macroscopic tribological studies allowing visualization and chemistry of the buried interface and 2) 'hybrid' nanoindentation, coupling high spatial resolution and surface sensitive, quantitative mechanical properties measurements of films as thin as a few nanometers. Using these techniques, the macroscopic tribological behavior can be correlated with mechanical properties of the nanoscale films and structures, e.g. the "third bodies" found in the sliding interface, yielding a powerful approach to examine worn surfaces and interpret tribological response. For example, one can learn "how" third body films form on the stationary counterface, "what" are their composition and mechanical properties, and "why" they provide low friction and prevent wear. We give examples from combined nanomechanics and tribological studies of MoS2 thin films The role of the transfer film mobility and mechanical properties in controlling friction behavior will be discussed.

SESSION U9: Mechanical Properties
Chairs: Yang-Tse Cheng and Oliver Kraft
Thursday Morning, December 4, 2003
Room 210 (Hynes)

8:30 AM *U9.1
The Mechanical Behavior Of Low K / Copper Interconnect Structures. T. M. Shaw[1], X-H. Liu[1], C. Murray[1], M. Y. Wisniewski[1], G. Fiorenza[1], M. Lane[1], S. Chiras[1], R. R. Rosenberg[1], R. Filippi[2], J. Mcgrath[2], H. Rathore[2] and V. Mcgahay[2]; [1]T.J. Watson Research Center, IBM Research Division, Yorktown Heights, New York; [2]IBM Microelectronics Div, East Fishkill, New York.

The introduction of low dielectric constant materials into back end of the line interconnect structures presents a number of mechanical challenges. In particular, polymeric based dielectrics with their high thermal expansion coefficient can result in mechanical strains being imposed on interconnects that exceed 1% during fabrication and thermal cycle testing. Also the lower modulus of the dielectrics results in a general loss of mechanical support for interconnects. The talk will present specific examples of how the thin film properties of low K dielectrics impact the mechanical behavior of interconnect structures. It will be shown that by careful consideration of the mechanical design of interconnect structures their mechanical integrity can be ensured.

9:00 AM U9.2
Determination of mechanical parameters for rotating MEMS structures as function of deposition method. Steve Bull[1], Sorin Soare[1], Anthony O'Neill[2], Nick Wright[2], Alton Horsfall[2] and Jorge dos Santos[2]; [1]CEAM, University of Newcastle, Newcastle upon Tyne, United Kingdom; [2]EECE, University of Newcastle, Newcastle upon Tyne, United Kingdom.

Reliability of interconnects is strongly dependent on the metallisation quality and, in modern processes, is crucially important to have the metallisation properties quantified. During wafer manufacturing an appreciable stress is generated in the metallisation and may lead to premature failure before the device has begun to operate. It has been shown that these stresses can be detected by a rotating structure. A finite element package has been used to model the behaviour of the structure in similar process steps to those used during metallisation. The input materials properties are very important in order to obtain results comparable to those produced experimentally. These were determined by nanoindention carried out on evaporated and sputtered aluminium films with thickness varying from 50 to 600nm. These aluminium layers were indented at various depths using proportional loading in order to minimise the effect of creep. It was found that the yield properties vary with the layer thickness and this has been observed also in the modelled loading curve. Using the right combination of yield stress - tangent modulus in the bilinear isotropic hardening materials model a good agreement between experimental and FE simulated data from thicker layers can be established. For thinner layers it becomes more difficult due to the presence of a considerable gradient in properties through the film thickness. The creep behaviour of the films has also been assessed from peak load holds and must be taken into consideration when modelling constant loading rate experiments.

9:15 AM U9.3
Direct observations of grain boundary phenomena during indentation of Al and Al-Mg thin films. Wouter A Soer[1,2], Jeff T De Hosson[1,2], Andrew M Minor[3], Miao Jin[3,4], Eric A Stach[5], Erica T Lilleodden[3] and J W Morris, Jr.[3,4]; [1]Department of Applied Physics, University of Groningen, Groningen, Netherlands; [2]Netherlands Institute for Metals Research, Delft, Netherlands; [3]Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, California; [4]Department of Materials Science and Engineering, University of California, Berkeley, California; [5]National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, California.

The deformation behavior of Al alloy thin films has been studied through the novel experimental technique of in situ nanoindentation in a transmission electron microscope. Here we investigate the role of alloying constituents in the transmission of plasticity across grain boundaries. Al thin films with varying amounts of Mg were deposited onto silicon substrates. The thickness of the deposited films was approximately 300 nm with an average grain size of the film thickness. The alloys included compositions both below and above the solubility level for Mg in Al. The presence of precipitates and/or solutes at the grain boundaries will be shown to affect grain boundary mobility. Initial results suggest that in the Al-Mg alloys the precipitates/solutes effectively pin the grain boundaries while in pure Al considerable grain boundary motion is observed. Additionally, comparisons will be drawn between the in situ TEM observations and quantitative measurements from conventional nanoindentation experiments of the same alloys.

9:30 AM U9.4
2D Analysis of the Grain-Size Dependent Plastic Relaxation in Thin Films. Lucia Nicola and Erik Van der Giessen; Netherlands Institute for Metals Research-University of Groningen, Groningen, Netherlands.

Stress relaxation in thermally loaded polycrystalline thin films is analyzed by discrete dislocation (DD) plasticity. The polycrystal is modeled as a 2D infinitely long array of rectangular grains. The orientation of each grain is specified by three slip systems. Grain boundaries as well as the film-substrate interface are treated as impenetrable for the dislocations. During cooling from a stress-free state, the semi-infinite elastic substrate progressively strains the film, which has a larger coefficient of thermal expansion. After a initial elastic response, the stress that develops in the film gets partly relaxed by dislocation glide. Simulations keep track of the stress evolution and of the dislocation dynamics in the film during thermal history. At a given time increment, the stress fields are computed by coupling the numerical solution of a boundary value problem to the analytical solution for the long-range fields of the dislocations, treated as line singularities in an infinite medium. Dislocation dynamics is governed by constitutive rules. DD simulations of single crystals [1] and of polycrystalline thin films [2] have shown a thickness-dependent response qualitatively similar to experimental findings [3, 4]. This paper aims to supplement this with an investigation of the grain size-dependence of hardening found in experiments [4, 5]. The power of the simple model presented here is that thickness dependence and grain-size dependence can be analyzed independently. References: [1] L. Nicola, E. Van der Giessen, A. Needleman, J. Appl. Phys. 93, 5920 (2003). [2] L. Nicola, E. Van der Giessen, A. Needleman, Proceedings of the MRS Spring 2003 meeting, San Francisco, April 2003, to appear. [3] O.S. Leung, A. Munkholm, S. Brennan and W.D. Nix, J. Appl. Phys. 88, 1389 (2000). [4] E. Arzt, Acta Mater. 16, 5611 (1998). [5] R. Venkatraman and J. C. Bravman, J. Mater. Res. 7 2040 (1992).

9:45 AM U9.5
Elastic and Plastic Properties of Niobium Pentoxide Films on Niobium. Christelle Callamand[1], Vijay Sethi[2] and Ronald Gibala[1]; [1]University of Michigan, Ann Arbor, Michigan; [2]Western Research Institute, Laramie, Wyoming.

By use of nanoindentation techniques, we have determined the elastic and plastic properties of [110]-oriented niobium single crystals and the amorphous anodic films of niobium pentoxide deposited on these crystals to thicknesses of 300 nm. The nanoindentation response of the Nb-Nb_2O_5 has been compared with that of similar tensile- and compression-deformed specimens investigated at lower temperatures and strain rates. The oxide film deforms compatibly with the niobium substrate, and the operative deformation mechanism in the the oxide film has been determined to be shear banding. The hardnesses of the oxide and metal are 12 GPa and 0.9 GPa, respectively. The Young's moduli are 180 GPa and 109 GPa, respectively. At indentation depths below approximately 500-600 nm, the film-coated niobium is harder than the corresponding uncoated material. At much larger indentation depths, e.g., 5000-6000 nm, the film-coated material is softer than the uncoated material, consistent with results on materials deformed in tension or compression at lower temperatures. The softening behavior is well explained in terms of enhanced dislocation nucleation into the niobium substrate from the film-substrate interface.

10:30 AM U9.6
Nanoindentation Study on MgO. Gang Feng and William D. Nix; Department of Materials Science and Engineering, Stanford University, Stanford, California.

There is a debate on whether the indentation size effect (ISE) is a measure of a true size effect in plasticity or an artifact due to oxide or deformation layers on the material surface. In this paper, indentations on both cleaved and epi-polished MgO surfaces showed significant Indentation size effects. The results for these two kinds of surfaces showed no observable differences, indicating that the ISE can be considered as an intrinsic property of crystalline materials. The model of Nix and Gao on the ISE predicts that the square root of hardness varies linearly with the reciprocal of indentation depth. The results of indentations on MgO showed that the model describes the indentation behavior well for indentation depths larger than 200nm. However, the data deviate from this law when the indentation depth is less than 200nm, with the model over-predicting the hardness in this regime. Three possible reasons for this deviation include: the imperfection of the Berkovich indenter, the effects of intrinsic lattice fiction, and the failure of the self-similarity rule at the nanometer length scale. These three points will be discussed. Using an etch-pit method, the dislocation configurations around indentations in MgO can be easily revealed by optical microscopy, SEM, and AFM; thus, some of the predictions of the dislocation theory of indentation can be verified directly. The relation between the size of the plastic zone and the indentation depth was studied to investigate the ISE. The effects of various indenter shapes on the ISE, including the Berkovich indenter, the cube corner indenter, and a spherical indenter were investigated. In addition, strain bursts in the early stage of indentation were also investigated. By comparing the indentation load-displacement curve with the dislocation configuration after indentation, strain bursts during indentation are shown to be associated with to local dislocation generation, even when there are pre-existing dislocations.

10:45 AM U9.7
Mechanical behavior of nano grained thin gold films. Jong Han[1], Aman Haque[2] and Taher Saif[1]; [1]Mechanical snd Industrial Engineering, University of Illinois at bUrbana-Champaign, Urbana, Illinois; [2]Dept. of Mechanical & Nuclear Engineering, The Pennsylvania State University, University Park, Pennsylvania.

We present an experimental apparatus to study mechanical behavior of nano scale free standing films. The apparatus is based on MEMS technology, and consists of a force and a displacement sensor to measure the stress and strain of the sample subjected to uniaxial tension. Co-fabrication of the sample and the micro sensors assures uniaxiality of loading and avoids any assembly. The apparatus is applied to study stress-strain response of gold films with thickness 80-350 nm and with grain size in the range of 15-100nm. We find that with decreasing grain size, elastic modulus of gold decreases, and gold behaves as a non-linear elastic material. In order to explore the role of anelasticity in reducing the elastic modulus, we carry of uniaxial tensile experiments at various temperatures and strain rates. It is found that elastic modulus decreases rapidly with temperature beyond 50C. At room temperatures, modulus seems to be independent of both strain rates and a change of temperature, although the value of the modulus is lower than the corresponding bulk value. The observation suggests that at room temperatures and below, anelasticity may not be the dominant mechanism for reduced modulus. As grain size decreases, the grain boundary region becomes abundant. The atoms within the grain boundaries are disordered compared to the grain interior, i.e., some atoms are closer to one another than the interatomic equilibrium distance, and some are farther apart. Thus, some atom pairs are subjected to attractive force, some are under repulsion giving rise to an overall force equilibrium. However, such disorder results in lower elasticity of the grain boundary. At small strains and low temperatures, when strained, a large part of the deformation is contributed by softer grain boundaries, and gold shows lower elastic modulus. Such deformation is elastic and reversible. As temperature increases, grain boundary diffusion becomes important, and gold behaves as an anelastic material.

11:00 AM U9.8
Micromechanical Properties of Pulsed-Laser Deposited Metal Films. James A Knapp[1], David M Follstaedt[1], Richard C Hugo[2,3] and Harriet Kung[2,4]; [1]Dept. 1111, Sandia National Laboratories, Albuquerque, New Mexico; [2]Structure/Properties Group, Los Alamos National Laboratory, Los Alamos, New Mexico; [3]Dept. of Geology, Portland State University, Portland, Oregon; [4]U.S. Dept. of Energy, Germantown, Maryland.

Ni, Cu and W metal films formed using pulsed laser deposition are very fine-grained and dense, showing little or no porosity under examination by TEM. We have deposited such films on fused silica, sapphire, and metal substrates. The grain sizes in the films, as characterized by electron microscopy, were varied by changing deposition rate and substrate temperature. The mechanical properties of the metal layers were determined by ultra-low load indentation that was analyzed using finite-element modeling. This analysis quantitatively determines the yield stress, Young's modulus, and hardness of the layer material from the indentation data, separating the properties of the thin films from those of the substrate. For example, results from Ni films are consistent with the empirical Hall-Petch relationship for grain sizes ranging from microns to as small as ~10 nm diameter, with the finest-grained layer showing a hardness of 12.6 ± 1.9 GPa. Selected films were examined with TEM during in-situ tensile straining, clearly showing evidence of dislocation-mediated deformation and ductility. The observed properties and mechanical behavior of these thin films suggests that pulsed-laser deposition forms nearly ideal materials, with potential for applications requiring hard, ductile metal films. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, supported by the US Department of Energy under contract DE-AC04-94AL85000. This work was partially supported through their Office of Basic Energy Sciences.

11:15 AM U9.9
Experimental Measurements of Surface Residual Stresses caused by Nano-scale Contact of Rough Surfaces. Junlan Wang[1], Pranav Shrotriya[1], HongHui Yu[2] and Kyung-Suk Kim[1]; [1]Division of Engineering, Box D, Brown University, Providence, Rhode Island; [2]Mechanical Engineering, The City College of The City University of New York, New York, New York.

A model analysis predicts that nano-scale contacts of surface steps induce nucleation of dislocations leading to pro-load and anti-load dislocation segregation near the contact surface. Such dislocation segregation generates a sub-layer of tensile residual stress in a much thicker layer of compressive residual stress near the surface. The sub-layer thickness is expected to be about 50 to 100 times of the step height. Experiments are performed on single crystal gold, polycrystalline aluminum and nano-crystalline nickel surfaces. In the first set of experiments with single crystal gold and nano-crystalline nickel, contact pressure is applied on the specimen surface using an atomically flat mica surface. In the second set of experiments, a fine polished stainless steel surface is used to press the polycrystalline aluminum surface. The statistical distribution of dislocation nucleation is measured with a surface roughness evolution spectroscopy (SRES) as the step heights of the surface evolve due to the contact loading. The variation of the residual stress along the thickness direction is measured with a newly developed high sensitivity curvature-measurement interferometer. The interferometer measures the curvature change of the back surface of a plate specimen of about 3mm thickness while the contact-loaded front surface is chemically etched. The residual stress distribution measured with sub-nanometer spatial resolution is compared with analytical predictions.

11:30 AM U9.10
Thickness-Dependence of the Yield Stress of Cu Thin Films on Kapton. Denis Y Yu and Frans Spaepen; DEAS, Harvard University, Cambridge, Massachusetts.

Cu films with thicknesses ranging from 0.2 to 3 μm on 13 μm Kapton substrates are tested with a microtensile tester with in-situ strain measurement by optical diffraction. The mechanical properties of the Cu films are determined by subtracting the contribution from the substrate. The modulus of the Cu films is independent of film thickness. The yield stress, σ_y, increases with film thickness, t, according to a Hall-Petch relation, $\sigma_y = \sigma_o + k\ t^{-1/2}$. The variation in microstructure through the film thickness is measured by cross-sectional focused ion beam microscopy. The effect of grain size and twin size on σ_y is investigated, showing that they alone cannot account for the large observed strengthening. Geometrically necessary dislocations appear to make a substantial contribution.

11:45 AM U9.11
Size Effects In Mechanical Testing Of Gold At The Microscale. Julia Rosolovsky Greer, Feng Gang and William D Nix; Materials Science and Engineering, Stanford University, Stanford, California.

The classical laws of materials science dictate that the mechanical properties of materials are independent of sample size; however the results of recent experiments in nanoindentation and molecular dynamics simulations suggest a strong size effect at the micron and sub-micron scales. In nanoindentation experiments, the surface of the film experiences permanent deformation upon its contact with the indenter, which results in a non-uniformity of stresses and strains within the test sample potentially responsible for the so-called indentation size effect (ISE). Indentation size effects have been widely observed in bulk materials and in thin films and manifest themselves as the apparent increase in hardness at shallow indentation depths. The observed ISE is explained in part by the strain gradient plasticity theory developed by Nix and Gao, which demonstrates a linear dependence between the square of the indentation hardness and the inverse of the indentation depth. While the Nix-Gao model explains the depth-dependent hardness for indentation depths above ~100nm, it cannot predict the observed discrete strain bursts characteristic of the elastic-to-plastic transition when much smaller volumes are tested. The discrete displacements found in single crystals during the initial stages of nanoindentation are most likely attributed to the nucleation of dislocations, the primary mechanism of plasticity on the nanometer scale. The results of molecular dynamics simulations indicate that plastic deformation at this scale is intrinsically inhomogeneous, that the yield strength depends on the sample size even in the absence of a strain gradient, and that for small single crystals, the yield strength scales with the surface area-to-volume ratio. Experiments on the mechanical properties of gold at the micron scale without the associated stress/strain gradients are reported in this paper. The test methodology involves two distinct fabrication processes for producing single-crystalline and polycrystalline free-standing cylinders of Au with sub-micron dimensions and the subsequent study of their uniaxial deformation properties. A nanoindenter with a flat punch tip is used to conduct these compression tests as it offers high-precision control of load, displacement, and nominal strain rate. The initial test results indicate that there is an increase in the flow stress with decreasing sample size.

SESSION U10: Properties and Performance
Chairs: Tom Buchheit and Steve Bull
Thursday Afternoon, December 4, 2003
Room 210 (Hynes)

1:30 PM U10.1
Mechanical Behavior of Amorphous ALD Alumina Films. John Jungk[1], Thomas Buchheit[2], Neville Moody[3], Thomas Mayer[2], Joel Hoehn[4], Steven George[5] and William Gerberich[1]; [1]Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, Minnesota; [2]Microsystem Materials, Tribology and Technologies, Sandia National Laboratories, Albuquerque, New Mexico; [3]Microsystems & Materials Mechanics, Sandia National Laboratories, Livermore, California; [4]Seagate Technology, LLC, Minneapolis, Minnesota; [5]Chemistry and Biochemistry, University of Colorado, Boulder, Colorado.

Atomic layer deposition (ALD) is a recently developed technique for producing thin, hard conformal coatings intended to improve the performance of microelectronic devices and microelectromechanical systems (MEMS). ALD is typified by the stepwise deposition of self-limiting half-reactions at a substrate. Through multiple half-reaction cycles, an ALD film may be grown until a specific thickness has been reached, producing dense, ultra-thin, highly conformal films with sub-monolayer precision. Films are usually grown to thicknesses ranging from 1-50 nm. This study investigates the mechanical characteristics of these films as a function of depth and film thickness through careful application of nanoindentation-based methods. Additionally, previous experiments on ALD-alumina films, deposited on silicon wafers with a native oxide, have implied that the native oxide acts as a debonding layer between the film and the silicon substrate. To further understand the role of the native oxide layer, ALD-alumina films were deposited on single crystal silicon wafers both with and without the layer. Film adhesion studies, evaluated through scratch testing, quantified the effect of this layer in ALD interfacial fracture. In this presentation, results from the property and adhesion studies will be discussed.
—————————————————— This work is supported by Seagate Technologies, Inc. through a MINT grant and a part of it was performed at Sandia National Laboratories. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States DOE under Contract

DE-ACO4-94-AL85000.

1:45 PM U10.2
Study of Anelasticity observed in pure Au films on Si substrates. Dae-han Choi and William D. Nix; Materials Science and Engineering, Stanford University, STANFORD, California.

Many techniques have been developed to study the mechanical properties of thin films on substrates; however, most present methods do not allow the dynamic mechanical properties of thin films to be examined. In the present work we have developed a testing system for studying the dynamic properties of thin metal films by measuring the free decay of the velocity of micromachined, bi-layer cantilever structures at various frequencies and temperatures. With this technique we are able to study anelastic, damping processes in thin metal films and identify the atomic processes responsible for these properties. A model system consisting of gold thin films on Si substrates has been chosen for study. Although gold has properties similar to other metals widely used in the IC industry, it does not form an oxide on its surface. Thus we are able to study damping without the presence of a passivating oxide. But we can simulate the effect of a native oxide by applying a very thin layer of tungsten to the surface of the gold film. In this way we are able to investigate the role of the passivating oxide in damping of thin metal films. We report on our studies of the anelastic behavior of pure gold films of various thicknesses, with and without passivating layers of tungsten. Various diffusional models will be presented to describe the observed damping processes and issues relevant to the applicability of these models will be discussed.

2:00 PM U10.3
Bulge Test on Free Standing Gold Thin Films. Yawen Li and Michael J Cima; Department of Materials Science and Engineering, MIT, Cambridge, Massachusetts.

The bulge test employs uniform pressure from underneath a free standing thin film to deflect it to the other side. There have been theoretical analysis and experimental studies that correlate the pressure with the film center deflection to extract its mechanical properties, such as residual stress and elastic modulus. Thin films used in conventional bulge tests generally have in-plane dimensions greater than 1mm. The gold thin films tested in this study, however, were prepared using the microfabrication process of a drug delivery MEMS device, and had in-plane dimensions ranging from 20 to 200mm. A direct consequence of this shrinking dimension is that the bending stiffness of the thin films, especially of those with in-plane dimensions smaller than 100mm, becomes important in the film deformation. Incremental pressure at 5psi interval was applied to the gold films, whose deflection was measured using interferometry. Finite element analysis was also performed using ADINA. Comparison of the experimental results with modeling extends the application of bulge test to thin films on smaller scales, and also provides useful knowledge of the mechanical integrity of the gold films, which has been known to closely correlate with the reliability of the drug delivery device.

2:15 PM U10.4
Microstructure-mechanical properties relationship of laser interference irradiated Ni/Al multi-film. Claus Daniel, Andres Fabian Lasagni and Frank Muecklich; Materials Science, Functional Materials, Saarland University, Saarbruecken, Germany.

Due to the corresponding intermetallic compounds, Ni/Al multi-layered thin film systems are important to protect the mechanical and chemical impact on the bulk component. The mechanical properties of these tough intermetallic compounds, NiAl, can be further improved by combining with other stiff phases. Especially, if the lateral surface composite can be made in such a way that the different phases are arranged periodically with a preferred orientation, micro-scaled period and reticulated phase interfaces, the mechanical properties would be optimised. Such optimised surface composites have been achieved by laser interference irradiation in a nano-grained structure. In this study, the thin film systems are produced by physical vapour deposition and subsequently irradiated by the interference pattern of two or more coherent laser beams. The corresponding periodical heat treatment has been analysed by thermal simulation and compared with the experimental results. Further, the phase transitions during laser interference irradiation are calculated. The structural investigations of irradiated films - grain sizes and deformation by TEM, stress and texture by XRD - are compared with the mechanical properties - hardness and Young's modulus by nano-indentational AFM.

2:30 PM U10.5
Stresses in (111) and (100) texture components in Cu thin films measured using synchrotron x-ray diffraction.
David E Nowak, Jonathan B Shu and Shefford P Baker; Materials Science & Engineering, Cornell University, Ithaca, New York.

Synchrotron x-ray diffraction experiments were used to study the thermomechanical behavior and isothermal stress relaxation of individual texture components in a Cu thin film. The film was deposited to a thickness of 500 nm on a SiN_x barrier layer on a Si substrate and then passivated with SiN_x. For the thermomechanical experiments the film was cycled from room temperature to 500°C. After cycling, the film was highly textured with grains having (111) and (100) parallel to the plane of the film. The stress in the plane of the film was determined as a function of temperature from plane spacings in each of these texture components measured using x-ray diffraction. The stresses resolved onto the slip planes were nearly identical for both texture components, indicating that the in-plane stress levels for the two textures are a function of slip plane orientation. Isothermal relaxation experiments were conducted at 150°C after heating from room temperature and at 100°C after cooling from 500°C. The isothermal relaxation at 100°C on cooling shows a typical relaxation behavior in both texture components. The isothermal relaxation at 150°C on heating shows anelastic stress recovery in both texture components. These results provide insight into the details of plastic deformation and stress levels in thin films.

2:45 PM U10.6
Indentation and FEA modelling investigations of the indentation size effect in aluminium coatings on borosilicate glass substrates. Ian Spary[1], Nigel M Jennett[2] and Andrew J Bushby[1]; [1]Materials Department, Queen Mary, University of London, London, United Kingdom; [2]NPL Materials Centre, National Physical Laboratory, Teddington, Middlesex, United Kingdom.

Nanoindentation is one of the few available methods and the most commercially widespread technique for investigating the elastic and plastic properties of small volumes such as thin films. Quantitative methods for obtaining the indentation (plane strain) modulus and hardness of a coating have been published and FEA models of the elastic-plastic response of indentation have been developed. Comparison of the FEA model output with actual indentation data has shown that, as the indentation size reduces, the apparent yield stress of the material increases. We have shown that the increase in yield stress is predictable and falls on a master curve (MRS paper U4-9, Spring meeting 2003). Predictions have been tested and agree for a range of metals (Cu, Al, W, Ir). This points to there being a fundamental length scale for dislocation-based deformation and raises the question as to whether the yield stress of thin films may be altered by reducing thickness. This study therefore investigates the indentation response of Al coatings on Borosilicate glass as a function of coating thickness and indentation depth. FEA models of the indentation contact will be compared with indentation data and AFM measurements of the surface profile to investigate the relative contributions of the indentation size effect and the effect of hardening due to the additional constraint of substrate proximity to the plastic zone. Results from the models are further validated by comparison with AFM measurements of pile up around the indentation site.

3:30 PM *U10.7
An Inchworm Actuator for MEMS Tribology Studies.
Maarten P. de Boer and Alex D. Corwin; Reliability Physics, Sandia National Labs, Albuquerque, New Mexico.

We have developed an inchworm actuator that enables a wide range of in-situ MEMS friction studies. The actuator consists of a plate that spans two frictional clamps, and occupies an area of 600 microns by 200 microns. By appropriately timing signals to the plate and to the clamps, the actuator can walk long distances (200 micrometers) in 40 nanometer steps. It employs a mechanical amplification scheme that achieves a large tangential force (1 milliNewton) against a stiff non-linear load spring. Normal load is applied electrostatically to the friction clamps in the range from 1 microNewton to 10 milliNewtons. Using the stiff load spring for measurements, we observe that the static coefficient of friction (*cof*) can be varied from 0.15 to 1.05 by applying different molecular monolayer coatings. Also, the static *cof* is shown to be independent of normal load over a broad range. However, using a weak linear load spring, we find that both dynamic and static frictional forces exist at zero applied normal load, and can quantitatively attribute this to adhesive forces. Even in air, adhesion-induced dynamic friction loss is responsible for 40% of the total energy dissipation. We also investigate the relevance of static friction to the actuator operation. Before the static friction force limit is reached, the friction clamps exhibit pre-sliding tangential deflections up to 200 nanometers in length. Because this distance is much longer than the step size of 40 nm, frictional interface stress-strain curves rather than the *cof* govern the operation of the actuator. This work has resulted in a better understanding of how friction can be utilized for high performance actuation and nanoscale positioning in MEMS.
Acknowledgment: Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security

Administration under Contract DE-AC04-94AL85000.

4:00 PM U10.8
Nanoscale Dynamic Mechanical Analyses of Polymers. Thomas J. Mullen[1], S.A. Syed Asif[2] and Kathryn J Wahl[1]; [1]U.S. Naval Research Laboratory, Washington, District of Columbia; [2]Hysitron, Inc., Minneapolis, Minnesota.

Time dependent mechanical properties of thin polymer films are of considerable interest for a diverse array of applications spanning biomedical, microelectronics, adhesive, lubrication, and paint technologies. Standard dynamic mechanical analyses techniques rely on large specimens (typically several cm in diameter) and relatively thick films (fraction of mm or thicker). Evaluating the mechanical properties of small or composite samples and submicron thickness films is possible if a smaller sample probe is used. In this paper, we present an approach for measuring the dynamic mechanical properties of thin, compliant polymer films using AC force-modulation coupled with a hybrid scanning indenter. This combination allows surface sensitive, quantitative mechanical properties measurements at a single point as well as while scanning. Dynamic response of the indenter is monitored during tip-sample approach, enabling sensitive detection of the surface, and during contact for evaluation of storage and loss moduli of polymer samples. Examples of dynamic mechanical properties analyses using force-displacement curves, frequency sweeps, and imaging will be presented for polyethylene, poly(dimethylsiloxane) (PDMS) and commercial adhesives. Results are in good agreement with macroscopic rheological measurements. Issues including strain rate sensitivity and measurement geometry will be discussed.

4:15 PM U10.9
Nanoscale Deformation Fields and Mechanical Properties of MEMS Materials. Ioannis Chasiotis and Sungwoo Cho; Mechanical and Aerospace Engineering, University of Virginia, Charlottesville, Virginia.

Sub-micron mechanical measurements in thin films require high precision in both force and displacement data. These measurements become more challenging to obtain when freestanding micron-scale thin film specimens are employed. With regards to freestanding MEMS samples, very few methods are currently available that allow obtaining both the forces and the displacements directly on specimens that often measure only a few microns in dimensions. This talk reports on our methodology to obtain the sub-micron and nanometer scale full-field mechanical deformations in thin film structures of different geometries and dimensions. Test samples have been fabricated of polycrystalline silicon at Cronos via the MUMPs process [1], and of hydrogen-free tetrahedral amorphous diamond-like carbon (ta-C) at the Sandia National Laboratories (SNL) [2]. The deformation and strain characterizations have been conducted via a microtensile apparatus that has been integrated with an Atomic Force Microscope (AFM). Based on high resolution (1000x1000 pixels) AFM images obtained on micron-scale specimens, the full-field deformations have been computed via Digital Image Correlation (DIC) algorithms [3]. This methodology provided full field displacements in 5x10 micron (or smaller) areas with resolution better than 2-3 nm. By utilizing direct local strain measurements in the vicinity of micronotches (radius of curvature 1-8 microns) and finite element analyses of local stress concentrations (as acute as K=27) a linear elastic behavior has been shown to hold in highly stressed sub-micron areas in brittle polycrystalline and amorphous thin films. For those specimen geometries, the local failure strength was as high as 12 GPa for ta-C and 3.5 GPa for polycrystalline silicon. This mechanical measurements methodology provides the background for the full-field characterization of inhomogeneous and anisotropic thin film materials and devices. In this frame, its application to obtain the local inelastic deformations in thin metal films employed in RF-switches will be discussed. [1] I. Chasiotis, W.G. Knauss, "The Mechanical Strength of Polysilicon Films: 2. Size Effects Associated with Elliptical and Circular Perforations", J. of the Mechanics and Physics of Solids 51 (8), pp. 1551-1572, (2003). [2] I. Chasiotis, S. Cho, T.A. Friedman, and J. Sullivan, "Mechanical Properties of Tetrahedral Amorphous Diamond-like Carbon (ta-C) MEMS", Proceedings of the Annual Conference of the Society for Experimental Mechanics, pp. 170-175, (2003). [3] I. Chasiotis, W.G. Knauss, "A New Microtensile Tester for the Study of MEMS Materials with the aid of Atomic Force Microscopy", Experimental Mechanics 42 (1), pp. 51-57, (2002).

4:30 PM U10.10
Hinge optimisation in a micro-rotating structure for stress gauging in integrated circuit interconnects.
Alton Barrett Horsfall[1], Jorge dos-Santos[1], Sorin Soare[2], Adrian Oila[2], Steve Bull[2], Nick Wright[1], Anthony O'Neill[1], Anthony Walton[3], Alan Gundlach[3] and Tom Stevenson[3]; [1]School of Electrical, Electronic and Computer Engineering, University of Newcastle, Newcastle, United Kingdom; [2]School of Chemical Engineering and Advanced Materials, University of Newcastle, Newcastle, United Kingdom; [3]Scottish Microelectronics Centre, University of Edinburgh, Edinburgh, United Kingdom.

The process-induced stress in interconnects within integrated circuits has a direct influence on the mean time to failure of the devices. To satisfy the requirements of a fast developing microelectronics industry, there is a demand for interconnect track widths to decrease and the number of levels of metallisation to increase. This requires researchers to deliver high quality, stress free metallisation since stressmigration can result in voids and hillocks resulting in open and short circuit conditions in interconnect features. Since measurement of stress in individual metallised lines is not possible by existing techniques another approach has been adopted where a test structure is generated during fabrication based on a micro-rotating cantilever sensor. The two fixed beams are connected off-centre to the opposite sides of the central rotating beam, using hinges consisting of 90 degree angles. Thus, the displacement of the fixed beams due to the released residual stress leads to the deflection of the rotating beam. To support the design finite element modelling has been performed. After processing, the sensor rotation was measured from reflected light micrographs. By comparison of the rotation predicted by finite element simulation and that observed experimentally, a clear discrepancy is observed which is critically dependent on the details of the sensor design and the pattern transfer of the lithographic process. To obtain more accurate results, a new degree of complexity for the finite element modelling must be implemented. Realistic geometry must be considered, together with the inclusion of materials properties (elasticity, plasticity, and creep) which have been obtained by nanoindentation. The full paper will present details of different sensor geometries (i.e., new hinge designs) used to optimise the sensor's sensitivity. More complex structures show a lower plastic deformation in the hinge region and enhanced rotation.

4:45 PM U10.11
Modelling and Optimal Design of Multilayer Cantilever Microactuators. Lianghao Han and T.J. Lu; Department of Engineering, University of Cambridge, Cambridge, United Kingdom.

Thermal expansion is an important actuation mechanism for microelectrmechanical system (MEMS). Thermally-actuated multiplayer cantilever microactuators, based on the thermal expansion coefficient difference of different layers, have been successfully implemented in MEMS applications such as optical microprojector, bubble jet printer and optical storage etc. We investigate multiple-layered cantilever microactuators actuated by resistive heating with an electric current. The actuator consists of films and sandwiched microheaters. An electro-thermal-elastic model for characterizing microactuators is developed. The general analytical expressions relating tip deflection with an applied electric field, temperature change, the film thickness and the applied load at the tip are obtained. The large deflection is considered in the model. The effect of cantilever width is also demonstrated using 3D finite element analysis. Based on the analytical solutions and a mixed variable programming (MVP) algorithm, the optimal design of the actuators is presented. A load is vertically applied at the tip of a multiplayer cantilever to simulate a weight. When actuated, the cantilever can lift the load over a specified minimum displacement. The optimization establishes the authority (product of end displacement and load to be lifted), at specified electric field by determining the minimum weight subject to the power consumption limitation, failure mechanisms (yielding and buckling) and geometry constraints. The algorithms optimize simultaneously with respect to the number of films and the material choices from a list of materials (or material database). The optimal parameters of the constituents, such as the number of films, the thickness ratios of different films, total thickness of the cantilever and material types of the films at the minimum weight emerge from the analysis. A discussion of modeling and design considerations is also presented.

SESSION U11: Poster Session
Chairs: Sean Corcoran, Young-Chang Joo, Neville Moody and Zhigang Suo
Thursday Evening, December 4, 2003
8:00 PM
Exhibition Hall D (Hynes)

U11.1 TRANSFERRED TO U5.31

U11.2 TRANSFERRED TO U7.7

U11.3
Mechanical Properties and Morphology of Polycrystalline 3C-SiC Films Deposited on Si and SiO2 by LPCVD.
Xiao-an A Fu[1], Jeremy L Dunning[1], S. Rajgopal[1], Ming Zhang[2],

Christian A. Zorman[1] and Mehran Mehregany[1]; [1]Electrical Engineering, Case Western Reserve University, Cleveland, Ohio; [2]Material Science and Engineering, Case Western Reserve University, Cleveland, Ohio.

SiC is an exceptionally attractive material for microelectromechanical systems (MEMS) because of its outstanding mechanical, chemical and electrical properties. Currently, polysilicon is the dominant material in MEMS, owing in part to high throughput deposition processes that yield films with favorable electrical and mechanical properties, thus setting the standard for materials such as SiC. Recently, advances in deposition technology have resulted in a LPCVD system for polycrystalline 3C-SiC (poly-SiC) that compares with polysilicon in terms of throughput [1]. Previous work investigating 3C-SiC films deposited on Si and SiO2 by APCVD indicates that the surface morphology and microstructure depend on the substrate material [2]. In this work, the residual stress, surface morphology, and microstructure for poly-SiC thin films grown on Si and SiO2 by LPCVD was characterized. The poly-SiC films were deposited on Si and SiO2 substrates in a large-volume, LPCVD furnace using dichlorosilane (DCS) and acetylene precursors. The deposition temperature and pressure were fixed at 900 C and 2 Torr, respectively, while the flow rate of DCS was varied between 18 and 72 sccm. A clear relationship between residual stress and DCS flow rate was observed, with tensile stresses occurring at low flow rates and compressive stresses at high flows. To the best of our knowledge, this is the first time that such a relationship has been observed for poly-SiC. XPS indicated that stoichiometric SiC films were deposited on Si and SiO2 over the entire flow range. X-ray diffraction showed that all films on Si and SiO2 were highly textured (111) 3C-SiC regardless of flow rate. The surface morphology and roughness of poly-SiC on Si and SiO2, as determined by AFM and SEM, indicated that the surface features were spherulitic aggregates, and the surface roughness increased with increasing film thickness. TEM analysis is ongoing and will be detailed in the extended paper. [1] C.A. Zorman, S. Rajgopal, X.A. Fu, R. Jezeski, J. Melzak, M. Mehregany, Electrochem. Solid State Lett., 5, (2002) G99. [2] C.H. Wu, C.A. Zorman, and M. Mehregany, Thin Solid Films, 355-356, (1999), 179.

U11.4
Residual Stress in Silicon Nitride Thin Films Deposited by ECR-PECVD. Elena Cianci and Vittorio Foglietti; Istituto di Fotonica e Nanotecnologie- CNR, Roma, Italy.

We have investigated the influence of process parameters in electron cyclotron resonance plasma enhanced chemical vapour deposition (ECR-PECVD) of silicon nitride, in order to obtain SiN_x films with optimal mechanical properties for micromechanical sensors. We have mainly studied the effect of silane to nitrogen flow rates ratio on the intrinsic stress of thin SiN_x films, being the control over stress highly desirable in MEMS fabrication process. Characterization of the stress in silicon nitride films deposited at 330°C was performed by means of passive strain sensors and showed that stress can be tailored over a wide range, from compressive stress using low SiH_4:N_2 flow ratio R, to tensile stress within a large window of R from 0.3 to 0.8, with a maximum value of 500 MPa, and then back to compressive stress for higher R. Samples were analyzed by FTIR for hydrogen content and by spectroscopic ellipsometry for refractive index n. Using the effective medium approximation (EMA) silicon nitride films were modelled as a mixture of Si_3N_4, amorphous silicon and voids. For SiH_4:N_2 flow ratio of 0.75 we found the minimum hydrogen concentration in the film, with hydrogen bonded both to silicon and nitrogen, and the minimum fraction of a-Si in the EMA model of the film. Increasing silane flow, SiN_x films become silicon-rich (n>2, increased fraction of a-Si in the EMA model, and high Si-H bond density); while for R <0.75 we found high percentage of voids and all H bonded to N, indicating that silicon nitride is nitrogen rich (n<2). In this way, the gas flow ratio was found to be a critical parameter for the tuning of the intrinsic stress in ECR-PECVD SiN_x films, and the composition of the films.

U11.5
Computer Simulation Of Spinodal Decomposition In Constrained Films With An Arbitrary Distribution Of Dislocations. Shenyang Hu, Yulan Li, Dongjin Seol and Long-Qing Chen; Department of Materials Science and Engineering, The Pennsylvania State University, University Park, Pennsylvania.

Spinodal decomposition in binary alloy thin films with an arbitrary distribution of dislocations and subject to substrate constraint is studied by phase-field modeling. Elastic solutions derived for elastically anisotropic thin films with arbitrary eigenstrains under stress-free surface and substrate constraint are employed in the three dimensional phase-field simualtion. Temporal evolution of Cahn-Hilliard equation under thin film boundary conditions (i.e. the fluxes normal to the surface and the film/substrate interface are zero) is solved by a semi-implicit Fourier spectral method. Numerical simulation without considering the coherency strain and dislocations illustrates that the morphology of decomposed phases depends on the film thickness and the composition. Within an appropriate range of the composition, the phase separation with coherency strain in an elastically anisotropic film shows the appearance of surface-directed spinodal decomposition. Negative elastic anisotropy in the cubic alloy causes the alignment of the phase along <100> elastically soft direction. The presence of the dislocations significantly affects spinodal decomposition process, hence the morphology.

U11.6
Surface Stress Induced Relaxation of Nanoporous Metallic Films. Douglas Crowson, Diana Farkas and Sean G Corcoran; Materials Science and Engineering, Virginia Tech, Blacksburg, Virginia.

Bicontinuous interconnnected nanoporous metallic bulk and thin films can be created by an alloy corrosion process known as dealloying. The resultant porous materials have extremely high surface area and can be tailored with pore sizes ranging from 3 nm to greater than 50 microns. These films are ideally suited for thin film sensor applications. One of the outstanding questions regarding these materials is the mechanism of their inherent brittle behavior and the role of surface stress on the stability of the structures. This paper presents embedded atom simulations (EAM) of the relaxation of nanoporous Au films as a function of pore fraction, pore size and surface stress. Owing to the size scale of the porosity, EAM techniques can be applied directly to "real" material length scales. The methods of generating a simulated nanoporous metal representative of real materials and compatible with embedded atom simulation will be presented. We will also review the experimental methods for creating nanoporous thin films and their characterization by neutron scattering.

U11.7
the stress of nanocrystalline diamond film deposited by ion bombardment. Changzhi Gu and Wei Liu; Institute of Physics, Chinese Academy of Sciences, Beijing, China.

Nanocrystalline diamond film is prepared by continuous H+ ion bombardment under various energies induced by applying a negative bias voltage at the substrate relative to the grounded vacuum chamber using hot filament chemical vapour deposition (HFCVD) method. The effects of substrate bias current density, temperature, total pressure and CH4 concentration on stress of a as-grown film are investigated. The results indicate that high bias current density and substrate temperature are benefit to synthesize smooth nanocrystalline diamond film, which generally relates to high compressive stress, this can be attributed to the high secondary nucleation rate and grain boundary density. The changes of stress presents complicated process at various pressures and CH4 concentrations. The film compressive stress can be controlled by modifying the grain size at different deposition parameters, and part of compressive stress can be released under suitable annealing. condition

U11.8
A Study on Etching of SiGe Layers in SiGe/Si Systems for Device Applications. Takashi Yamazaki, Tomohide Sekikawa, Shinya Morita, Yoshitaka Hakamada, Hiroyuki Ohri, Shun-ichiro Ohmi and Tetsushi Sakai; Department of Information Processing, Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan.

The selective etching of SiGe layers for SiGe/Si systems was investigated for device applications. The one of the typical application of the selective etching is to fabricate the multi Si channel layers in multi-layer channel MOSFET (ML-MOSFET) proposed by Sakai et al. [1]. The multi Si channel layers are fabricated by the selective etching for Si/SiGe stacked layers grown by LPCVD. Selectivity on the etching of SiGe layer on Si has been reported by using HNO_3:H_2O:HF solution [2], so that only the SiGe layers in SiGe/Si stacked layers are laterally etched after channel patterning. This method is very attractive for various device applications. However, it was found that the etching rate was strongly depended on the strain in the layers. In this paper, the effect of annealing on the selective etching for Si/SiGe stacked layers was studied to investigate the effect of strain in the SiGe layers for selective etching. The SiGe (30 nm) and cap-Si (30 nm) layers were grown on Si (100) by LPCVD at 500°C and 550°C, respectively. The Ge content in the SiGe layers evaluated by SIMS measurement was 20%. Then, the samples were annealed at 650°C or 1000°C for 30 min in N_2. The Si/SiGe layers were patterned by RIE, and then the SiGe layers were laterally etched by HNO_3(61%):H_2O:HF(50%)=60:180:1 solution. The strain was evaluated by X-ray diffraction (XRD), and the cross-sections of the samples were observed by scanning electron microscopy (SEM). From the XRD spectra, the SiGe peak of the sample annealed at 650°C did not change compared to the as-grown sample. On the other hand, the peak after 1000°C annealing shifted to higher angles, which indicates the strain was relaxed by the annealing process. For the as-grown samples and even after 650°C annealing, the SiGe layers were able to be selectively etched. The laterally etched depth was confirmed as 430 nm, and no mechanical damage was observed in the remained Si layers by SEM observation. However, the SiGe layer of the sample annealed at 1000°C was not able to be etched. Similar results were observed, when the thickness of SiGe layers was relatively thick such as 75 nm. From these results, it is considered that the strain in the SiGe layers acts an important roll for the selective etching in SiGe/Si systems. [1] T. Sakai, S. Ohmi, D. Sasaki, M. Sakuraba and J. Murota, International SiGe Technology and Device Meeting, Meeting Abstract, pp.31-32 (2003). [2] A. H. Krist and D. J. Godbey, Appl. Phys. Lett., pp.1899-1901 (1991).

U11.9
InAs epilayers grown on GaAs substrates by using the prelayer technique. L.C. Cai, H. Chen, C.L. Bao, Q Huang and J.M. Zhou; Institute of Physics, Chinese Academy of Sciences, Beijing, China.

The different growth conditions of InAs /GaAs is examined by X-ray double-crystal diffraction, optical absorption and low temperature photoluminescence (PL). X-ray diffraction indicates that the strain is fully relaxed within 7nm thick InAs layers by 90o misfit dislocations under In-rich conditions. The absorption spectra show that InAs grown under In-rich conditions has no absorption at band edge. The optimized growth condition for high quality InAs epilayers from low temperature PL is following: first InAs is grown 20nm thick under In-rich conditions at 500oC with the appropriate V/III ratio of 8, then InAs is continuously grown under As-rich conditions at 500oC. The new way to obtain the high quality InAs epilayers on the GaAs substrates is a two-step growth process: InAs grown directly on the GaAs substrates as the prelayers under In-rich conditions, and then growth of the InAs layers on such prelayers under As-rich conditions.

U11.10
Forces that Drive the Self-assembly of Nanovoids on Solid Surface. Wei Lu, Mechanical Engineering, University of Michigan, Ann Arbor, Michigan.

The ability of producing ordered nanoscale structures is important for many modern technological applications. It was long known that voids may nucleate in a metal when it is subjected to irradiation at elevated temperature. Sometimes the voids, with diamaters and distances in 10~100nm, may order into a regular superlattice. We developed a thermodynamic model to explain and simulate the remarkable phenomena. Surface stress was found to play a key role in stabilizing the distance between two voids. In an isotropic solid, the elastic energy decreases when two voids come closer, which induces attractions between voids. Surface stress, which causes the surface energy to depend on the strain state, induces repulsion. The competition determines the void size and arrangement. Our simulation suggests a significant degree of experimental control in growing superlattice of nanovoids.

U11.11
Effect of Substrate Constraint on the Magnetic Domain Structures in Thin Films. Jingxian Zhang, Yulan Li and Longqing Chen; Department of Materials Science and Engineeering, Penn State University, University Park, Pennsylvania.

A phase-field model for investigating the effect of substrate constraint on the ferromagnetic domain structure evolution in ferromagnetic thin films is developed. The Landau_Lifshitz equations of magnetization dynamics are employed to describe the ferromagnetic domain structure and domain evlution. The magnetoelastic energy induced by substrate constraint and coherent strain is included in the total free energy besides the magnetocrystalline anisotropy energy, exchange energy and stary field energy. It is calculated based on an analytical elastic solution derived for a ocnstrained film with arbitrary eigenstrain distributions. The effect of the substrate constaint on the magnetic domain structures in the film is simulated and discussed.

U11.12
Universal Scaling Behavior in the Indentation of Bi/Unixial Strained Materials. J. David Schall and Donald W. Brenner; North Carolina State University, Raleigh, North Carolina.

Through the use of molecular simulations, we show that changes in contact area during plastic indentation of materials under applied in-plane stress lead to large changes in measured hardness and modulus when areas are estimated using an elastic half-space model. Our simulations demonstrate that these changes, first reported for macro-scale hardness measurements dating back to 1932, also occur for contact regimes at the nanometer scale. This plastic behavior represents a universal material property that apparently scales over 10 orders of magnitude in contact area, from ~mm2 to ~100nm2. As suggested by Tsui, Pharr, and Oliver, direct measurement of contact impressions shows that these changes in modulus and hardness are falsely represented by the elastic half-space model. With accurate measurement of contact areas left by plastic indentation, modulus and hardness are found to be independent of the applied in-plane stress. Additional analysis of the simulated loading curves shows that for our nanoscale contact area, indentation prior to yielding can reflect real changes in surface elastic properties with in-plane stress, a result that is consistent with second-order elastic analysis of bulk single crystal material.

U11.13
Indentation Curve Analysis for Pile-up, Sink-in and Tip-Blunting Effects in Sharp Indentations. Yeol Choi[1,2], Baik-Woo Lee[1], Ho-Seung Lee[1] and Dongil Kwon[1]; [1]School of Materials Science and Engineering, Seoul National University, Seoul, South Korea; [2]Research Center, Frontics, Inc., Seoul.

Hardness and elastic modulus can be derived from instrumented sharp indentation curves by considering the effects of materials pile-up or sink-in, tip blunting, surface roughness, and so on. In particular, this study quantifies pile-up and sink-in effects. Two approaches, finite element simulation and theoretical modeling, were used to describe the detailed contact morphologies including aforementioned effects. The ratio of contact depth to maximum indentation depth was proposed as a key indentation parameter for detailed analysis. The parameter selected was found to be independent of indentation load and to be a material constant as a function of mechanical properties. In addition, it could be evaluated strictly in terms of indentation-curve properties, such as loading and unloading slopes at maximum depth and the ratio of residual depth to maximum indentation. These relationships between indentation parameters were verified by finite element simulations. Furthermore, a fitting analysis of the loading curve was made to handle accurately and quickly the combined effects of indenter tip blunting and specimen surface roughness. Nanoindentations were made on fused quartz, glass and pure Cu and surface profiles after indentation were observed by atomic force microscope. Finally, the analysis and observation results were compared to verify the newly developed indentation-curve analysis method.

U11.14
Determining stress-strain curves for thin films by experimental/computational nanoindentation. Baik-Woo Lee, Yeol Choi, Yun-Hee Lee, Ju-Young Kim and Dongil Kwon; School of Materials Science and Engineering, Seoul National University, Seoul, South Korea.

The ongoing miniaturization of thin films, coatings, and micro-electromechanical system (MEMS) components demands new tools and methods for characterizing mechanical properties. The nanoindentation technique is very promising for evaluating mechanical properties such as nanohardness and elastic modulus at the micrometer- or nanometer-scale level, since sample preparation and testing procedures are very simple. However, the nanohardness cannot be directly related to basic material-flow properties. Thus, many attempts have recently been made to extract stress-strain behavior from nanoindentation test results: a scaling approach based on dimensional analysis, empirical testing with a spheroconical indenter, and a trial computational load-depth curve matching method have all been explored. Here an efficient experimental/computational method is proposed to extract stress-strain curves based on finite-element modeling (FEM) of nanoindentation. The stress-strain curves, assumed to be expressed as linear stepwise curves, were determined when the load-depth curves calculated by FEM matched those measured experimentally. We were able to do this effectively by finding the relationship between the load for nanoindentation deformation and the modulus of linear segments in the stepwise curves. The relationship was derived from the constitutive equations for the materials taking into account pile-up and sink-in effects (locally severe deformations around the contact). This method was applied to bulk Al and various thin films deposited on a Si-substrate and verified by comparing the stress-strain curve extracted with that obtained in tensile testing. This method permits determination of stress-strain curves at very small scales that cannot be easily obtained by other methods.

U11.15
Effects of External Electrode and Heat Treatment on Stress Anisotropy and Residual Stress in $BaTiO_3$-based Ni-MLCCs. Dong-Ho Park[1], Yeon-Gil Jung[1], Ungyu Paik[2], Jin-Young Yu[3] and Jeong-Wook Kim[3]; [1]Department of Ceramic Science and Engineering, Chagwon National University, Changwon, South Korea; [2]Department of Ceramic Engineering, Hanyang University, Seoul, South Korea; [3]Samsung Electro-Mechanics Co., Ltd, Suwon, South Korea.

Herein, we report the effects of external electrode and heat treatment on stress anisotropy and residual stress in each direction and plane in margins of multilayer ceramic capacitors (MLCCs). The residual stress estimated using an indentation method is compared in regions with and without the external electrode as heat treatment parameters such as heating time, temperature, and heating/cooling rates. As a result, the stress anisotropy was not changed with heat treatment parameters, independent of the external electrode. However, the residual stress depended on the direction and plane, showing the different stress terms. In margins with the external electrode, the residual stresses at the x-z plane parallel to lamination were -58.3 ± 23.5 and 10.5 ± 15.4MPa in the directions parallel and perpendicular to the internal electrode, respectively, and those at the y-z plane perpendicular to lamination were 224.5 ± 38.0 and 147.6 ± 33.0MPa in the directions parallel and perpendicular to the internal electrode, respectively, After heat treatment, the stress was changed from tension to compression at the y-z plane perpendicular to lamination. Also, the external electrode affected the formation of residual stress (e.g., -39.9 ± 24.5 and 31.1 ± 12.0 MPa with and without the external electrode, respectively, in the direction perpendicular to the internal electrode at the y-z plane parallel to lamination). The relationship between residual stress and heat treatment parameter was discussed extensively.

U11.16
A Theoretical Study on the Indentation of Viscoelastic Materials. Guanghui Fu, Lam Research Corporation, Fremont, California.

Nanoindentation experiments have become a commonly used technique to investigate mechanical properties of polymeric thin films. However, there is a lack of fundamental theoretical studies in this field. In this paper, the problem is modeled as normal indentation of a viscoelastic half-space by a rigid smooth frictionless axisymmetric indenter. An analytical solution, which relates the indentation load and the penetration depth, is presented. The solution is valid if the indenter is in complete contact with the half-space and the contact area is simply connected. The load-displacement relationship is given in Laplace space. Inverse Laplace transform is needed in order to obtain the time-dependent load-displacement relations. Complicated loading histories (either load-controlled or displacement-controlled) can be handled by this method. Further derivation shows a similar fundamental relation among contact stiffness, contact area and Young's modulus in Laplace space. For a viscoelastic material with a fixed Poisson's ratio, constant-load indentation tests will give the material's creep function, and constant-displacement tests will give its stress relaxation function. As examples of the presented solution, explicit analytical solutions of these two cases are derived. The presented solution may be used to interpret nanoindentation experimental data of viscoelastic materials, such as polymeric thin films. It also gives new ways of measuring viscoelastic properties of thin films. Suggestions for further fundamental studies of nanoindentation problems are given at the end.

U11.17
Scaling Relationships for Analyzing Instrumented Spherical Indentation Experiments. Wangyang Ni[1,3], Yang-Tse Cheng[1], Che-Min Cheng[2] and David S Grummon[3]; [1]Materials and Processes Lab, General Motors Research and Development Center, Warren, Michigan; [2]Institute of Mechanics, Chinese Academy of Sciences, Beijing, China; [3]Department of Chemical Engineering and Materials Science, Michigan State University, East Lansing, Michigan.

Using dimensional analysis and finite element calculation, we studied spherical indentation in elastic-plastic solids with work hardening. We report two previously unknown relationships between hardness, reduced modulus, indentation depth, indenter radius, and work of indentation. These relationships, together with the relationship between initial unloading stiffness and reduced modulus, provide an energy-based method for determining contact area, reduced modulus, and hardness of materials from instrumented spherical indentation measurements. This method also provides a way of calibrating the effective radius of imperfectly shaped spherical indenters. Finally, the method is used to probe the "indentation size effect" in several materials, including copper, aluminum, and tungsten.

U11.18
Indentation-induced deformation of SiGe thin films.
Jodie E Bradby[1], James S Williams[1], Michael V Swain[2] and Paul Munroe[3]; [1]Electronic Materials Engineering, The Australian National University, Canberra, Australian Capital Territory, Australia; [2]Mechanical and Mechatronic Engineering, The University of Sydney, Everleigh, New South Wales, Australia; [3]Electron Micrscopy Unit, The University of Sydney, Sydney, New South Wales, Australia.

It is well known that Si undergoes a series of phase transformations during mechanical deformation whilst Ge, a structurally-similar Group IV semiconductor, deforms by twinning and the formation of slip bands. Thus, the deformation behavior of SiGe alloys of different compositions is a topic of interest. In this study, the indentation-induced deformation of SiGe thin films, grown epitaxially on Si, is examined by nanoindentation, atomic force microscopy and cross-sectional transmission electron microscopy (XTEM). A number of epitaxial films were examined containing Ge compositions ranging from ~10-50 atomic %. Results show that at Ge concentrations of only 9%, the deformation behavior of SiGe is quite different compared to that of pure Si. From previous studies it has been shown that diamond-cubic Si-I transforms to a metallic Si-II phase on loading (at a pressure of ~11 GPa). On pressure release, Si-II undergoes further transformations to form either an amorphous phase or a mixture of the high-pressure phases Si-III and Si-XII depending on the rate of pressure release. Fast pressure release favors the formation of the amorphous phase, while slow unloading leads to the nucleation of polycrystalline Si-III and/or Si-XII. XTEM images of SiGe films containing 9% Ge after spherical indentation reveals that, similar to Si, a pressure-induced phase transformation has occurred in the thin SiGe film. From selected-area diffration patterns it was found that the remnant phases in the underlying Si substrate were Si-III/Si-XII. In contrast, the phases observed in the SiGe films after indentation was purely amorphous. Changes in the deformation behavior were studied as a function of Ge content. The authors would like to thank Prof R. Elliman for the SiGe samples used in this study.

U11.19
Effects of Thickness and Tip geometry in Nanoindentation of Nickel thin films. Padma Lakshmi Parakala[1], Reza Mirshams[1], Seifollah Nasrazadani[1] and Kun Lian[2]; [1]Engineering Technology, University of North Texas, Denton, Texas; [2]Center for Advanced Microstructure and Devices (CAMD), Baton Rouge, Louisiana.

Effects of thickness and tip geometry on Ni thin films deposited on Cu were studied using nanoindenter and Atomic Force Microscopy (AFM). The deformation mechanisms in correlation to hardness measurements were determined at various loads and depths of penetration. Berkovich, cube corner and conical tips have been used in this study. Initially the hardness and modulus of elasticity were measured at a depth of 10% of film thickness. Gradually the depth of penetration was increased to 20% to observe the variations in mechanical properties. The hardness and modulus of elasticity were calculated by Olive and Pharr method. Analyst software is used to analyze results. The penetration effects were examined with Scanning electron microscopy (SEM) and AFM.

U11.20
Microstructural Evolutions in Ru-Al Multifilms at High Temperatures. Kaiwen Liu and Frank Muecklich; Materials Science-Functional Materials, Saarland University, Saarbruecken, Germany.

B2 structured RuAl is considered to be a promising high-temperature materials with excellent high-temperature corrosion and oxidation resistance and good room-temperature toughness. In this work, single B2-structured RuAl thin films are prepared from corresponding elemental layers by isothermal annealing at various high temperatures. The elemental Ru and Al films with different thickness are prepared by ion-gun sputtering. The phase formation evolution, i.e. grain growth, stress and texture are investigated by X-ray diffraction (XRD), Thermal Analysis (DSC) and Transmission Electron Microscopy (TEM).

U11.21
The Thermo-tribological Behaviour of Nano-structured Hard PVD Coatings. Ghassan Nayal, surface Engineering, Materials Research Institute, P O Box 793, Aleppo, Syria.

The isothermal tribological behaviour of nano-structured hard PVD sputtered coatings (TiAlN/VN, TiAlYN/VN, TiAlN/CrVN, TiAlN/CrN, and Cr/C)was characterised using a heated thermo-tribometer, TGA, SEM, TEM, X-RD, and other dedicated characterising techniques. Tests were conducted in a range of temperatures (25-700C) using a media of atmospheric air. Within the lower temperature range, the sliding wear rates for the V and C containing coatings (TiAlN/VN, TiAlYN/VN and Cr/C) were found to be significantly lower than the corresponding values for the Cr containing coatings (TiAlN/CrVN, TiAlN/CrN). This was attributed to the reduced levels of friction between the former coatings and the counterpart material (Al2O3). Conversely, the sliding wear rates of the V and C containing coatings within the higher temperature range were significantly higher than the corresponding values for the Cr containing coatings. This was found to be linked to the oxidation kinetics as well as the nature and properties of the oxidation product of individual coating materials investigated. Within the higher temperature range, both V and C containing coatings revealed stronger tendencies for oxidation. Additionally, the oxidation product of the latter coatings was characterised as being less protective with reduced levels of thermal stability relative to the oxidation product of the Cr containing coatings. Finally, a correlation between the thermal stability of the coating material and the evaluated mechanical behaviour, which evolved with increasing temperature, was noted. This constitutes a further useful tool in the prediction of the high temperature tribological performance of nano-structured hard PVD sputtered coatings.

U11.22
Nanostructured Reactive Foils Used for the Joining of Titanium. Alan Duckham[1], Etienne Besnoin[2], Stephen J Spey[1], Jiaping Wang[1], Omar M Knio[2] and Timothy P Weihs[1]; [1]Materials Science and Engineering, The Johns Hopkins University, Baltimore, Maryland; [2]Mechanical Engineering, The Johns Hopkins University, Baltimore, Maryland.

Freestanding nanostructured multilayered foils are being used to develop a novel joining technique. These foils are capable of undergoing a self-sustaining exothermic reaction that results in a rapid increase and decrease in temperature and an accompanying small intense burst of heat. The heat released can be harnessed to induce melting of adjacent solder or braze layers to effect bonding of various components. The advantages of using this method of joining over conventional joining techniques are many-fold. Most notably: the need for a furnace is eliminated; it can be performed in any atmosphere; and there is no significant heating of the components. To date, various materials have been joined successfully using reactive foils and solders. More recent attempts include the use of braze layers that have significantly higher melting temperatures compared to solders. Ensuring a sufficiently rapid transfer of heat from the reactive foils into the braze layers is therefore critical. In this study, factors influencing kinetic heat transfer have been studied systematically by investigating the joining of titanium to titanium using a silver based braze and freestanding sputter deposited Al/Ni multilayered foils containing thousands of nanoscale layers. Tensile shear strengths of joints have been measured, and microscopy on both fracture surfaces and cross-sectioned untested joints has been conducted. The heat transfer during the reactive joining process has also been modeled numerically. Based on a combination of experimental and numerical results it is clear that the maximum temperature reached by the reacting foil is critical in determining the amount and the duration of melting of braze layers. It is known that that the maximum temperature reached by the foil is largely determined by the specific heat for the foil's reaction. By careful control of the structure of the multilayer foils we are able to control the heat of reaction (which can be measured by calorimetry) and hence ensure successful joining using brazes.

U11.23
Thermo-mechanical behaviour of Cu, Fe thin films and Cu/Fe multilayers. Nail R Chamsoutdinov and Amarante J Bottger; Materials Science, TU Delft, Faculty of Applied Science, Delft, Netherlands.

The thermo-mechanical behaviour of magnetron sputtered copper and iron polycrystalline films of thicknesses between 50 nm and 1 micron have been investigated. The state of stress has been determined by means of wafer curvature and X-ray diffraction (sin2y-method), both methods are in good agreement for layers of thicknesses above 200 nm. For specimens of smaller layer thicknesses, however, the average stresses as measured by X-ray diffraction are systematically higher than those observed by wafer curvature experiments. The results can be interpreted in terms of differences in microstrain/dislocation densities and grain size. The latter were obtained by X-ray peak profile analysis. The grain sizes were compared with those obtained by transmission electron microscopy. Thermal cycling experiments were performed between RT and 773 K. The yield strengths are discussed in the framework of constrained dislocation motion in thin films.

U11.24
Deposition and Tribological Property of Magnetron Sputtered TiN- MoS2 Composite Coating with Ti-TiN Graded Interlayer. Md. Mahfujur Rahman, NCPST and MPRC, Dublin City University, Dublin, Ireland.

In order to provide optimum wear protection for sliding surfaces, it is necessary to use a better solid-lubricant coating with low friction combined with high hardness, the ability of formation of a low friction transfer film on the opposing surfaces and good adhesion combined with mechanical properties which leads to very high load bearing capacity. In this work, several Ti-TiN-(TiN-MoS2) graded composite thin coatings were deposited on steel substrate by Close Field Unbalanced Magnetron sputtering (CFUBMS) from Ti and MoS2 targets to combine all this properties. The microhardness and adhesion strength of the deposited coating as a function of thickness and composition of top composite layer were investigated by microhardness measurements and scratch testing respectively. These properties were correlated with the tribological properties of this coating, which was done by Pin-On-Disk test. Further film properties were investigated by scanning electron microscopy (SEM) and X-ray diffraction (XRD).

U11.25
Abstract Withdrawn

U11.26
Investigation of Thermo-mechanical behavior of multi-layer thin-film structures for MOEMS and Power MEMS applications. Zhiqiang Cao[1,2], Biao Li[2] and Xin Zhang[1,2]; [1]Department of Manufacturing Engineering, Boston University, Boston, Massachusetts; [2]Fraunhofer USA Center for Manufacturing Innovation, Boston, Massachusetts.

Recent, rapid developments in MOEMS (micro-opto-electro-mechanical systems) and Power MEMS (micro-scale heat engines, coolers, fuel cell chips, and related components) frequently not only require higher electrical and mechanical endurance of the devices (such as for a micro generator), but also more precise control of shapes of the structures (such as for a micro mirror array and an IR micro bolometer). Layer-by-layer deposition of thin films is one of the fundamental steps in the fabrication of these complicated devices. As a result of performance requirements, such thin film structures need to be either very flat (for MOEMS), very thick (for Power MEMS) or both (for process integration purposes). Stress modes in fabrication of such demanding structures are not well understood. However, residual stresses in such multi-layer thin film structures during and after deposition process often cause fracture, bulking and delamination, etc, rendering these devices useless. Stress-related problems during multi-layer thin film deposition are many of the most important failure mechanisms of MOEMS and Power MEMS devices, and they need to be better understood before guided novel engineering solutions can be proposed. As a starting point, this paper adopts thermal cycling and annealing as a simulation tool for MEMS fabrication and discusses thermo-mechanical behavior of multi-layer thin films during and after thermal deposition. Thermo-mechanical behaviors were investigated for various types of multi-layer thin film structures usable in several MOEMS and Power MEMS applications (micro generators, micro mirror arrays, and IR micro bolometers). The experimental results were then analyzed to elucidate the control mechanisms of intrinsic stress generation. Furthermore, stresses modes were modeled to provide insights for the guidance of the development of engineering solutions to improve manufacturability, planarity, and reliability of MEMS devices.

U11.27
Thermomechanical Behavior and Phase Transformation in Tantalum Thin Films. Robert Knepper and Shefford P Baker; Materials Science and Engineering, Cornell University, Ithaca, New York.

Tantalum thin films are used in a number of microelectronic and x-ray optics applications. Stresses that build up in these films during thermal cycling can cause reliability problems in devices. Sputter-deposited tantalum thin films are often prepared so as to have a metastable tetragonal crystal structure (β phase). Such films may undergo changes in stress on the order of several GPa in the tensile direction if the film transforms to the stable bcc structure (α phase) during thermal cycling. Changes on the order of GPa in the compressive direction may arise from oxygen incorporation into the film at elevated temperatures. We prepared β-Ta films in an ultra-high vacuum sputter deposition system and measured the stresses that arose during thermal cycles to 750° C using an in-situ substrate curvature stress measurement system. The oxygen content in the film was controlled during both deposition and thermomechanical testing. In films with essentially no oxygen contamination, phase transformation from β to α takes place in distinct "jumps." Addition of controlled amounts of oxygen to the film inhibits the phase transformation, causing an increase in transformation temperature as well as altering the magnitudes of the "jumps" in stress. The stresses in Ta films are thus found to be very sensitive to oxygen content as well as thermal history.

U11.28
Thin Film Material Parameters Derived from Full Field Nanometric Displacement Measurements in Non-uniform MEMS Geometries. Jaime Cardenas-Garcia[1], Ioannis Chasiotis[2] and Sungwoo Cho[2]; [1]Mechanical Engineering, University of Maryland, College Park, Maryland; [2]Mechanical and Aerospace Engineering, University of Virginia, Charlottesville, Virginia.

MEMS-scale polycrystalline silicon thin film specimens fabricated via the Multi User MEMS Processes (MUMPs) have been employed to obtain the non-uniform nanometric displacement fields in the vicinity of prefabricated circular and elliptical micron-sized perforations [1]. For the hole diameter-to-specimen width ratios considered in this work, and for all practical purposes, the displacement solution for a hole in an infinite plate was applicable. This method requires the ability to reliably and repeatably acquire nanometer level displacements from freestanding thin films. These tensile tests were conducted by a custom microtensile tester with the aid of an Atomic Force Microscope (AFM) and a special gripper that makes use of electrostatically assisted UV adhesion to handle and load miniature MEMS specimens [2]. This non-conventional procedure for material parameters determination relies on Digital Image Correlation (DIC) to compare two AFM images [3], one before and one after specimen loading, and thus compute the nanometer-level displacement fields (<50 nm global displacements) in 15x15 um (or smaller) areas, with better than 2-3 nm resolution in displacements. By posing and solving a non-linear least squares inverse problem, where, for known applied loads and measured displacement fields in an infinite plate with either a circular [4] or an elliptical hole, it is possible to recover the elastic modulus (E) and Poisson's ratio (n) of the material. The main advantage of this approach is the full utilization of high-resolution displacement measurements over a small specimen area, using only measurements acquired at two load levels. Further statistical measurements of material properties may be obtained at varying load amplitudes. [1] I. Chasiotis, W.G. Knauss, "The Mechanical Strength of Polysilicon Films: 2. Size Effects Associated with Elliptical and Circular Perforations", J. of the Mechanics and Physics of Solids 51 (8), pp. 1551-1572, (2003). [2] I. Chasiotis, W.G. Knauss, "A New Microtensile Tester for the Study of MEMS Materials with the aid of Atomic Force Microscopy", Experimental Mechanics 42 (1), pp. 51-57, (2002). [3] I. Chasiotis, W.G. Knauss, "Microtensile Tests with the Aid of Probe Microscopy for the Study of MEMS Materials", Proceedings of SPIE 4175, pp. 96-103, Santa Clara, CA (2000). [4] J. F. Cardenas-Garcia, S. Ekwaro-Osire and J. M. Berg, "Solution to the moire hole method problem", Mechanics Research Communications 28 (1), pp. 13-32, (2001).

U11.29
Stress Temperature Dependence of Thin Films Based on Wafer Curvature Method. Mircea Capanu[1], Andrew Cervin-Lawry[1], Atin Patel[1], Ivo Koutsaroff[1], Paul Woo[1], Lynda Wu[1], James Oh[1], Jake Obeng[1], Brett McClelland[1] and Vladislav Domnich[2]; [1]Technology R&D, Gennum Corporation, Burlington, Ontario, Canada; [2]Materials Engineering Department, Drexel University, Philadelphia, Pennsylvania.

Thin film properties such as the thermal expansion coefficient (TEC), the biaxial modulus, Young modulus, the Poisson ratio and the temperature coefficient of Young modulus are important to know for the modeling, design, manufacturing and testing of micromachined devices, especially for MEMS. When the stress-temperature dependence of a thin film is measured on two different substrates that gives TEC and elastic constants of that film if no irreversible change occurs in the film during heating and cooling. The stresses can be measured using Stoney equation. This method is nondestructive and does not require any further processing, any special equipment or any technical skills, making it very attractive, especially for industry. The biaxial modulus and TEC were measured by other researchers for the following films: BN, a:Si:H, a-C:H, a-GeCx:H, Ag, Al, Al-Cu, Cu, SiO2. In the present work, measurements not only of the Biaxial modulus and TEC, but also their temperature coefficients for various thin films, such as PolySi (mainly used in MEMS), low stress LPCVD SixNy, (Ba,Sr)TiO3 (BST), Pt, Au/TiWN are determined. For a thin film that requires an adhesion layer, a bimorph measurement model has been developed in order to distinguish the properties of each of the two thin films. For example, for BST thin film on various substrates, an adhesion layer of TiOx for the Pt base layer is required. In the case of Au thin film, the adhesion layer was TiWN. In order to measure the temperature coefficient of the biaxial modulus, the corresponding coefficients for the substrates were needed. To improve the precision, perfect circular and thin wafers are used (no reference flat): 4 inches of 280um thick <111>Si and 450um thick fused silica. The measurements were performed using a commercial Tencor FLX3220 Stress Measurement System. The temperature range is from room temperature to 200-300 oC. In the case of BST films, the measured thermomechanical properties have been correlated with the dielectric properties (C-V, I-V, capacitance density, dissipation factor) for films processed on equivalent substrates under identical processing conditions.

U11.30
Damage and Wear of the Femoral Head Surface Layers: Nano-Scale Morphology. Volf Leshchinsky[1], Ya M Soifer[2], A. Verdyan[2] and Hanna Weinert[1]; [1]Powder Metallurgy, Metal Forming Institute, Poznan, Poland; [2]Depatrment of Sciences, Holon Academic Institute of Technology, Holon, Israel.

For a femoral head articulating against polyethylene, wear of softer polyethylene is known to be caused by the hard asperities on the surface of the femoral head, that destroy polyethylene surface and detach polyethylene particles. In the present work in order to better understand the mechanism of femoral head prosthesis wear process we studied the femoral head surface morphology after implant articulation and modeled the damage of the femoral head surface by nanoindentation and nanoscratching with Atomic Force Technique. The Atomic Force Modeling of scratching process revealed an intensive surface damage cobalt-chrome and alumina femoral head surface. The nano-scale surface damage of femoral heads is shown to result in head material particles generation that may be one of the main reasons of polyethylene wear at least at the first stage of hip implant articulation .

U11.31
Characterization of Dynamical Mechanical Properties of Pressure-Sensitive Adhesive (PSA) Thin Film and Bone Cells. Patricia M McGuiggan[2], Yan Deng[1], Brian R Lawn[1] and Sheldon M Wiederhorn[1]; [1]Materials Sceince and Engneering Lab, NIST, Gaithersburg, Maryland; [2]Polymer division, NIST, Gaithersburg, Maryland.

An atomic force microscopy (AFM) is used to study the dynamical mechanical response of pressure-sensitive adhesive (PSA) thin film. The AFM tip was a modified spherical metal ball with radius 30 mum glued to the cantilever to produce a low-stress surface contact. In this experiment, the amplitude and phase of a cantilever resting on an oscillating film is monitored as a function of frequency over the range of 10-2 Hz to 103 Hz. From these measurements, storage moduli and loss moduli were calculated for the PSA films. Frequency-dependent adhesion forces and indentation forces were also measured with the AFM spherical probe. This dynamical testing protocol was used to assess attachment forces between MC3T3-E1 bone cells and synthetic bone substrates of calcium phosphate cement (CPC) under different frequency conditions. Correlations between the cell attachment forces and CPC substrate biocompatibility were investigated.

U11.32
Characterizing Thin-Film Stress Fields by Resonance of Membrane Arrays. Roxann Louise Engelstad, Edward George Lovell and Michael F. Cash; Mechanical Engineering, Univ. of Wisconsin - Madison, Madison, Wisconsin.

Controlling thin-film stress magnitudes and nonuniformities is a persistent and pervasive challenge for applications ranging from the semiconductor industry to nanotechnology. Consequently, the ability to accurately measure the intensity and spatial distribution of film stress is essential for fabrication process optimization. Induced substrate curvature is often used to estimate film stress, but is inaccurate except for uniform distributions. Alternatively, average stress in a freestanding membrane can be accurately determined from measurements of its fundamental frequency of vibration. This paper describes a novel extension of the membrane resonance method to characterize global film stress fields. Thin films are first deposited on silicon substrates. Back-etching is then used to create a large array of membrane windows which are sequentially resonated with a compatible drive electrode. The automated process is carried out in a dedicated vacuum chamber (to eliminate squeeze-film and added-mass effects) and uses a scanning laser vibrometer to identify the natural frequencies and mode shapes. Frequencies are converted into membrane stress, window by window, providing a discrete assessment of the in-plane film stress gradient. A procedure will also be presented for identifying principal stresses in the each membrane, i.e., at an array of locations in the original film. In addition to diagnostic analysis, the automated membrane resonance method was used to determine stress uniformity across Electron Projection Lithography masks, which incorporate membrane window arrays. Masks were fabricated from boron-doped silicon wafers with membrane windows that were 1.0 mm square and 2.0 microns thick. (A typical 200-mm mask has over 8,500 membranes.) Resonant testing of individual windows identified stress gradients within the pattern area. In this paper, it will be shown that such results are essential to facilitate optimization of the mask fabrication process to reduce membrane prestress magnitudes and nonuniformities in order to minimize image placement errors on the device wafer.

U11.33
Thermal Stresses in Optical Films and Their Effects on Optical Performance. Min Huang, Princeton University, Princeton, New Jersey.

Stresses can be induced in the optical films due to thermal mismatch. Because of the elasto-optic effect, these stresses can induce anisotropic and inhomogenous distribution of refractive index in the optical waveguides, which could effect the optical performance. In this paper, the close form solutions are obtained to estimate the thermal stresses in the cladding layers and the core of an embedded channel wavguide.

And the effects of the stresses on the polarization dependence are discussed. Several methods to tune the optical performance by applying the thermal stresses are pointed out.

U11.34
Measurement of Thin-Film Stress, Stiffness, and Strength Using an Enhanced Membrane Pressure-Bulge Technique.
Aaron J. Chalekian, Roxann L. Engelstad, Edward G. Lovell and Phillip L. Reu; Mechanical Engineering, University of Wisconsin, Madison, Wisconsin.

Accurate mechanical properties of thin films are essential for viable design and fabrication applications as diverse as semiconductor devices and microelectromechanical systems. Relevant properties of thin films such as intrinsic stress, fracture strength, and biaxial modulus can be significantly different than the corresponding bulk values, but are more difficult to measure. However, such data can be obtained from the pressure-deflection response of clamped freestanding membranes, i.e., the so-called pressure-bulge test. Experimental challenges include leakage prevention, ensuring proper structural boundary conditions, and accurately measuring applied pressure and transverse displacements of the membranes. In addition, with previously-developed pressure-bulge instruments which rely on vacuum pumping, loadings have been limited to one atmosphere, which is inadequate for burst testing of high-strength films. Consequently, an enhanced pressure-bulge tool has been developed and will be described in this paper. It incorporates positive pressure to overcome the one-atmosphere load limitation, improved edge constraints, and the ability to test arrays of membrane windows. In-plane gradients of mechanical properties are assessed by first depositing the film on a silicon wafer which is then back-etched to create a diagnostic array of freestanding membranes. Membrane deflection from pressure is accurately mapped across the entire window with a scanning laser vibrometer. The load-deflection response provides the biaxial modulus and prestress, window by window, to characterize gradients in these film properties. Likewise, by identifying burst pressure for each window, fracture strength is determined using correlations with analytical and finite element modeling. This technique was directly applied to the analysis of electron projection lithography masks, which are designed and fabricated as a wafer grillage supporting an array of freestanding silicon membranes. Typical membranes are 1.0 to 4.0 mm square, with a thickness of 2.0 microns. This paper will illustrate how the pressure bulge test is used to identify in-plane stress gradients of the silicon film, as well as the burst strength.

U11.35
Thermal-Mechanical Evaluation of Plated Electro-Magnetic NiFe for MEMS Generators.
Yibin Xue[1], Keithan Hillman[1] and David Veazie[1]; [1]Engineering, Clark Atlanta Univ., Norcross, Georgia; [2]Electrical Engineering, Georgia Tech, Altanta, Georgia.

The Army concept, the Objective Force Warrior, requires compact energy sources that provide energy and power density that are at least ten times higher than the best batteries of today can offer. A power microelectronic mechanical system is being developed to meet this challenge. This power MEMS device is intended to be a combination of a micro turbine engine powered by the combustion of hydrocarbon fuel and a coupled micromagnetic generator. This conceptual MEMS generator is to be compact, light in weight, and generating high-density power, which would constitute a good portable power unit for solders. The proposed device comprises a stack of at least five silicon wafers thus requires stringent geometric confinement and operating accurately under the specified opertation conditions. Therefore, an accurate thermal-mechanical evaluation of the electric plated magnetic film, NiFe (60/40), is vital for the reliability of the MEMS generator. NiFe films of 10~30 micron in thickness were electroplated onto glass slices with a sputtered 100 nm Ti seed layer and a 1000 nm thick Cu as electrode. The NiFe films were profiled using a laser blade into dog-bone shape tensile specimens. The freestanding films were then annealed at different temperatures (500 or 700 degrees Celsius in various environments based on the needs for joining the Si wafers). The mechanical properties of NiFe were tested at temperatures from 25 degrees Celsius to 300 degrees Celsius. The NiFe film as-deposited exhibits pure elastic deformation and brittle fracture at room temperature; but NiFe film after subjected to annealing, especially those annealed at 700 degrees Celsius, exhibits stain hardening and ductile fracture , which are shown in Table 1. In addition, the mechanical properties of bulk NiFe alloy of the similar Ni and Fe contents were tested and listed in Table 1. Microstructures and crystalline structures of the plated NiFe were also studied along with the various conditioning, which will be correlated to the changes in thermal-mechanical properties. SEM was used to observe the changes in microstructures (including changes in grain sizes) between as-deposited and annealed NiFe films. XRD was utilized to study the crystalline structure and residual stresses in plated metal films. The changes in surface morphology were characterized by AFM. In addition, the CTE of the plated NiFe was measured using TMA. It is our hope that a constitutive law will be developed for the NiFe films along with the annealing conditions.

U11.36
Compositional Stress and Diffusion in Titania Films.
Sidharth Bhatia, Eric Cohen and Brian W. Sheldon; Division of Engineering, Brown University, Providence, Rhode Island.

Certain transition metal oxides are known to exhibit large compositional changes in response to variations in the oxygen partial pressure. In thin film form, these composition changes can lead to stress because of the constraint imposed by the underlying substrate. To investigate these effects, a system was built to accurately monitor substrate curvature at elevated temperatures, in controlled atmospheres. Polycrystalline TiO2 films were then produced by metal organic chemical vapor deposition. The resulting curvature measurements show substantial differences in the behavior of anatase and rutile films, which appear to be caused by differences in their defect chemistry. By monitoring the stress changes in situ, it was also possible to obtain diffusion data. Films with different grain sizes were studied to investigate the role of grain boundary diffusion.

U11.37
The Effects of Passivation Layer and Film Thickness on The Mechanical Behavior of Freestanding Electroplated Cu Thin Films with Constant Microstructure.
Yong Xiang and Joost J. Vlassak; Division of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts.

The goal of this paper is to investigate the effects of film thickness and the presence of a passivation layer on the mechanical behavior of electroplated Cu thin films. Both dislocation dynamics and strain-gradient plasticity models suggest that these factors play important roles in thin film plasticity. In order to study the effect of passivating layers, freestanding Cu membranes were prepared using standard silicon micromachining techniques. Some of these Cu membranes were passivated by sputter depositing thin Ti films with thicknesses ranging from 20 nm to 50 nm on both sides of the membrane. The effect of film thickness was evaluated by preparing freestanding films with varying thickness but constant microstructure. To that effect, coatings of a given thickness were first vacuum annealed at elevated temperature to stabilize the microstructure. The annealed films were subsequently thinned to various thicknesses by means of chemical mechanical planarization (CMP) and freestanding membranes were prepared both with and without Ti passivation. The stress-strain curves of the freestanding Cu films was evaluated using the bulge test technique. The grain structure and crystallographic texture of the Cu films were determined by means of electron back scattered diffraction (EBSD), the dislocation structure by means of transmission electron microscopy (TEM). Yield stress, Young's modulus, residual stress, and work hardening behavior of the films were measured and correlated with film microstructure and thickness.

U11.38
Analysis of Pop-in Behavior During Nanoindentation.
Hongbin Bei[1,2], Easo P George[2,1], Jenny L Hay[3] and George M Pharr[1,2]; [1]The University of Tennessee, Knoxville, Tennessee; [2]Oak Ridge National Laboratory, Oak Ridge, Tennessee; [3]MTS Systems Corporation, Oak Ridge, Tennessee.

Sudden displacement excursions (also known as pop-in behavior) have been observed during nanoindentation of many materials. Experiments and simulations show that pop-in in a single crystal is most likely caused by dislocation nucleation (incipient plasticity). In most studies, the geometry of the Berkovich indenter is simplified as a sphere. In this study, the precise shape of the Berkovich indenter is analyzed by carefully measuring the area function, and the influence of the shape on the maximum shear stress at the pop-in event is evaluated. Using the precise indenter shape, the measured load-displacement curve obtained by indenting a Cr_3Si single crystal can be well reproduced by assuming a perfectly elastic relationship before pop-in. Furthermore, the stress distribution in the specimen at the pop-in load can be simulated by 2D finite element analysis. Results show that the difference between the maximum shear stress resolved along the slip systems of the crystal and the theoretical shear strength ($G/2\pi$) are within 10%, indicating that the pop-in behavior is caused by dislocation nucleation at the theoretical strength of the material.

U11.39
Grain Size and Thickness Effects on Mechanical Behavior of Freestanding Aluminum Thin Films.
Paul A. El-Deiry, Nicholas Barbosa and Richard P. Vinci; Materials Science and Engineering, Lehigh University, Bethlehem, Pennsylvania.

It is known that the yield stress of a thin film increases with a reduction of grain size and thickness. The independent functions of grain size and thickness, however, are not well known. Because grain size scales with film thickness for typical physical vapor deposition methods, mutual separation of the two variables is not a straightforward task. Additionally, it is known that the presence of a substrate affects the mechanical behavior of a thin film. In this study, grain size and thickness effects are mutually separated using an anodizing and etchback technique. Furthermore, the films are freestanding. Al thin film microbeams 600μm long and 100μm wide have been tested using a custom-built microtensile system. Film thickness ranges from 0.25μm to 1.0μm, and grain size ranges from 0.25μm to 1.25μm. Monotonic stress-strain, stress relaxation, and fatigue tests have been performed. Strain rate effects on mechanical behavior have also been observed.

U11.40

Major Advancements in Laser-Acoustic Analysis of Thin Film Properties. Ronald Berry and Richard P. Riegert; Quad Group, Spokane, Washington.

A laser can be used to induce acoustic surface waves that can be used within minutes to non-destructively (and simultaneously) determine thin film thickness (to the nanometer range), hardness, elastic modulus, Poison's ration, and the ability to detect delamination problems. The fundamentals of this process are deceptively simple, apply an energy source (the laser) to induce vibrational quantum mechanical transitions across a thin film (and substrate). However until recently, the application of this technology has too complicated and costly for most users. Most laser-acoustic systems use a low-power ultraviolet laser to induce an instantaneous thermal expansion in a thin film. As this surface wave is transmitted through the film, there will be selective frequency losses from the wave directly related to the material properties of the film. The useful acoustic output is in the ultrasonic range of 50-250 MHz. There are two basic types of information that can be determined; systemic and asystemic. Systemic information includes thickness, hardness, elastic modulus and Poison's ratio. Asystemic information is detection of impurities in the thin film and/or substrates, crystalline orientation, and adherence/coherence defects. While this method is not only is it non-destructive and able to quickly measure a number of important physical parameters simultaneously, is that it measures the physical properties of the top-most two thin film stack layers (or thin film and substrate layers if not a stack coating). It can also be used as a before and after snapshot of surface modifications, thin film degradations, and wear. We will demonstrate the fundamental principles of laser-acoustic spectrometry and the recent advances in both understanding and application this phenomenon. Also to be discussed is its potential use in a number of industrial and academic settings, as an NDT tool for research and development and quality control for thin film processes.

U11.41

Faceted Dislocation Surface Pits. Danxu Du[1,2] and David J Srolovitz[1,2]; [1]Dept. of MAE, Princeton University, Princeton, New Jersey; [2]Princeton Materials Institute, Princeton, New Jersey.

Surface pits are commonly formed where threading dislocations emerge from a thin film. Observations of facetted pits are common in complex materials where the Burgers vector tends to be large. Such materials also commonly exhibit hollow core dislocations/micropipes. In this presentation, we analyze the shapes of surface pits in anisotropic materials. The analysis, based upon variational principles, balances the elastic strain energy agains the anisotropic surface energy to determine equilibrium pit shapes. New analytical results will be presented for the isotropic surface energy case that extend the classical analyses. Numerical results will then be presented for cases in which the surface energy is highly anisotropic (i.e., where there are cusps in the surface energy vs. surface orientation plot). We show that faceting is dominated by the cusps in the surface energy, while the elastic energy associated with the dislocation dominates largely controls the size of the pits. In cases in which the cusps in the surface energy are sufficiently large that the surface is completely faceted, analytical predictions for the pit size are made. In such cases, two types of solutions are possible; namely, flat surfaces with no pits and facetted pits. Application of this method to interpret observations of dislocation pits in GaN and InGaN/GaN superlattices will be presented.

U11.42

The Thermal Annealing Effect On The Residual Stress And Interface Adhesion In The Compressive Stressed DLC Film. Heon Woong Choi[1], Myoung-Woon Moon[1], Tae-Young Kim[1,2], Kyang-Ryel Lee[2] and Kyu Hwan Oh[1]; [1]School of material science engineering, Seoul National University, Seoul, South Korea; [2]KIST, Seoul, South Korea.

We studied on the thermal annealing effect on the residual stress and the mechanical properties in thin compressive stressed diamond-like carbon film on Si substrate. Annealing experiments were carried out with Rapid Thermal Procedure system at 200-600 degree of celcius, and the stress change with annealing temperature was investigated by in-situ stress measurement system. The apparent stress reduction occurred with minimal structure changes. In order to measure the change of chemical structure of diamond-like carbon film by annealing, we used Raman spectrometer. The adhesion deterioration in interface has been detected as annealing temperature increased. In the compressive stressed DLC film, we observed the dramatic evolution of interface delamination at certain high temperature using in-situ heating stage built in Environment SEM (FEI XL-30 FEG / PHILIPS). The quantitative change of adhesion affected by annealing process was also measured with scratch testing. For exploring the interface structure affected by the thermal annealing process at high temperature, the cross section of the annealed film has been observed with HR TEM.

U11.43

Effect of Monazite Coating on Tensile Properties of Nextel 720 Fibers, Tows and Minicomposites. Devdas M. Pai[1], Sergey Yarmolenko[1], Jagannathan Sankar[1], Balasubramanian Kailasshankar[1], Christopher L. Murphy[1], Edwardo Freeman[1] and Larry P. Zawada[2]; [1]Mechanical and Chemical Engineering, NC A&T State University, Greensboro, North Carolina; [2]Materials, Ceramics & NDE Division (AFRL/MLLN), Air Force Research Laboratory, Wright-Patterson AFB.

Ceramic Matrix Composites (CMCs) typically consist of ceramic fibers, a matrix and one or more fiber/matrix interfacial coatings (fiber coatings). The fiber coating provides a weak fiber-matrix interface that prevents matrix cracks from penetrating the fibers, thus providing damage tolerance to the composite. Fiber coatings also protect fibers from environmental degradation during composite fabrication and use. Fiber coating thickness is typically $\sim$ 250-500 nm. The fiber coatings must be chemically and mechanically stable in high temperature corrosive environments in order to maintain the fiber coating-matrix debond characteristics necessary for damage tolerance. Carbon and boron nitride (BN) are the traditionally used fiber coating materials; however, they suffer the problems of oxidation in the presence of water and also at intermediate temperatures. This has prompted research into oxide coatings (as a substitute for carbon or BN) that are stable with the matrix and fiber at high temperatures. A leading oxide coating under investigation is monazite, which is stable at high temperatures with many common oxides including alumina. There is also some evidence to suggest that monazite-alumina interphase boundaries deflect cracks. Single fiber tests were performed at room temperature and at 1200 deg. C for both coated and uncoated Nextel 720 (largely mullite) fibers. Also, the effect of thermal soaking of the fibers at elevated temperatures prior to testing was studied, for a total of over 1,500 tests. Weibull failure statistics were developed for the single fiber tests. Single fiber testing was followed by a similar battery of tests on Nextel 720 tows as well as on Nextel 720 minicomposites, made by impregnating individual tows with an alumina matrix. This paper discusses our findings in terms of the effects of the coatings on the tensile behavior.

U11.44

Ti-Si-C MAX-Phase Thin Films. Jens Emmerlich[1], Jens-Petter Palmquist[2], Jon M. Molina-Aldareguia[3], Zsolt Czigany[4], Hans Hoegberg[1], Per Eklund[1], Ulf Jansson[2] and Lars Hultman[1]; [1]Thin Film Physics, Linkoepings University, Linkoeping, Sweden; [2]Materials Chemistry, Uppsala University, Uppsala, Sweden; [3]CEIT, San Sebastian, Spain; [4]Research Institute for Technical Physics and Materials Science of Hungarian Academy of Sciences, Budapest, Hungary.

Ternary $M_{n+1}AX_n$-phases (n = 1, 2, 3; M: early transition metal; A: A-group element; C: Carbon and/or Nitrogen) are new materials that combine ceramic and metallic properties. For example, nanolaminated Ti_3SiC_2 have good resistance against oxidation, low density, high decomposition temperature (> 1800 °C), good conductivity (22 $\mu\Omega$cm) and high ductility. Extensive research has been carried out on polycrystalline bulk materials produced by hot isostatic pressing or self-propagating high-temperature synthesis. We report the successful DC magnetron sputtering from three single-element targets (Ti, Si and C) of single-phase and single-crystal Ti_3SiC_2 thin films on MgO(111) substrates at 900 °C as well as synthesis and epitaxial growth of metastable Ti_4SiC_3 on Al_2O_3(0001) at 1000 °C. For substrate temperatures T_S below 700 °C the MAX-phase nucleation could not be initiated, instead a TiC-like fcc structure was observed through the entire film by XRD and TEM/EDS analysis. Complementary studies with XPS and ERDA measurements suggest Si to be substitutionally incorporated in TiC during co-deposition of Ti, Si and C at $T_S \leq 700$ °C. Depositions at ambient temperature resulted in nanocrystalline growth. A lateral growth mode of Ti_3SiC_2 was observed using AFM and TEM imaging techniques. With low maximum indentation loads a hardness of 15-18 GPa and a Young's modulus of 260 to 280 GPa was obtained. Nanoindentation combined with TEM imaging revealed kink formation and delamination at higher indentation loads resulting in decreased hardness values. Four point probe measurements quantified a low bulk resistivity of 29 $\mu\Omega$cm for Ti_3SiC_2, while the Ti-Si-C films grown without assisting heating, exhibited an increased resistivity value up to 1700 $\mu\Omega$cm.

U11.45
Abstract Withdrawn

U11.46
Interfacial strain and morphology in $YBa_2Cu_3O_{7-x}$/(Ba, Sr)TiO_3 multiple layer thin films. Yichun Luo, Robert Hughes, John S Preston and Gianluigi A Botton; Brockhouse Institute of Materials Research, Mcmaster University, Hamilton, Ontario, Canada.

$YBa_2Cu_3O_{7-x}$ (YBCO) films grown by laser deposition on (100) $LaAlO_3$ (LAO) substrates show a strong thickness dependence of the electrical properties. For example, for film thicknesses above 0.3 micron, the critical current density decreases with increasing thickness of the films. However, nano-composite films consisting of multiple layers of $YBa_2Cu_3O_{7-x}$/(Ba, Sr)TiO_3 (BSTO) thin films having a total thickness of 5 microns show excellent electrical properties. In order to understand these effects, a detailed microstructural characterization has been undertaken. Transmission electron microscopy (TEM) observations show that nanoscale porosity, stacking faults and precipitation of secondary phases are present on the single-layer films while a high-quality microstructure is observed for the nanocomposite multiple-layer films although defects at YBCO/BSTO interface are still present. In addition, in the nanocomposite films the surface roughness is minimized, the overall defects density in each YBCO layer is reduced and the spiral growth in YBCO is modified. In this complex microstructure, the YBCO/BSTO interfaces and the lattice mismatch strain play therefore a crucial role in controlling the nature of the defects, stability of phases and growth mechanisms. In order to understand the role of the BSTO layer and the strain effects on the structure of the films, the interface structure and defects in BSTO layers are analyzed by high-resolution transmission electron microscopy (HRTEM).

SESSION U12: Multilayers and Nanolaminates
Chairs: Sean Corcoran and Young-Chang Joo
Friday Morning, December 5, 2003
Room 210 (Hynes)

8:30 AM *U12.1
Multilayer Foils and Their Use as Rapid Heat Sources for Soldering and Brazing Components. Tim P. Weihs, [1]Reactive NanoTechnologies, Hunt Valley, Maryland; [2]Department of Materials Science and Engineering, Johns Hopkins University, Baltimore, Maryland.

Multilayer foils that react exothermically are a relatively new class of nanostructured materials that can be used to solder or braze components together. By inserting a free-standing foil between two solder (or braze) layers and two components, heat generated by the reaction of the foil melts the solder and consequently bonds the components. The joining process can be completed in approximately one second, in air or in vacuum. The use of reactive foils as a local heat source eliminates the need for torches, furnaces, or lasers, speeds soldering and brazing processes, and dramatically reduces the total heat that is needed. Thus, temperature-sensitive components and metals and ceramics can be joined without thermal damage. The multilayer foils range in thickness from 20 to 200μm, and they contain hundreds of nanoscale layers that alternate between materials with large heats of mixing, such as Al and Ni. By controlling the composition and thickness of the alternating layers, reaction velocities and temperatures can be nanoengineered for a variety of joining applications. In this presentation the self-propagating reactions will be described, and thermal predictions and IR images of the joining process will be presented. Examples of joints between dissimilar materials will be reviewed, along with characterizations of the resulting interfaces: mechanically, thermally, and microstructurally. Finally, applications such as the mounting of heat sinks onto Si dies will be discussed; in this case demonstrating a 10x drop in thermal resistance compared to common thermal interface materials.

9:00 AM U12.2
Role of Mn alloying and concentration modulations in electrodeposited Ni films. Emmanuelle A Marquis[1], Jim J Kelly[1], A Alec Talin[1], Thomas F Kelly[2] and Steve H Goods[1]; [1]Thin Film and Interface Science, Sandia National Laboratories, Livermore, California; [2]Imago Scientific Instruments, Madison, Wisconsin.

High-strength and temperature resistant electrodeposited Ni films for MEMS applications are obtained by incorporation of small amounts of Mn, which raise the recrystallization temperature significantly, contributing to the thermal stability of the alloys. However, Ni-Mn alloys that are plated from Ni-sulfamate baths suffer from high internal stresses, which can cause problems such as delamination of the film from the substrate. In order to limit the film stresses accumulated during deposition, pulse-plating conditions are used to create a material that is compositionally modulated at the nanoscale. The resulting material is believed to consist of regions of high-stress Ni-Mn separated by regions of low-stress Ni. These pulse-plated Ni-Mn films have good mechanical properties and good thermal stability. Characterization of the compositional structure of these materials is challenging because of both the fine spacing of the modulations and the low Mn concentrations. Here, we have employed three-dimensional atom-probe microscopy to measure the concentrations of Mn and other solute atoms. This technique provides chemical information with near-atomic spatial resolution, and is uniquely suited to examine the effects of microalloying and of solute interactions. We find that the Mn concentration is indeed modulated with a spacing of about 5 nm, and that the spatial distributions of Mn atoms and S or O atoms are correlated. Changes of the solute distribution after annealing at high temperature are also presented, and the role of the solute elements on the thermal properties and the strengthening mechanisms operating in these Ni-Mn films are discussed.

9:15 AM U12.3
Stress-field in sputtered Mo Thin Films and Mo/Ni Superlattices : origin and evolution after ion-irradiation. Gregory Abadias, Aurelien Debelle, Anny Michel and Christiane Jaouen; Laboratoire de Metallurgie Physique, UMR 6630, Universite de Poitiers, Futuroscope-Chasseneuil, France.

The physical and especially the mechanical properties of thin films depend strongly on their intrinsic stress state and, in some cases, this stress can reach values near the theoretical shear strength, which may lead to delamination or crack formation. For this reason, the ability to control the stress level is of prime importance, either $in-situ$ during the film growth, or $ex-situ$ during thermal treatment or by post-irradiation with energetic ions. For thin films grown by sputter deposition, large compressive stresses are usually measured at low working pressure. Although numerous studies have shown a direct link between the energetics of the deposition process and the magnitude of stress, with interpretations based on the atomic peening mechanism, a comprehensive stress model is still lacking. The strain-stress state and the stress-free lattice parameters of fibre-textured (110) Mo thin films and epitaxial (110)Mo/(111)Ni superlattices grown by ion beam sputtering have been determined by X-ray diffraction. The evolution of the residual stress has been studied after irradiation with 400 keV Ar ions at low fluences ($\sim 10^{14}$ ions.cm^{-2}). For Mo thin films, the effect of the deposition rate, nature of sputtering gas and film thickness on the intrinsic stress has been systematically investigated on the as-grown samples. A stress model is presented to reproduce the evolutions of the $\sin^2\psi$ plots before and after ion irradiation-induced stress relaxation. It is shown that the initial state of stress is triaxial, with an hydrostatic component arising from the presence of growth-induced point defects. For the Mo/Ni multilayers, it is shown that the state of stress in Mo sublayers is the sum of non-equal biaxial coherency stresses, due to the epitaxial relationship between the two sublattices, and a hydrostatic stress, analogous to that found in the pure Mo samples. During irradiation, the hydrostatic component of the total stress is relaxed, with no significant changes of the coherency stresses. The value of the stress-free lattice parameter much lower than the bulk Mo for small bilayer periods is interpreted in terms of intermixing between Mo and Ni atoms during growth. The presented results demonstrate the importance of ion beam irradiation as a tool for a controlled modification of thin films, thus giving insight in the physical processes that take place during the film growth.

9:30 AM U12.4
Super Hardnening of W/NbN Nanolayers Under Shallow Nanoindentation. Scott X. Mao and B. M. Ennis; Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania.

Superlattice materials are nanocomposites that exhibit a hardness at small bilayer repeat periods which exceeds the hardness predicted by the rule of mixtures for normal composites. The objective of this investigation was to utilize the experimental data obtained from nanoindentations and image scanning to examine the behavior of the superlattice material, W/NbN. Nanoindentations and in situ surface imaging were conducted over a range of applied loads on samples of W/NbN with two different bilayer periods (5.6 and 10.4nm), and monolithic samples of the niobium nitride (NbN) ceramic and the tungsten (W) metal which comprise the superlattice material. Additional shallow nanoindentations were made to a depth equal to the individual layer thicknesses of the nanocomposites. The mechanical properties were determined using the Oliver and Pharr method and compared for all the samples. The load versus displacement curves are also compared. The energies of indentation are calculated. The characteristics of the material pile-up resulting from the nanoindentations are determined from the scanned surface images. The experimental results are discussed to evaluate the influence of the different factors to the observed increase in hardness. The results indicate that the elastic modulus does not influence the hardness of the superlattice materials. The hardness and load versus displacement curves for the shallow indentations show little difference in behavior between the NbN sample and the two superlattice materials. However, an increase in hardness is observed in the superlattice materials at deeper indentation depths. The results indicate that this increase in hardness is related to the nature of the interface between the layers in the superlattice materials.

9:45 AM U12.5
Asymmetry in the Adhesion Of Silver to Zinc Oxide in Multilayers Sputtered On Glass. Etienne Barthel, Olivier Kerjan and Elin Sondergard; Surface du Verre et Interfaces, CNRS/Saint-Gobain, Aubervilliers, France.

Film adhesion is an essential parameter in thin film and multilayer design because, in combination with the other mechanical characteristics of the film, it controls delamination and durability. Thin film deposition is a widespread surface modification technique in the glass industry. We have developed a technique to measure the adhesion energy of thin sputtered multilayers on thick glass substrates and present the results obtained on silver/ceramics films. Method: several adhesion tests prove incompatible for operation with a typically 50 nm thin film (e.g. inertia based tests like spinning wheels or shock waves) or a thick brittle substrate (e.g. bending, indentation). Tests which are mechanically unstable were also discarded because they are very sensitive to film inhomogeneities and flaws. We have therefore developed a wedge test (or double cantilever beam at fixed grip): the film, as deposited on its substrate, is glued (epoxy) to another glass sheet. Cleavage is induced by insertion of a wedge. In this configuration, the crack propagation is stable and the crack length can be measured, on the same sample, for several crack opening values. From this data, the adhesion energy is calculated. Such a procedure results in reliable data collection. XPS and AFM analysis of the cleaved surfaces have shown that in our typical Ag/ceramics multilayers, cleavage of these sandwiches result in crack propagation at a well defined interface within the multilayer. Results: In the glass/ZnO//Ag/ZnO system (where // denotes the interface of rupture), the adhesion energy we obtain is 1.6 J/m2. As a reference, we tested the glass/Si3N4//Ag/ZnO stack. The lower adhesion energy (0.7 J/m2) is consistent with lower gap of ZnO and also the better affinity of Ag for O. Much higher values are obtained for other low gap oxide underlayers like TiO2 and SnO2 (glass/SnO2/Ag//ZnO: 2.0 J/m2). Note that the interface of rupture has now shifted to the upper Ag/ZnO interface. The ZnO/Ag interface is clearly weaker than the Ag/ZnO. This result reflects the impact of the structure of the interface on the adhesion energy. Although the method allows for adhesion energy and chemical or structural characterization right at the interface of failure, the adhesion energy values reported here appear somewhat overestimated. The typical thermodynamic adhesion energy of Ag to large gap oxides, as measured by the sessile drop technique, is in the 0.3-0.4 J/m2 range, and is expected to double for lower gap oxides. The discrepancy suggests the mechanical response of the glue has to be experimentally minimized and taken into account more accurately in the model. For that purpose, we are complementing the wedge test by a stressed overlayer technique in order to try and obtain converging values by two different techniques.

10:30 AM U12.6
Correlation of Stress State and Nanohardness in Heat Treatment of Nickel-Aluminide Multilayered Thin Films.
Evan Andrew Sperling and Peter M Anderson; Materials Science and Engineering, The Ohio State University, Columbus, Ohio.

The experimentally observed enhancement in hardness and yield strength in nanoscale metallic multilayers is often explained by the confinement of dislocations to individual layers. However, it is not clear whether such confinement stems largely from the structural resistance to dislocation transmission provided by interfaces or from the oscillating bi-axial stress state between layer types. To address this issue, Ni(Al)/Ni_3Al multilayered thin films with (001) texture were fabricated by DC magnetron sputtering to a thickness of 5 microns, and removed from the NaCl substrates. The microstructural parameters varied were volume fraction (0.4 to 0.6 Ni(Al)) and bilayer thickness (300 nm and 50 nm). The bi-axial stress oscillation was deduced from x-ray diffraction measurements of the (002) peak position. Nanoindentation provided the hardness of the multilayers relative to a reference film. The stress oscillation and hardness of the as-grown multilayers varied by as much as 25% for the different volume fractions and bi-layer thicknesses considered. Heating these free-standing multilayers in an Argon environment at 673 K produced ~100% increase in biaxial stress oscillation. The relative hardness increased only 25% for the 300 nm samples but 100% for the 50 nm samples. Heating at 1073 K produced a morphological breakdown of the 50 nm samples and pronounced thermal grooving of Ni3Al layers in the 300 nm samples. The latter samples displayed a 70% decrease in stress oscillation and a 50% drop in hardness. The results suggest that the stress state and interfacial structure of free-standing multilayers can be modified by post-deposition heat treatment and further, stress oscillation is an important ingredient for multilayer hardness, particularly at smaller bi-layer thickness.

10:45 AM U12.7
Design Maps for the Tensile Yield Strength of Nanoscale Metallic Multilayers. Adrienne V Lamm and Peter M Anderson; Materials Science & Engineering, The Ohio State University, Columbus, Ohio.

Metallic nanolayered composite materials often exhibit yield strengths well beyond that of the constituents from which they are constructed. However, experimental data frequently shows that there is a critical layer thickness below which the strength no longer increases. To help understand the origins of this critical layer thickness and to guide future design of multilayers, maps of the internal stress and overall tensile macroyield stress are shown as functions of volume fraction and bi-layer thickness, for fixed lattice parameter mismatch and elastic modulus mismatch. Adopted here is a premise suggested by embedded atom simulations of Cu/Nb multilayers and recent experimental work on γ -Ni/ γ '-Ni_3Al multilayers that the overall tensile strength is determined by the applied stress necessary to eliminate the compressive bi-axial stress in the alternating layers. The results indicate that at large bi-layer thicknesses, decreasing the scale of the multilayer does increase its tensile strength. However, a critical bi-layer thickness exists below which continuing to decrease the scale does not serve to increase the overall strength. Below this critical bi-layer thickness, multilayer tensile strength is most effectively improved by increasing the stored compressive stress. This is achieved by decreasing the volume fraction of the compressively stressed phase.

11:00 AM U12.8
Nanomechanics, Chemistry and Structure of White Spot Lesions. Adrian B Mann, Ceramic and Materials Engineering, Rutgers University, Piscataway, New Jersey.

During the early stages of tooth decay the dental enamel's surface is attacked by acid that de-mineralizes the inorganic-organic matrix to create a white spot lesion. These are highly compliant, soft regions where the ratio of hydroxyapatite to protein in the enamel has been greatly reduced. The lesions show large variations in their mechanical properties with distance from the enamel surface. It is, however, surprising to find that there remains a thin layer close to the surface that is less de-mineralized and mechanically less compliant than the lesions' interiors. Thus, from a mechanical perspective a lesion resembles a thin, relatively hard and elastic, layer on a soft, compliant interior, which is itself on a hard and elastic substrate (the undamaged enamel). Since the interior of the lesion offers little support to the surface layer it is very easy for the surface layer to be damaged during mechanical loading (e.g. mastication). By a combination of mechanical wear, chemical attack and erosion white spot lesions can develop into caries (cavities) that will need to be filled with a restorative material. Characterization of the mechanical properties, chemical variations and structural changes across the lesion demonstrates how each is important in the processes of dental decay. Treatments for lesions involve exposure of the enamel surface to topical chemicals containing ions such as calcium and fluoride. Under ideal circumstances the remineralized surface would be an exact recreation of the healthy enamel. Unfortunately, this is rarely the case as remineralization tends to be greatest at the lesion's surface. Successful treatments for white spot lesions must over come this problem. This study has examined the effects of chemical treatments on the mechanics of lesions, and hence, how treatments can be improved to prevent the development of lesions into caries.

11:15 AM U12.9
Mechanical Properties of DLC/Hydroxyapatite Nanocomposites. Roger Jagdish Narayan and Bryan Bell; Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia.

Studies of orthopaedic implant failures have shown that mechanical failure of an implant almost exclusively occurs at the biomaterial-tissue interface. Hydroxyapatite (HA) mimics the behavior of natural bone, and provides a strong, long-lasting adhesive interface between a bone replacement implant and the surrounding tissue. Currently, thin film HA is not commonly used because it contains defects, porosity, and cracks. Delamination of the hydroxyapatite film and the creation of hydroxyapatite particles may lead to implant wear and loosening. Furthermore, the coating also acts only as a temporary barrier to ion release from the bulk biomaterial. One method to improve the tribological properties of a bioactive coating is to strengthen the microstructure of the coating through the placement of a DLC interlayer. DLC coatings possess properties close to diamond in terms of hardness, atomic smoothness, and chemical inertness. We have developed a diamondlike carbon/HA bliayer, in which the bilayer surface (nano-HA) is bioactive and the interlayer (DLC/metal nanocomposite) is biocompatible, wear resistant, and corrosion resistant. We have successfully deposited hydroxyapatite and DLC films by ablating a hydroxyapatite target and a graphite target using a KrF laser. A novel target design was adopted to incorporate alloying atoms into the films during pulsed laser deposition. These alloying elements possess unique biological properties. Surface morphology was studied using SEM, interfacial structure was studied using TEM, and HA phase microstructure was studied using XRD. Hardness and modulus of DLC interlayer and the DLC/HA were determined using a nanoindentation instrument. Examination of the Raman spectra revealed a reduction of compressive stresses for DLC nanocomposites. It was found that addition of a small amount of noncarbon atoms significantly reduced the internal stress and enhanced the tribological performance of the DLC films. By varying the dopant concentration as a function of distance from the interface, it is possible to create a functionally gradient DLC film. The DLC/nanocrystalline HA bilayer material is potentially useful for several orthopedic implant designs.

11:30 AM U12.10
Residual Stresses in Sputter Deposited Austenitic 330 Stainless Steel Thin Films and Copper/330 Stainless Steel Multilayers. Xinghang Zhang, Amit Misra, Haiyan Wang, Mike Nastasi, John D Embury, Terry E Mitchell, Richard G Hoagland and John P Hirth; Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, New Mexico.

Sputter deposited single layer 330 stainless steel were found to have face centered cubic (fcc) crystal structure, and high tensile residual stresses. The origins of these intrinsic, growth stresses and the effects of these stresses on the microstructure, in particular, the formation of nanoscale {111} growth twins as revealed by high resolution transmission electron microscopy and surface morphology are discussed. The variation of residual stress as a function of film thickness as well as substrate bias is studied systematically. The residual stress evolution in single layer 330 stainless steel films is compared with Cu/330 stainless steel multilayers to gain insight on the role of interfaces on residual stress. This research is funded by DOE, Office of Science, Office of Basic Energy Sciences.

11:45 AM U12.11
Investigation of Structural and Mechanical Properties of UV and Microwave Irradiated Al_2O / ZrO_2 Multilayers by Sol-Gel Coating. Phani Ratna Ayalasomayajula and Henry Haefke; Micro and Nanomaterials, CSEM, Swiss Center for Electronics and Microtechnology, Neuchatel, Switzerland.

Thin film multilayers of Al_2O_3 / ZrO_2 as well as individual Al_2O_3 and ZrO_2 films have been deposited on silicon(100) wafer, quartz, AISI 440C steel substrates at room temperature by sol-gel dip coating technique followed by annealing at different temperatures ranging from 200^{o}C to 800^{o}C for 10 hours. Prior to annealing the deposited films on quartz and 440C steel have been exposed to UV radiation (248 nm wavelength) and microwave (2.45 MHz at 800 W) for 10 min. Unlike other preparation methods microwave heating is generally quite faster, simple, and very energy efficient. Therefore, it provides a processing technique to synthesize various inorganic materials. The lower temperature and shorter time with microwave irradiation might be ascribed to the activating and facilitating effect of microwave on solid phase diffusion. X-ray diffraction and scanning electron microscopy (SEM), energy dispersive X-ray analyzer techniques have been employed to characterize structural, morphological and elemental composition of the films. Adhesion strength failure measurements on films performed by scratch test in progressive loading sequence have shown critical loads up to 35 N. Nanohardness indentation tests of the films exposed to microwave have shown hardness 13 GPa with elastic modulus 95 GPa when compared to the unexposed sample 3.5 GPa with elastic modulus 47 GPa. The friction properties of the films have been measured at low and high relative humidities by standard pin-on-disc method. The wear rates have been calculated and wear mechanisms of the films are discussed based on the SEM observation of the worn surface morphologies.