SYMPOSIUM L
Thin Films-Stresses and Mechanical Properties IX

November 26 – 30, 2001

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Symposium Support
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as Volume 695
of the Materials Research Society
Symposium Proceedings Series

*Invited paper
SESSION 1.1.1 STRAIN RELAXATION AND STRENGTHENING MECHANISMS

Chair: R.P. C. Curnier and Guanhui Xu
Monday Morning, November 26, 2001
Room 304 (Hynes)

8:30 AM #1.1.1 SOLID SOLUTION STRENGTHENING IN Pt-Ru THIN FILMS.
Richard P. Voeck, Lehigh University, Dept of Materials Science and Engineering, Bethlehem, PA.

The general effects of solid solution alloying on microstructure, development and strength are well known for bulk materials. However, the mechanical properties of solid solutions of thin films have not been fully studied because interest has traditionally focused on relatively pure materials. Because the mechanical behavior of thin films is often dominated by different mechanisms than those that control bulk behavior, the relative importance of solid solution effects on residual stress and stress relaxation cannot be simply extrapolated from bulk models. As a result, the effectiveness of solid solution alloying relative to other strengthening mechanisms unique to thin films is not well understood. In this work, thin films of Pt alloyed with Ru were studied with the goal of improving our fundamental understanding of thin film strengthening behavior. We have selected Pt-Ru as both a model system and a system with potential commercial application for integration with high-K dielectric materials such as BST. Pt films forming complete solid solutions from 0% to 20% Ru were prepared by magnetron sputtering. The relative strengthening effects of grain size, film thickness and solute fraction were evaluated using a combination of techniques including substrate curvature and nanoindentation. Comparison between experimental results and simple models show that solid solution strengthening can have a large effect on the mechanical behavior of thin Pt films. This should also be relevant for other PCC materials such as Cu and Al.


Heteroepitaxial films of aluminum bi-crystals grown on silicon offer a model system in which to study thin film plasticity of polycrystalline materials. This is because these films have well defined orientation relationships with respect to the substrate, and thus allow a quantitative correlation to be made between experimental observations and elasticity theory analyses. In this work, we have determined the strain state and dislocation density in these films during thermal cycling using x-ray diffraction. A sin^2(θ) analysis was used to find the strain state in the film at each temperature and a peak width analysis was used to estimate dislocation densities. Strain-temperature curves are smooth and surprisingly similar in form to the stress-temperature curves commonly obtained from polycrystalline films using substrate curvature methods. X-ray peak widths are strongly orientation dependent. Peak widths from (133) and (422) planes changed by a factor of two between room temperature and 450°C while peak widths from (222) planes remained unchanged. Direct comparisons will be made between the analyses of strain and dislocation density as determined by x-ray diffraction and in situ transmission electron microscopy observations of dislocation nucleation and glide in these films. The relevance of these observations to general polycrystalline behavior will be discussed.

9:15 AM #1.1.3 DIRECTION DEPENDENT GRAIN INTERACTION MODELS FOR THE DIFFRACTION STRESS ANALYSIS OF THIN FILMS. Udo Wekel, Peter Lamparter, Eric J. Mittmeyer, Max Planck Institute for Metals Research, Stuttgart, GERMANY; Matteo Leoni, Università di Trento, Dipartimento di Ingegneria dei Materiali, Trento, ITALY.

X-ray diffraction is a powerful tool for the stress analysis of polycrystalline thin films. However, it can provide quantitative data only if the diffraction elastic constants (relating the measured diffraction strains to strains) are known. Usually, the diffraction elastic constants are calculated from single crystal elastic constants by adopting a grain interaction model. It has been recently shown [1] that the grain interaction models devised for bulk polycrystals, for instance the models of Reuss, Voigt, Voigt-Needles-Hill and Eakley-Kroner, can be inappropriate for the strains recorded from thin films. As a result, a new, so-called direction-dependent grain interaction model based on the grain interaction assumptions first formulated by Voeck & Witt [3] has been proposed [1, 2], which is more appropriate for grain interaction in thin films. In this work, the concept of direction-dependent grain interaction models for the diffraction analysis of thin films is generalised, overcoming some unrealistic (extreme) grain interaction assumptions involved in the cited literature [1,2,3]. Simulated as well as experimental diffraction strain data for untextured and fibre textured thin films are analysed by applying both the traditional and the new direction-dependent grain interaction models. The results are compared critically and checked for consistency by simultaneous analysis of strain data for different (independent) reflections (i.e. crystallographic directions). Theoretical models are shown to extract erroneous stress estimates from diffraction strain data (errors as large as 50%) when the grain interaction in the thin film is direction-dependent. [1] M. van Keulen, J. J. B. Kurkijärvi, E.J. Mittmeyer, J. Appl. Phys. 86 (4), 1994-1994 (1999). [2] M. Leoni, U. Wekel, P. Lamparter, E.J. Mittmeyer, Phil. Mag. A, 81 (3), 597-623 (2001). [3] R.W. Voeck, F. Witt. J. Appl. Phys. 61 (7), 2165-2171 (1986).


The ability to produce short wavelength optoelectronic devices based on III-nitrides has been severely limited due to the residual tensile stress of AlGaN grown on GaN. Tensile-strained (0001)-oriented wurtzite heterostructures lack the critical resolved shear stress necessary for first-order slip systems to be effective in relieving strain via misfit dislocation generation. Previous work in our group has shown that catastrophic stress relaxation in AlGaN occurs prior to the generation of misfit dislocations, which subsequently nucleate at crack tips and propagate through the film. Early in situ stress measurements revealed that up to 10% of the strain was in approximate 2 seconds during the growth of AlGaN at 0.9µm/hr at a thickness of 350-400nm. Cracking could only account for ~30% of the stress relief, and the crack density essentially remained constant in samples examined before and after the catastrophic relaxation. In this work, we have investigated the microstructural changes and energetics associated with catastrophic relaxation of AlGaN. Thin films of Al0.6GaN0.4 were grown on top of 5 µm of GaN by metalorganic chemical vapor deposition on a sapphire substrate. The stress evolution of AlGaN was monitored in situ via a wafer curvature laser deflectionmetry technique. AlGaN films of varying stages of stress relaxation (or, thickness) were studied. Stages investigated include immediately post- and post-catastrophic relaxation, and at earlier stages of relaxation. The density and types of dislocations generated through each stage of relaxation have been analysed via transmission electron microscopy. The role of cracking in the stress evolution of AlGaN along with dislocation nucleation and glide mechanisms will be discussed.

9:45 AM #1.1.5 INTEGRAL STRESS ENHANCEMENT IN POLYETRAFLUOROTHENEY/Al NANO-MULTILAYERS. Eiji Kusanou, Nooto Kikuchi, Akira Kinbara, Advanced Materials Science Ctr. Kanazawa Inst. of Technol., Matto, JAPAN.

By multilayering polytetrafluoroethylene (PTFE), which demonstrates a low surface energy of about 18mJ/m2, with metal or metal alloy, a film with high interface energy can be fabricated. In such a system, PTFE/Al nano-multilayer film is expected to have low effects of accumulated interface free energy on mechanical properties such as internal stress, hardness, and modulus. Both PTFE and Al layers were deposited by magnetron sputtering. A bulk PTFE disk was used as a target. The thickness of multilayer was 200 nm. The modulation period was changed from 6.7 nm to 200 nm. Aluminosilicate glass with a thickness of 0.9 mm was used as a substrate. Hardness was measured by a nanoindentation tester. Internal stress was analyzed by a cantilever method using 0.075 mm thick borosilicate glass as a test substrate. In addition, the strain in Al layers was evaluated by X-ray diffraction. A single layer of PTFE yielded a contact angle of about 110°, which is almost equal to that of the bulk PTFE. Internal stresses of a monolithic layer of Al and PTFE were about 0.09 GPa and 0.02 GPa respectively and both tensile. By multi-layering the two materials, the internal stress changed from tensile to compressive and increased monotonically with decreasing modulation period. The maximum of about 0.6 GPa was obtained for a film with a modulation period of 6.7 nm. The strain in Al layers was also increased to about 0.8%. The hardness was enhanced with decreasing modulation period. The interface free energy accumulated in multilayer is estimated to be about 500-1000 J/m2. This equates to about 250-500 MPa of internal stress. The fact that the experimental results well agree with the estimated values suggests that the accumulated interface energy enhances internal stress of the film.
10:30 AM L1.6
PLASTIC RELAXATION IN THIN Cu FILMS. Jonathan B. Sui, Sheffield P. Baker, Department of Materials Science and Engineering, Cornell University, Ithaca, NY.

We have previously shown that the addition of a small amount of oxygen to a passivated Cu film can cause profound changes in its thermomechanical behavior. Compared to normal passivated thin film behavior, oxygen-containing films exhibit more plastic deformation at lower temperatures and stresses, a region of increasing flow stress with increasing temperature, and a plastic memory effect that is not observed in those films studied by substrate curvature in situ. In the present work, we have relaxation measurements of passivated Cu thin films over a wide range of temperatures in an effort to study plastic deformation mechanisms. The effects of film thickness and oxygen content on the thermomechanical behavior of these films has been investigated. Quantitative analysis of relaxation data is compared with existing models of the thermomechanical behavior of copper.

10:45 AM L1.7
STRESS RELAXATION IN FREE-STANDING METALLIC THIN FILMS AND MULTILAYERS. Denis Y. Xu, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.


11:00 AM L1.8
RESIDUAL STRAIN/STRESS DETERMINATION IN TEXTURED L1$_2$, Cu$_{6}$(Fe,Al)$_{3}$Mn$_{98}$THIN FILMS. Lamamde, Salah Hayek, Harid Garmestani, PAM-FSU College of Engineering and MARTECH National High Magnetic Field Laboratory/ConNMs Tallahassee, FL.

X-ray diffraction (XRD) technique was used to evaluate the residual strain/strain in textured L1$_2$, Cu$_{6}$(Fe,Al)$_{3}$Mn$_{98}$ (LSMO) thin films. The films were deposited by liquid delivery metal-organic chemical vapor deposition (LD-MOCVD) on single crystal substrates. Measurements at different temperatures and on multi-layers are underway. The conventional LPS in the so-called crystallite group methods were used to determine the residual strain/stress. Thermal stresses were estimated and compared to the total stress assuming a linear heat source. with $\Delta$ and $\Delta^2$ plots resulted in other curve or an oscillation. A correlation between residual strain/strain, lattice mismatch, and microstructure is observed.

11:15 AM L1.9
MICROSCOPIC PHENOMENOLOGY OF PLASTIC DEFORMATION IN POLYMER/METAL LAMINATES. Sachin Rastogi, Willem-Pier Velling, Han Meier, Materials Technology, Mechanical Engineering, Eindhoven University of Technology, Eindhoven, THE NETHERLANDS.

In an increasing number of applications interfaces between metals and polymers are required to survive treatments that involve plastic deformation of both metal and polymer. In these cases there is a clear need to understand and characterise the deformation mechanisms of metal and polymer as well as the ways in which the constraints posed by the interface influence these mechanisms in a laminate. As plastic deformation may involve localization on many scales that are not known a priori we have engaged in an effort to obtain a clear phenomenological, microscopic picture of the deformation mechanisms, which will have to be determined in-situ. These involve in-situ measurements during uniaxial tensile deformation of PET-steel and PP/steel laminates using optical microscopical techniques, mainly reflection birefringence microscopy, as well as ec-scan techniques, e.g. CLSM and AFM. Results will be presented of the deformation mechanisms in the PET and PP as a function of annealing treatments of the laminate, relevant for processing in industry, will be shown.

11:30 AM L1.10
CURVATURES AND STRESSES IN MULTILEVEL METAL INTERCONNECT STRUCTURES ON Si SUBSTRATES. Tae-Soon Park, Ming Doo, Subhasish Sarkar, Massachusetts Institute of Technology, Dept. of Materials Science and Engineering, Cambridge, MA; Daniel Pantuso, Subhasish Sarkar, Intel Corporation, Technology CAD Group, Hillsboro, OR.

Curvature and thermal stress evolution in single- and multi-level interconnect line structures are analyzed both theoretically and computationally. Theoretical models are made for two limiting line structures. When the line is very wide compared to its height, each metal dielectric line behaves like an individual thin film in equilibrium, but stress is not equilibrium since it is not in contact with other lines. On the other hand, the line structure is homogenized into a single composite layer with different effective thermomechanical properties along and across lines due to geometrical misfit. Effects of passivation geometry and stiffness on the stress evolution in the lines is also presented. Since theory predicts average stress values as a function of line aspect ratio, and spacing between metal lines, it also enables the analysis of interaction between neighboring lines on the same level, which cannot be predicted by Edelkamp-type inclusion theory. The accuracy and validity of the model are examined by recourse to finite element analyses and by comparing with available experimental data for stresses and curvatures. For the purpose of capturing variation of the thermal stresses on different metallization levels and for analyzing stress effect of the upper level line arrangements involving aligned and misaligned lines on the stress states on the lower level, full 3-D finite element analysis was employed. Interaction between levels in the vertical direction and applicability of curvature and stress modeling based on the single-level structure to the multi-level structure are also examined.

11:45 AM L1.11
ELECTROPLATED Cu FILM RESIDUAL STRESS AND MECHANICAL PROPERTIES. Alex A. Volinsky, N.V. Edwards, Joseph B. Vella, Motorola DigitalDNA Labs, Process and Materials Characterization Lab, Mian, AZ; William W. Gerberich, Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN.

Copper films of different thicknesses of 0.2, 0.5, 1 and 2 microns were electroplated on top of the adhesive-promoting TiW and Ta barrier layers on <100> single crystal silicon wafers. Residual stress was measured after each processing step using wafer curvature technique employing Stoney’s equation. High stress variability and thickness dependence of the warpage factor was found. Controlled Cu grain growth was achieved by annealing films at 350°C for 3 minutes in vacuum. Annealing increased the average residual stress by about 200 MPa for all the films, bringing it within 100 MPa close to the yield stress. Residual stress increase is attributed to the thermal mismatch and partial recrystallization of electroplated Cu films. The Cu film microstructure was characterized using Focused Ion Beam Microscopy. Elastic modulus and hardness were measured using Hysteresis Tribotester with the continuous stiffness method (CSM) of the Nanoindentor XP. Thicker films appeared to be softer in terms of plasticity, exhibiting a classical Hall-Petch relationship between the yield stress and grain size. Residual stress data obtained from the wafer curvature measurements is correlated with the X-ray stress analysis.

SESSION L2: DEFECTS FORMATION
Chair: Zhigang Suo and Jacob N. Israelachvili
Monday Afternoon, November 26, 2001
Room 304 (Hyne)

1:30 PM L2.1
FLOW STRESSES AND DISLOCATION MECHANISMS IN EPITAXIAL AND POLYCRYSTALLINE THIN METAL FILMS. G. Delim, H. Etsager, Max-Planck Institut für Metallforschung, Stuttgart, GERMANY; B.L. Jensen, Department of Materials Science, University of Oxford, Oxford, UNITED KINGDOM.

Flow stresses of thin metal films (thickness $\leq 1$ μm) deposited on rigid substrates are significantly higher than those observed in the corresponding bulk metal. Furthermore, the flow stresses increase with decreasing film thickness and grain size. In this study, dislocation mechanisms in epitaxial metal films on α-Al$_2$O$_3$ substrates and in polycrystalline metal films on diffusion barrier-coated Si substrates are investigated by in-situ TEM. Thermal cycling experiments in the TEM revealed that the film/substrate interface influences thin film plasticity. The film/substrate interface acted mainly as a dislocation source for crystalline/crystalline interfaces, while for crystalline/amorphous interfaces it acted as a dislocation sink. The TEM observations indicate that for epitaxial metal films on α-Al$_2$O$_3$ the increase in flow stress with decreasing film thickness is caused by...
dislocation segments deposited near the film/substrate interface, as assumed by the Nic-Freund model. In contrast, the TEM results imply that these dislocation segments are not caused by the high, fixed normal stresses in polycrystalline metal films on diffusion-barrier-coated Si substrates, because we observed that interfacial dislocations disappeared at crystalline/amorphous interfaces.

200 PM L2.2
ACOUSTIC EMISSION MONITORED PLASTICITY INITIATION UNDER CONTACT LOADING - Nadia Fyfe, Antonio Virgilio, and Christian Klemm, Materials and Chemical Engineering, University of Minnesota, Minneapolis, MN.

The present study utilizes AE monitoring for the evaluation of yield point phenomena on 100 W surfaces with several mm of oxide and freshly cleaved (100) Mo. For Mo, previous studies suggest dislocation mechanism onset at the start of the yield excursion. In contrast, the dislocation mechanism affected by the oxide film may take place in W including plasticity initiation prior to yield excursions. Evaluation and comparison of AE signatures for both samples provides additional information towards understanding the yield point mechanisms as affected by the presence of surface films. Incorporating an AE sensor into an indentor tip eliminates sample size effects and provides a greatly enhanced detection resolution for kinks below 1 mm (Hystron, Inc.). Synchronization error between load displacement and AE data acquisition less than 10 microseconds enables a precise time scale correlation between AE and indention curves. AE transients precede yield excursions by time intervals ranging 0.01-0.09 seconds for both evaluated materials. For indentation depths prior to the yield excursion ranging from 8 to 60 nm, relative acoustic energies associated with yield excursions increased nearly three orders of magnitude. It is demonstrated that such an increase requires involvement of an additional mechanism besides the oxide film delamination. Full scale energy release associated with the oxide fracture may prevail initially, an additional mechanism becomes the dominant source of energy release as the yield point load increases. The observed trends in AE energies are consistent with the plastic instabilities assuming the number of participating dislocations determined by the displacement excursion lengths. Advanced signal analysis provides additional information on separation of plasticity and oxide fracture induced contributions of AE waveforms.

2:15 PM L2.3
DISLOCATION EMISSION AND SUBSEQUENT INTERACTIONS WITH Σ = 3 BOUNDARIES IN PENTAXIAL GOLD THIN FILMS. Gene Lucchino and Douglas L. Medlin, Sandia National Laboratories, Thin Film and Interface Science Department, Livermore, CA.

We report on the origin and nature of dislocation arrays formed near facet junctions in 111 Au thin film bicrystals. Using room temperature and in situ hot stage transmission electron microscopy (TEM), we have found that dislocations, originating from orthogonal Σ = 3 (111) and Σ = 3 (112) faceted grain boundaries. Dislocation arrays were observed with b = 1/6(112) in (111) twin boundaries near (112) facet junctions. Experiments conducted in the TEM at elevated temperature showed that the arrays originate from the emission of secondary grain boundary dislocations (SBGDs) and that the motion of the emitted dislocations is coupled with the migration of the (112) facet junctions. The SBGDs are present to accommodate misorientation and accommodate dislocation-free crystallographic domains. This interpretation was corroborated by measurements showing that the domains in the vicinity of the facet junctions deviate slightly (≤ 1°) from the exact Σ = 3 coincident dislocation lattice (CSL) orientation. We propose that the driving forces for this motion are provided primarily by the climb stress between the SBGDs arising from the disconjugate character of the (112) boundary at the intersection with the horizontal twin plane. This work was partially supported by the U.S. Department of Energy, Office of Basic Energy Sciences, under contract number DE-AC03-94ER45000.

2:30 PM L2.4
FUNDAMENTAL CRITERIA FOR THE PROPAGATION OF TELEPHONE CORD BUCKLES BENEATH DLC FILMS ON GLASS SUBSTRATES. Muyoung-Woon Moon, Kye Hwan Oh, School of Materials Science and Engineering, Seoul National University, Seoul, KOREA; Anthony G. Evans, Princeton Materials Institute, Princeton University, Princeton, NJ; Jin-Won Chang, Kwang Ryool Lee, Korean Institute of Science and Technology, Seoul, KOREA.

The topology of telephone cord buckles that form beneath compressed diamond-like carbon films (DLC) on glass substrates has been characterized with Atomic Force Microscopy (AFM) and with the Focused Ion Beam (FIB) cross section analysis. Using AFM, we measured the wavelength and amplitude of the buckles and their profiles have been measured. It has been found that, within each wavelength, the profile has symmetric and asymmetric segments but the amplitude is invariant. These changes are related to different mode mixity around the periphery of each repeat unit along the buckle, resulting in a fundamental rationale for the factors governing the wavelength. Sections made through various segments of the buckle by using the FIB imaging system result in local changes in the shape and size of the buckles that provide further insight into the buckle propagation criterion.

2:45 PM L2.5
MOCVD ZnS: Mn FILMS: CRYSTAL STRUCTURE AND DEFECT MICROSTRUCTURE AS A FUNCTION OF THE GROWTH PARAMETERS. Kathleen D. Bonsack, Katherine Dowdengo, Anna W. Topal and Alan E. Watenberg, University of Michigan, Ann Arbor, MI.

Thin film electroluminescent devices employing zinc sulfide doped with manganese are extensively used for applications in which the weight, brightness and mechanical robustness requirements preclude the use of other types of displays such as cathode ray tubes or liquid crystal displays. The physical and electrical properties of phosphors such as ZnS:Mn can often depend strongly on microstructure, which in turn depends on the growth and processing of the film. For this study, ZnS:Mn layers were fabricated by metalorganic chemical vapor deposition in the 290°C-500°C range on an AlN/InSb/Glass substrate. Selected samples were then subjected to a post-deposition anneal in H2/SiO2 at 700°C for up to 4 hours. The microstructure of the ZnS:Mn films was examined by Transmission Electron Microscopy (TEM). For all growth and annealing conditions, the film consisted of a columnar grain whose column axis was parallel to the growth direction, and which widened laterally through the thickness of the films. For the un-annealed films, the crystal structure was found to be predominantly 2H structure, with the 8H polytype being identified in the low-temperature ZnS:Mn films. The 700°C post-deposition annealing was found to initiate a solid state transformation to the cubic (3C) ZnS crystal structure. All films contained high densities of stacking faults and micropipes, whose role in the 2H-3C transformation is discussed. The possibility of preferred orientation in these films was examined by selected area and convergent beam diffraction in the TEM, as well as X-ray Bragg-Brentano X-ray Diffraction scans. The correlation between the defect microstructure and the mechanical behavior of these phosphor films is discussed.

3:30 PM L2.6

Thin films of Au can be grown on (001) Ge single crystal substrates in two equivalent [110] orientation variants, related to each other by a 90° rotation about the surface normal. There is equal probability to nucleate both variants, so the morphology of the film is of mazed bicrystal - it is polycrystalline, but all grains are in only two orientation variants. All grain boundaries are of the type Σ99 and display tilt character. Their morphology and atomic structure, as well as their evolution during annealing, was investigated by conventional and high resolution electron microscopy. Previous investigations have reported that grain boundaries in metals with low stacking fault energy undergo extended relaxations that can at times lead to a thin layer of a different structure. In this work, HREM observations of different relaxation modes in 250 grain boundaries in Au are reported and discussed in terms of mechanisms of formation and misorientation of grain boundary energy. This work is supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

3:45 PM L2.7
DISLOCATION MOTION AND FLOW STRESS IN THERMALLY STRAINED COPPER THIN FILMS. T. John Bahl, Gerhard Dehm and Eduard Arz, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

The stresses that exist in thin film metallizations can be significantly higher than those in which bulk metals yield. Moreover, the maximum stress that develops during thermal cycling increases with decreasing film thickness. However, the exact deformation mechanisms responsible for the observed strengthening are not known. In this study of thin film plasticity, the relationship between the microstructural behavior and dislocation motion has been investigated in copper constrained by a silicon substrate. The stress-temperature behavior as determined from wafer curvature experiments has been compared to deformation microstructures observed during in situ thermal cycling of both plan-view and cross-sectional specimens in the transmission electron microscope (TEM). The effects of film
thickness on dislocation motion and on the evolution of flow stress in thin copper films will be presented. Wafer curvature experiments revealed that the temperature flow stress rises linearly with inverse film thickness, but then exhibits a plateau at approximately 600 MPa for films 400 nm and thinner. Stress relaxation experiments were performed, during cooling, at temperatures between 100°C and 250°C in order to determine the activation volumes for dislocation motion. The values correspond to apparent dislocation pinning distances of less than 8% of the film thickness. Thermal cycling in both 20 kHz and 1 MHz TEMs revealed that dislocations move rather smoothly in the initial stages of cycling at 250°C. However, below approximately 200°C, the dislocations tangle and constrain one another, resulting in sporadic motion. Moreover, unexpected dislocation activity tends to be unstable on the film surface, which should experience no resolved shear stress, provides potential evidence for the occurrence of constrained diffusional creep in a 200 nm film. Such observations are relevant to the largely unknown dynamical interactions between dislocations and the surface/interface, which are likely to increase in importance during the permanent miniaturization of thin film geometries.

4:00 PM L2.8
EFFECTS OF MICROSTRUCTURE AND STRESS STATUS OF IMP TA THIN FILMS AS DIFFUSION BARRIER FOR Cu METALLIZATION. L. He b, H. Liu, Z. Q. Zeng, C.Y. Liu, Joseph Xi a, b; “Advanced Materials, Singapore-MIT Alliance, SINGAPORE; aDISC Dept., Institute of Microelectronics, SINGAPORE; bRodolphe Technologies, Inc., USA.” School of Materials Engineering, Nanyang Tech. Univ., SINGAPORE.

Effects of the microstructure and stress status of Titanium (Ta) barrier layer on its performance against Cu diffusion were studied in this paper. The Ta barrier layers were deposited by Ionized Metal Plasma (IMP) technique. After that, the de-oxidized Cu/Ta/Si samples were annealed in oxidizing ambient at 250°C, 550°C, 650°C, respectively, for 30 minutes to evaluate the barrier performance. Microstructures and residual stress of the barriers were investigated using X-ray diffraction, Film stress measurement (FSM) and Transmission electron microscopy (TEM). Four-point probe (FPP), X-ray diffractometry (XRD) and Rutherford backscattering spectroscopy (RBS) were used to study the failure behavior and mechanism. Under different growth conditions, microstructure of the Ta barriers varied with different materials of the interface were obtained. Meanwhile the stress status would change with the microstructure. The results of the sheet resistance measurements indicated that the failure temperature of the samples with more interfaces have higher than the 9 others. And according to XRD spectra, the failure could be confirmed by the appearance of new compound Cu3Si. Annealing process would not influence the interface structure of the barrier, while the residual stress could be strongly affected by annealing. Based on our results, two kinds of diffusion path coexist in the barriers: one is the grain boundary of Ta, through which Cu would diffuse into the Si substrate and lead to barrier failure. The other path is the interface in Ta, which could help out the diffusion from the grain boundary. The dominant diffusion mechanism would depend on the microstructures and stress status of the barriers, and the existence of interface in Ta barrier was believed to enhance the effectiveness of barrier layer against Cu diffusion.

4:15 PM L2.9
OCCURRENCE OF TEXTURE, TWINNING AND STRESS IN SPATTERED THIN COPPER FILMS. Brando Okole, Udo Wohlf, Peter Lamparter, Thomas Wagner, Eric J. Mittmeier, Max Planck Institute for Metals Research, Stuttgart, GERMANY.

Thin copper layers with thickness of about 500nm were magnetron sputtered at various deposition gas argon pressures (0.05-0.7 Pa) onto silicon wafers with different surface layers, namely amorphous silicon oxide or silicon nitride in a twin and sputtering system. The occurrence of texture and twinning and the development of film stress were investigated employing x-ray diffraction methods, focused ion beam and transmission electron microscopy. Substrate roughness was characterized by atomic force microscopy (AFM) and X-ray reflectivity measurements. The [111] fibre texture of the substrate as determined by AFM measurements was found to be 0.343m for the silicon oxide surface and 0.383m for the silicon nitride surface. Texture measurements performed by x-ray diffraction on the thin film samples, showed no other layer than a (111)-fibre texture with minor additional texture components. In paricular, a (111)-fibre texture component resulting from twinning was observed. Texture changes increased with decrease in substrate roughness. The twin plate probability, analysed by diffraction line broadening, was found to be higher by a factor of about two in the films deposited on the smoother silicon nitride substrates. Tensile stress of about 100 MPa was observed, by employing the x-ray diffraction 0-20-method, in the film on the smoother substrates whereas the stress was 2000 MPa for the film deposited on the rougher silicon oxide substrates. The deposition gas pressure was found to be of minor importance in texture, twinning and stress development. The combination of twinning, texture and stress development to the surface roughness is discussed.

4:30 PM L2.10
EFFECTS OF SURFACE CONSTRAINTS ON STRESSES IN HETEROEPITAXIAL FILMS GROWN ON COMPLIANT SUBSTRATES. Zhahan Peng, Edward G. Lowe, Rostrum L. Englot, University of Wisconsin, Computational Mechanics Center, Mechanical Engineering Dept., Madison, WIS.; Thomas F. Kuech, University of Wisconsin, Chemical Engineering Department, Madison, WI.

During film heteroepitaxy on a freestanding thin template, excessive stresses and high dislocation densities are generated in the template, but stresses in the film are very low. A defect-free pseudomorphic film can be produced if the film thickness does not exceed limits set by, for example, the Matthews-Blakeslee criterion. But it is not easy to realize this goal, since the freestanding thin template is mechanically unstable. In practice, a handle wafer is needed. The details of the interface between the bonded template and the handle wafer are the key issue. Ideal constraints should limit the extra degrees of freedom of the template and keep it stable, but not decrease its deformation compliance. Asymmetric three-dimensional finite element models were created to simulate the film and template, as well as the constraints between the template and the handle wafer. Transient stress analyses provided accurate values for all stress components in the film and template at each stage of the film growth. Effects of different constraint types on the stress were studied. The stress factors influencing the stresses, such as lattice mismatch strain, film and template thickness, were also examined. Analysis results demonstrate that a freestanding template provides the lowest stresses in the film. If the template is fixed in both out-of-plane and in-plane directions, the stresses in the film reach their maximum values. In other cases, the stresses are between these two limits. Bonding the template to the handle wafer by an interlayer with a lower elastic modulus is a potential solution, which can keep the template stable and reduce the stresses in the film. The simulation models and stress analysis facilitate an understanding of the effects of compliant substrate surface constraints on stress and dislocation generation. The results provide guidelines for optimization of substrate parameters to produce low-defect films.

SESSION 13: POSTER SESSION
Chair: Cheng Suan Oczan and Robert C. Camrattero
Monday Evening, November 26, 2001
8:00 PM
Exhibition Hall D (Hynes)

L3.1
ADAPTIVE PROTOCOL FOR ROBUST ESTIMATES OF COATINGS PROPERTIES BY NONINDENTATION. Michael M. Johnen* and Nature Physical Laboratory, Materials Centre, Teddington, Middlesex, UNITED KINGDOM; *Department of Materials, Queen Mary, University of London, London, UNITED KINGDOM.

The determination of the elastic modulus and hardness of a wide range of coating systems has recently been studied in the European Commission funded project “INDICoAT.” This paper describes a protocol for determining coating properties, which has been developed and tested in that project. The procedure contains strategies, simple to implement, to evaluate the response of the coating and design a suitable series of indentation experiments that enable a reasonable estimation of coating-only properties. The protocol directs how the experimental design adapts the experimental parameters to each sample. An adaptive protocol is essential to cope adequately with the different indentation responses. Indentation response depends, for example, on coating thickness, the relative properties of coating and substrate and creep response. The protocol also has to adapt itself so that it can reliably target the range of indention depth with respect to the coating thickness necessary to obtain the coating properties from the composite indentation response. Results presented show that the parameters and approach for measurement of hard coatings are very different to those required for soft or ductile coatings. The systems studied are DLC on tool steel, Au coated Ni, aluminium oxide coated Ni and aluminium coated optical glass (BK7).

L3.2
IN VIVO NONINDENTATION STUDY OF DROSOPHILA MELANOGASTER DEVELOPMENT. Antanas Daigels, Hiroshi Kutomi, Hysitron Inc; Michael Kohn, Distance Co; Jerry T. Wyrcoh, Hysitron Inc; Minneapolis, MN.

A nanometer scale quantitative quasi-static indentation technique has
membranes with $d = 2000 \mu m$ settle into a butterfly-like state with two remaining reflection symmetries and no rotation symmetry. A small increase drives these membranes into a state with rotational symmetries only and without reflection symmetries. This state is identical to that found immediately after the micromanipulation, before the first load step. The complex load-deflection behavior of the membranes was modelled and reproduced by extensive finite-element simulations covering a wide range of prestrains and pressures. The simulations enabled the extraction of $\psi$ and $E$ of the investigated SiN thin film. Secondly, it was found that the normalized displacement $u/w$ depends only on the reduced dimensionless coordinates $x/a$, $y/a$, Poisson’s ratio $\nu$, prestrained $e_{0}/\nu^{2}/h^{2}$ and reduced pressure $P^{*}(1 - \nu^{2}/Eh^{2})$. The method and results can therefore be carried over to the characterization of all compressively prestressed thin films.

L3.3

SCRATCH RESISTANCE AND MECHANICAL PROPERTIES OF POLYMER FILMS. C.D. Eisenbach, E. Kline, Research Institute for Pigments and Coatings, Stuttgart, and University of Stuttgart, Institute of Applied Macromolecular Chemistry, Stuttgart, GERMANY.

The scratch resistance of the surface of polymer films such as organic coatings is of primary importance for many applications. However, the commonly employed scratch test procedures give only limited information about structure-properties relationships. The mechanical surface stability of organic coatings in terms of the scratch resistance has been investigated for a variety of clear coats by using a new type of single scratch test. The coatings varied in the composition and the degree of curing. The dependencies of the coatings failure and the scratch profile on the scratch conditions revealed information about viscoelastic properties of the coatings surface regions including the plastic flow as well as brittle fracture, and also partial recovery of the scratch deformation (rejuvenation) after relief. The combination of the surface viscoelastic data of the coating with the stress-strain properties of free standing films allowed a correlation of these physical material data with, e.g., the glass loss of a multi-scratched coatings surface. The implication of the correlation of the optical appearance of the surface of organic coating with its mechanical and viscoelastic properties for the evolution of the coatings performance was discussed. The work was supported by the Cooke Foundation of Industrial Cooperative Research Associations “Otto von Guericke” e.V. (AfF) [Grant No. 11806] and the companies Audi AG, Daimler-Chrysler AG, DuPont Performance Coatings GmbH and Volkswagen AG.

L3.6

NANOINDENTATION AND NANOWEAR STUDIED OF SPUTTER-DEPOSITED ULTRATHIN TIN OXIDE FILMS ON GLASS SUBSTRATE. J.G. Wang, M.E. Schley, and C.G. Pentano, Materials Research Institute & Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA; D. Yang, T. Anderson and L. Kahn, Hysitron, Inc, Minneapolis, MN.

Tin oxide thin films on glass are used in a variety of applications ranging from transparent conductors to anti-corrosive and anti-scratch coatings. The surface chemistry, mechanical properties and wear resistance are crucial to the application of the tin oxide films. In the present study, ultrathin tin oxide films were sputter-deposited on the glass substrate. Surface characterization has been determined by X-ray Photoelectron spectroscopy (XPS). Nanoindentation, nanoscratch and nanowear experiments were performed on the tin oxide films with different thickness by using Hysitron TriboScope in different loads. Hardness and Young’s modulus of elasticity were determined from the depth sensing load-displacement data. A lower friction coefficient was observed on the thinner film using the ramp load scratch test. Critical loads were determined by the ramp scratch test and maintained until the critical load was observed. Above the critical load however, the wear increases sharply. Discussion on the thickness dependence of the effect of surface chemistry on the mechanical properties and were made.

L3.7

ON THE RELATION BETWEEN MORPHOLOGY AND ELASTIC PROPERTIES IN AMORPHOUS COLOMNAR TIN FILMS. Thomas J. Varick, Jr., Pedro C. Anid, Charles Hardow, Francesco Cozzamari, Gary L. Gray Penn State University, Laboratory for Parallel and Computational Mechanics, Department of Engineering Science and Mechanics, University Park, PA.

The optical, electromagnetic and mechanical properties of thin films (TFs) are directly correlated to their morphology at the nanoscale. This, in concert with the fact that

- new deposition techniques are enabling the growth of thin films with very complex morphologies,

We present results from a study of the effects of pressure on the structural and electronic properties of thin films of carbon-based materials. Thin films were deposited by laser ablation and characterized by scanning electron microscopy, x-ray photoelectron spectroscopy, and Raman scattering. In addition to thin films, we also present data on polycrystalline samples that were quenched from high pressure and high temperature conditions. The pressure dependence of the structural, vibrational, and electronic properties of these materials was studied using x-ray diffraction, Raman scattering, photoelectron spectroscopy, and diamond anvil cell. We discuss the effects of stress on the structural and electronic properties, in particular, the structural phase transitions in fullerenes at high pressures. This research is supported, in part, by grants from the Department of Energy, grant No. DE-FG03-00ER45540/A000 and Welch Foundation, Houston, Texas.

L3.11 STRUCTURAL PROPERTIES OF CARBON NITRIDE FILMS DEPOSITED BY REACTIVE PULSED LASER DEPOSITION TECHNIQUE. A.R. Pachit, J.E. Kresnowski, Department of Mechanical Engineering, University of New Hampshire, NH.

Carbon nitride films have been deposited by reactive pulsed laser deposition technique by ablating carbon in the nitrogen atmosphere at different substrate temperatures and different back pressure conditions. N(111) substrates are used in the present investigation. Deposition gases are used for the deposition of polycrystalline thin films. The deposition rates depend on laser fluence, back pressure, and target substrate temperature. The nitrogen concentration in the deposited films increases with increasing back ground nitrogen gas pressure and laser fluence. Fourier transform infrared spectroscopy has been employed to distinguish the C-C and C-N bonds. X-ray photoelectron spectroscopy has been used to study the composition of the deposited films. X-ray Diffraction and Transmission electron microscopy techniques reveal that the deposited films show a preamorphous film oriented microcrystalline structure at relatively lower laser fluence. This could be due to the high kinetic energy of the radicals in the laser produced plasma plume. Electronic, mechanical and tribological properties of these films have also been discussed.

L3.12 STRESS CHANGES IN CVD TUNGSTEN SILICIDE (POLYCYIDE) FILM MEASURED BY X-RAY DIFFRACTION. Isidro Paredes, Eyal Elish, Ben Gurion University of the Negev, Dept. of Materials Engineering, Beer-Sheva, ISRAEL.

Stresses in chemical vapor deposited polytungsten disilicide (poly - Si/WSi2) were measured at each stage of the fabrication. The individual layers of the Si/SiO2/Polycrystalline Si/WSi2 polynomial structure, were deposited sequentially on separate wafers and subjected to X-ray diffraction analysis in the Bragg-Brentano Bragg incidence conditions. The changes in the strain occurring in the WSi2 film. Samples cut from wafers containing all the layers were either heat treated at 1183K for 30 min or cycled with a 25nm thermal cycle in the strain in the WSi2 film. This change in the strain of the WSi2 layer, following each step of the fabrication process, was evaluated by the lattice parameter of the c-axis. The stress is affected by the layers of the multilayered film. As an deposited polycrystalline layer on top of WSi2 reduces its stress, since it introduces a compressive component, which further decreases upon annealing. It also maintains Si supply at the poly-Si/SiO2 interface, thus, eliminating Si outdiffusion during heat treatment in an oxygen-containing ambient. Cracking the system by a thin oxide layer modifies the stress pattern of the WSi2, which becomes compressive. Average microstrains in WSi2, were evaluated by XRD line broadening according to the Williamson - Hall method. It is shown how the stress can be minimized by a modified approach involving the addition of passivation and thermal oxide layers in the production process without altering the basic technique of fabrication.

L3.13 EVALUATION OF THERMAL STRESSES IN THIN FILMS USING X-RAY DIFFRACTION. Josef Koeke, Erich Schmidt Inst and Inst of Microelectronics, Leibn. Rechenzentrum, Munich, Germany.

X-ray diffraction is used to characterize residual stresses and...
unstrained lattice parameters in a variety of heterostructural structures [e.g., GaN/Al\(_2\)O\(_3\), GaN/Al\(_2\)O\(_3\)/Si, Al\(_2\)O\(_3\)/Al\(_2\)O\(_3\)], which indicates that the temperature-dependent
magnitude of anisotropic thermal expansion coefficients and the deposition
method define the stress sign and the amount of stress changes in the films with the temperature. In the case of metal layers the results indicate the presence of plastic flow in contrast to the crystalline layers which exhibit no yielding. The results are consistent with the model of thermal stresses originating from the mismatch of thermal expansion coefficients of thin film and substrate.

L3.14

A compressively strained elastic film bonded to a viscous layer can relax its stress by buckling and in-plane extension as the viscous layer flows underneath. Experimental observations suggest that, while buckling is significant at the center of a large film and in-plane extension is important at the edges and small film islands, the two mechanisms are essentially coupled during the relaxation process. The present study provides a theoretical model for the coupled relaxation process. The model is validated as a thin plate under the combined action of lateral loads and forces in the middle plane. The flow of the viscous layer is approximated by the theory of lubrication. The interface between the film and the viscous layer is assumed to be perfect with no slipping or debonding. A linear stability analysis is performed to determine the critical wave number of the buckling instability for an infinite film under the plane strain condition. For each fixed wave number, the critical wave number decreases as the critical wave number, there exists a constrained equilibrium state, at which the buckling

L3.15
NANOINDENTATION ANALYSIS OF VISCOPLASTIC COATTINGS ON Ti-6Al-4V. M. Bozorgzadeh, University of Minnesota, Dept. of Chemical Engineering and Materials Science, Minneapolis, MN; Bradford Sun, Intel Corporation, Santa Clara, CA; William W. Gerberich, University of Minnesota, Dept. of Chemical Engineering and Materials Science, Minneapolis, MN.

The mechanical properties of two polymer coatings, namely, a polyimide and a spin-on polymer on silicon substrates were evaluated using nanoindentation. The previous knowledge of the viscoelastic unloading model, the static modulus and hardness were obtained. Nanoindentation creep tests were also performed, where a value for the modulus was extracted by fitting the data to an equation based on the three-element standard linear solid model. Aside from being comparable to values reported in the literature, the moduli from creep experiments are at most 1.3 times lower than the static moduli. This discrepancy can be attributed to inherent limitations of the elastic-plastic model. In a two-dimensional viscoelastic model, the assumption of the absence of adhesive forces and thus the absence of adhesive forces upon pull-off.

L3.16
PREDICTING TRENDS IN METAL-CERAMIC ADHESION. Donald J. Siegel, University of Illinois at Urbana-Champaign, Dept. of Physics, Urbana, IL; Louis G. Hector, Jr., General Motors Corp, Materials and Process Lab, Warren, MI; James B. Adams, Arizona State Univ, Chemical and Materials Engineering Dept, Tempe, AZ.

Metal-Ceramic interfaces play a crucial role in applications ranging from catalysis to microelectronic devices. However, there exist no general guidelines for understanding such fundamental properties such as the work of adhesion, which impacts the mechanical strength of the joint. Using first-principles simulation methods, we have conducted a systematic study of a broad class of interfaces, involving adhesive interactions of aluminum oxides, carbon, nitrides and oxides, the effects of surface polarity, alloying agents, and non-oxidic bonding.

L3.17
TRANSIENT SPUTTERING OF SILICON STUDIED BY MOLECULAR DYNAMICS SIMULATIONS. Edwin F.C. Holdeman and Barend J. Thijssen, Delft University of Technology, Department of Materials Science and Technology, Delft, NETHERLANDS.

Molecular dynamics simulations of the initial sputtering of Si(001) were performed in order to study the transient sputtering phase that is important for interpreting the ion scattering measurements of transient layers. Our main goal is to get a better understanding of the variations in the processes that happen in the initial stages of sputtering, before the steady state is reached. The Si(001) sputtering targets have a surface area of \(1 \times 10^2 \text{ cm}^2\) and contain 60 ML of Si. The targets were exposed to an Ar beam of 500 eV and 1.5 A\(^2\). The simulations were performed using the Amber force field.

L3.18
STATIC SOFTENING AND DYNAMIC INSTABILITIES IN AL-BASED FOILS USING THE BULGE TEST. Mipoon-Cieler, Ayatollah Karami, Jean-Luc Martin, Swiss Federal Institute of Technology (EPFL), Department of Physics, Lausanne, SWITZERLAND.

Effects of recovery and recrystallization on subsequent stress - strain behavior were studied in foils of non-age hardenable Al based alloy. A bulge testing device for the mechanical testing of thin free standing films and foils enabling the detection of large strains was employed. The bulge tester was used at RT both for the predeformation of the foil in the biaxial mode and also for the study of softening after subsequent one-step annealing. Three stages of the strength drop as a function of the annealing treatment were observed between RT and 500°C. The contribution of different annealing processes to the softening was established using transmission electron microscopy. It was found that below 200°C only recrystallization inside dislocation cells and the appearance of the cell substructure occurred. Between 200°C and 300°C the formation of a subgranular structure was observed. The softening process is terminated by a partial recrystallization at higher annealing temperatures. The dynamic interaction of solutes with dislocations was revealed during prestraining as well as poststraining of foils. This effect resulted in the appearance of a negative strain rate sensitivity and dynamic instabilities after appropriate prestraining and annealing conditions. The above results show that bulge testing of Al foils allows to study the successive annealing stages by measuring the most important microstructural parameters of plastic deformation (yield stress, work hardening rate, ductility, strain rate sensitivity, etc.). These stages could be related to the microstructure evolution.

L3.19

We have developed a new and general x-ray microbeam technique for submicron resolution 3D investigation of structure, orientation, elastic strain, and plastic deformation in thin-films, substrates, and bulk materials. The technique utilizes polychromatic (white) x-ray monochromators, different aperture diffraction beam shaping, and computer automated analysis of the crystallographic orientation and strain distribution in submicron resolution 3D volumes of single crystals, polycrystals, heterostructures, and deformed materials. The basic features of the technique will be described and the results of
Present study utilizes AE monitoring for the evaluation of yield point phenomena in a thin film of TiAl intermetallic with several nm of oxide and freshly cleaved (100) Mo. For MoO, previous studies suggest dislocation nucleation onset at the start of the yield excursions. In contrast, more complex mechanism affected by the oxide film may take place in W including plasticity prior to yield excursions. Evaluation and comparison of AE signatures for both samples provides an additional information towards understanding of the yield point mechanisms as affected by the presence of surface film. Incorporating AE sensor into an indent tip eliminates sample size effects and provides a greatly enhanced detection resolution for the loads below 1 mN (Hytrobin, Inc.). Synchronization error between local displacement and AE transient acquisition less than 10 microseconds enables a precise time scale correlation between AE and indentation curves. AE transients precede yield excursions by the time intervals ranging 0.01-0.09 microseconds for both evaluated materials. For indentation depths prior to excursion ranging from 6 to 80 nm, retard acoustic energies increased nearly three orders of magnitude. It was demonstrated that such increase requires involvement of an additional mechanism besides cracking or oxide film delamination for W. While energy release associated with the oxide fracture may prevail initially, other mechanism becomes a dominant source of energy release when yield point load increases. The observed trends in AE energies were consistent with the plastic instabilities preceding yield excursions assuming the number of dislocations participating in the instabilities determined by the displacement excursions lengths. Advanced signal analysis provided an additional information on separation of plasticity and oxide fracture induced contributions of AE waveforms.

L3.21

STUDY OF TiAl, AND TEMPERATURE-DEPENDENT DEFORMATION OF Cu THIN FILMS BY MICRO-TENSILE TESTING, A. Brederode, O. Kraft, and E. Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

The stress-strain behaviour of Cu films was investigated using a micro-tensile machine, which is mounted into an X-ray diffractometer and equipped with a heating stage for temperatures up to 1500°C. Films with a thickness of 200 nm and grain size of about 200 nm were produced by magnetron sputtering onto polisided substrates, which can be deformed elastically up to a maximum strain of 3%. The sample strain and the film stress are measured using a laser scanning system and X-ray diffraction (sin^2 omega-method). In contrast to the commonly used wafer-curvature method, this technique allows the film strain to be varied independently from the temperature. In addition, the evolution of the dislocation density during the experiments was determined by monitoring the intensity of the 200 peak of the X-ray peaks. The films were strained with a strain rate of about 10^-3 s^-1 to different total sample strains between 0.5 and 2.0% and the subsequent stress relaxation measured. Even at room temperature, stress relaxation of about 5% of the yield strength was observed by this method. The width of the X-ray peaks. The strain between the YSA and the subsequent relaxation process was independent of the initial strain state and accompanied by a decrease in the X-ray peak width, indicating a decrease in the dislocation density. One of the key experiments focused on the relaxation behaviour at elevated temperatures with the aim to characterize the temperature dependence of the relaxation process and to obtain an understanding of the underlying deformation mechanisms.
DLC (Diamond-like carbon) films have been widely used in the last few years for a number of applications. The DLC films are characterized by a different sp2/sp3 ratio, which influences the mechanical and tribological properties. Gas cluster ion beam (GCIB) assisted deposition is proposed as a novel method in DLC synthesis. In this method, energetic gas cluster ions deliver extremely high densities into very localized and shallow atomic level regions of a substrate surface and subsequently, because the impact point of a cluster ion for an instant attains conditions of high pressure and high temperature, it is considered that the phase transition from sp2 to sp3 is enhanced even when the substrate is held at room temperature. The characteristics of the DLC films produced by GCIB assisted deposition were studied by Ne/C X-ray Absorption Fine Structure (NEXAFS) study. The carbon K-edge data show that NEXAFS is sensitive to the local structure around the carbon atom. The resonance from carbon to pi to pi* orbitals in sp2 carbon and the resonance from 1s orbital to sigma orbital in sp3 carbon present a distinguishable difference in energy and a simple identification of each contribution can be made. Thus a quantitative determination of the sp2/sp3 ratio can be performed. Our data indicate that the GCIB assisted deposition is superior method to form the hard DLC film and NEXAFS technique is useful for the characterization of DLC films.

L3.26

PROPERTIES OF TETRAHEDRAL AMORPHOUS CARBON FILMS DEPOSITED BY THE FILTERED CATHODIC ARC METHOD

Matsuyama, Fuji Electric Corporate Research and Development, Ltd., Material Science and Technology Laboratory, Yokosuka-city, Kanagawa, JAPAN.

An application of tetrahedral amorphous carbon (ta-C) to a hard disk media overcoat is expected due to its high hardness. We are studying the growth of ta-C films by using the Filtered Cathodic Arc (FCA) method. In this work, we paid close attention to the relationship between the sp3-C ratio and other properties such as hardness and Raman peaks. The sp3-C ratio is an important parameter which represents the structure of carbon. Several kinds of films were deposited by the FCA system at the different substrate bias voltages. For comparison purposes, sputtered amorphous carbon (a-C) films and High Oriented Pyrolitic Graphite (HOPG) were also prepared. The sp3-C ratio could be estimated by separating the sp2 and sp3 components in the C 1s X-ray photoelectron spectroscopy spectra. Hardness measurements were performed by nanoindentation. As a result, some films deposited by the FCA method had high hardness and high sp3-C ratio up to 75GPa and 0.75 respectively, and we found that the hardness of the carbon films including sputtered a-C and HOPG increased in proportion with the sp3-C ratio. Due to this relationship, it is suggested that the microstructure of carbon films is closely related to those mechanical properties. Furthermore, as the sp3-C ratio decreases, the number of the G-peak in the Raman spectrum increased linearly from 1540cm-1 to 1570cm-1, while the ratio of intensity of the D-peak to that of the G-peak decreased monotonically from 1.1 to 0.2. We consider that these parameters are related with the sp3-C configuration and useful indices correlated to the sp3-C ratio.

L3.27

THIN FILM CHARACTERIZATION USING THE POINT-DEFLECTION METHOD

Ming Tung, Roxanne L. Engenak, Edward G. Lovell, Computational Mechanics Center, University of Wisconsin Madison, WI.

The Point-Deflection Method (PDM) is a potentially useful technique for measuring the internal stress of freestanding thin films. Compared with the conventional pressure bulge test, it overcomes the difficulties normally used to measure the stress sensitivity to boundary constraints. Another intrinsic merit of the method is that it involves no micromachining in sample preparation and thus is nondestructive in nature, which is an advantage over most microbeam diagnostic procedures. The PDM is similar to traditional depth-sensing indentation tests in that it also uses extremely low-load instruments, e.g., a Nanoindenter, to apply a small concentrated transverse load at the center of a freestanding pretrained thin film. Instead of the semi-infinite solid model used in typical depth-sensing analyses, the film is considered as a membrane/plate-like component. The response is a combination of uniform biaxial in-plane tension and flexure from the external point load acting at its center. Load-deflection relationships are observed based on classical mechanics. Results indicate that even at the loading point (which is theoretically a singularity), the small out-of-plane displacement of the film is linearly related to the applied force and inversely proportional to the film thickness. Thus by measuring the transverse displacement at locations other than the center, the average internal stress is readily obtained. The analysis has been completed for films of both circular and rectangular shapes. Theoretical analyses have been correlated with finite element simulations. The feasibility of the method has been proven in the stress measurement test of a photomask pellicle film. The test was relatively easy to perform, nondestructive, and highly repeatable. The results show good agreement with stress data obtained from resonant and nonresonant tests on the same membrane. Consequently, the PDM has excellent potential for both research applications, and further development, as a production-related tool for technologies incorporating membrane-like components, such as next-generation lithography masks.

L3.28

NANOINDENTATION AND MAGNETO-OPTICAL STUDY OF OXIDIZED COBALT THIN FILMS: S.R. Mistry, G. Sherfy, T. Buckner, The University of Memphis, Dept of Physics, Memphis, TN; J. Rho, M. Roy, The University of Memphis, Dept of Biomedical Engineering, Memphis, TN.

We have studied the structural, micromechanical and magneto-optical properties of oxidized Co[2500 Å/Co[1000 Å] thin films as a function of annealing temperature. The electron beam deposited films were postoxidized in the presence of oxygen at various temperatures. The oxidation process leads to formation of double-layered structure of FeCoO4 on top of the metallic Co. The grain growth is monitored using atomic force microscopy. Transmission electron microscopy reveals the hexagonal structure is the predominant phase coexisting with minor reflections in the Co layer. The microstructural hardness measured using a nanoindentor as a function of grain size and annealing temperature shows a linear relationship. Furthermore, the magnetic magneto-optical Kerr performed using HeNe laser (MOR) shows negligible difference in the coercivity of the film as a function of annealing temperature although difference in magnitude and sign of MOR signal is noticed.

SESSION L4: MECHANICAL PROPERTIES AND NANOSCALE TESTING I

Chairs: Congit Sim, Otken and Richard P. Vinci
Tuesday Morning, November 27, 2001
Room 304 (Hyne)

8:30 AM L4.1

THE STIFFNESS OF THIN FILMS: From Spigen, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

The stiffness of thin films is often considerably lower than that of their bulk counterparts. This effect has been observed in different types of materials (metals, ceramics, ...), in uniaxial and in biaxial loading, and in free-standing as well as supported films. The possible microstructural origins of a stiffness deficit (porosity, cracking, dislocation and grain boundary amelioration, ...) will be reviewed and applied to the experiment data.

9:00 AM L4.2

EFFECT OF GRAIN SIZE ON ANELASTIC BEHAVIOR OF PURE ALUMINUM AND COPPER MICRO-WIRES. P.A. Elders, R.P. Vinci, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA.

It is well known that as material dimensions become smaller, the physical mechanisms for deformation and failure are sometimes unlike those of bulk materials. One other prominent difference between the mechanical behavior of the micro-world and macro-world is the apparent elastic modulus. It has been shown that modulus values determined by microtensile testing can depend strongly on strain rate. This is likely to be due to mesoscopic mechanisms possibly related to grain boundary sliding (GBS) or dislocation motion. In order to determine the role that grain boundaries play in anelastic relaxation of thin films and small-scale structures, we have performed uniaxial stress relaxation and anelastic reloading tests on 98.99% pure 15µm diameter Al and Cu micro-wires and lightly tempered and slightly annealed micro-wires. Effective elastic moduli of the Al and Cu micro-wires were measured by microtensile tests at various strain rates. Back-scattering electron scattering microscopy (FIBSEM) was used for imaging grain sizes. Furthermore, electron-backscatter pattern (EBSP) techniques were used in determining grain orientation. Results from the received wires are compared with the annealed wires to illustrate the effects of grain size on effective modulus.

9:15 AM L4.3

EXPERIMENTAL SIMULATIONS OF SURFACE DEFORMATION DURING NANOINDENTATION. K.J. Van Vilet and S. Suresh, MIT, Cambridge, MA.

We present in-situ observations of the effects of crystal orientation,
A newly developed in situ Acoustic Emission (AE) monitoring technique is a synergy of highly advanced high-resolution contact characterization and advanced digital signal processing algorithms. High speed elastic and Surface Acoustic Wave (SAW) propagation phenomena are monitored simultaneously with quasi-static loading, by means of an AE sensor integrated into the nanindentation test instrument. Cracking at nano scale occurs due to loading as well as adhesive unloading of the tip and generates elastic and SAW propagating from the contact zone into the surrounding media. Nanometer scale crack nucleation and growth phenomena are identified by scanning transmitted acoustic waves and AE signals. Experimental quasi-static loading-unloading curves are correlated with the AE signal bursts. Advanced signal processing is an essential feature of the newly developed technique. Digital wavelet transforms and joint time-frequency analysis are used for AE signal decomposition and adaptive filtering. The proposed simultaneous AE monitoring technique targets brittle films and substrates.

Nanindentation test was performed for analysis of the structure polycrystalline films using the sensitive technique recently developed for the measurement of indentation hardness, Young’s modulus and elastic recovery of very thin films. A function of this system consists of indentation displacement with a resolution of several nanometers by a Vickers or Knoop indenter tip as a function of very light applied force. Resulting force-displacement curve indicates elastic and inelastic deformation behavior of the polycrystalline film. In addition, a nano-scale flow in the amorphous carbon and carbon nitride films is also detected by this technique. In this study, we formed amorphous polycrystalline films on the single crystalline silicon substrate and analyzed them using the nano-indentation technique. The creep behavior of the polycrystalline film will be described and discussed.

Nanindentation has been successfully used as a mechanical properties nanoscale microscope to characterize the elastic properties of materials. However, in an isotropic material, it is not possible to measure the two independent elastic constants by nanindentation. Normally, a value of Young’s modulus is determined using a measured value of Poisson’s ratio. It is possible to use a nanoscale microscope in its x-ray mode to measure the elastic constants of a surface. This too produces a composite measurement of the elastic properties, which can be represented in terms of Young’s modulus and Poisson’s ratio. By using both techniques in the same area, it is possible to make two independent measurements of the elastic properties and thus determine both Young’s modulus and Poisson’s ratio. This method has been used in well-characterized bulk materials (e.g. silicon films and stainless steel) to demonstrate that it produces consistent measurement. It has also been used to characterize thin films of TiN and TaN/AIN multilayers. These results show that, although a thin film there is need to improve the analysis of the mechanics, the combination of nanoindentation and acoustic microscopy shows promise.

With the wide application of microelectronics and MEMS devices, accurate measurement of the mechanical properties of thin metal films is becoming more and more important in this study, the elastic behavior of the substrate and its nanoscale contact analysis. We have developed a sample preparation technique that is
based on standard micromachining techniques and that is nearly independent of the material of interest. This technique has been used to fabricate the membranes of Cu and Al films without putting any constraints on the metal deposition processes. The films were deformed both under plane-strain and equibiaxial stress conditions. Yield stress, Young’s modulus, and residual stress were determined as a function of film thickness and deformation rate. Young’s modulus tends to be somewhat lower for bulk materials, a phenomenon that has been observed in microtensile tests as well, and that can be attributed to residual stress relaxation in the films.

SESSION 1.5 MECHANICAL PROPERTIES AND NANOSCALE TESTING II
Chaires L. B. Freund and Robert F. Cook
Tuesday afternoon, November 27, 2001
Room 304 (Hynes)

1:30 PM 1.5.1 MICROSTRUCTURAL INVESTIGATIONS OF FATIGUE EFFECTS IN THIN Cu FILMS. R. Schwager and O. Kraft, Max-Planck-Institut f. Metallforschung, Stuttgart, GERMANY.

Thin metal films must be able to withstand high stresses created during the deposition process, but also repeated stresses during operation due to mechanical vibrations, temperature changes, or high operating frequencies that are typical for small-scale devices. It is well established that bulk metals deform in bulk materials in well-defined crystallographic structures, such as veins, slip bands, and cell structures evolve, which typically extend over several micrometers. For (sub)micron dimensions the fatigue behavior is likely to be different than in bulk materials in the volume of material available is too small to allow the formation of these typical dislocation structures. Thin Cu films with a thickness between 0.2 and 3 μm on polycrystalline substrates were fatigue at a total strain amplitude of 0.5%. Fatigue was examined and transmission electron microscopy have been performed on the cyclically deformed samples. The appearance of surface extrusions is reminiscent of fatigue damage typically found in bulk single- and polycrystalline fcc metals. However, the extrusions are not related to a defined dislocation structure, only single dislocations have been observed. Furthermore, extrusions rather occur in [100] than in [111] oriented grains. A general observation on fatigue thin film specimens is the formation of voids as the films-substrate interface beneath extrusions, which is interpreted as the result of the annihilation of edge dislocations. In this paper an attempt will be made to describe the mechanisms leading to fatigue damage in thin films on the basis of the experimental findings and current understanding of thin film deformation and bulk fatigue.

2:00 PM 1.5.2 MECHANICAL PROPERTIES AND STRESSES OF THIN GOLD FILMS ON A SILICON SUBSTRATE. Changjin Xie, Dept. of Mechanical Engineering, Yale University, New Haven, CT; Richard Emery, Intel Corp., Chandler, AZ; Wei Tong, Dept. of Mechanical Engineering, Yale University, New Haven, CT.

Nanoindentation tests are now routinely used to evaluate the mechanical properties of ultrathin film coatings. As most of thin film coatings are highly textured and may have a significant level of internal stresses, it is necessary to determine the constraints imposed by the substrate during the film deposition process, there is a need to assess the effects of both material texture and internal stresses on the mechanical property data obtained by the nanoindentation. In this talk, we present a study of material characterization and mechanical property measurements of thin gold films deposited on a silicon substrate. Because the gold films are free of any surface oxidation, results of this study will provide a more direct comparative evaluation on the nanoindentation data and the tensile test data. Specifically, both microtensile tests of free-standing thin gold films and nanoindentation tests of the thin gold films attached to the silicon substrate are performed in this study. The microtensile and microtensile of the gold films are characterized by means of X-ray diffraction and Orientation Imaging Microscopy (OIM), respectively. In addition, the internal stresses in the gold films attached to the silicon substrate are measured by the X-ray diffraction as well. Crystal plasticity finite element analyses of both microtensile test and nanoindentation tests are then carried out. The crystal plasticity model of the gold films is calibrated via the simulation of the microtensile tests and the nanoindentation tests. The nanoindentation tests of the gold films on a silicon substrate are finally analyzed to assess the effects of both the film texture and internal stresses on the nanoindentation numbers.

2:15 PM 1.5.3 EFFECT OF MICROSTRUCTURE ON MECHANICAL PROPERTIES OF AIN THIN FILMS. Shoichi Miyake, Masami Aono, Nobuhiko Kanazawa, Yoshifumi Watanabe, National Defense Academy, Univ. of Aichi, Japan.

Aluminum nitride (AlN) thin films have been synthesized on Si (100) wafers kept at near room temperature, by ion-beam assisted deposition (IBAD) method. The kinetic energy of the nitrogen ion beam was fixed at 0.2 keV and 1.5 keV under the constant current density. The former produces a columnar structure film of 300 nm in thickness, the ratio of the maximum penetration depth to the film thickness is about 20%, and the microhardness is about 12 GPa, which is close to the hardness of the silicon. When the film thickness increases to 600 nm, the ratio R is reduced to 10% and the microhardness is found to be about 22 GPa. For the granular structure film of 700 nm in thickness, the ratio R is about 13% and the microhardness is found to be about 14 GPa. These results reveal that the microhardness of the AlN film itself can be evaluated when the ratio R is about 10%, and the microhardness strongly depends on the film microstructure, which can be controlled by regulating the nitrogen ion beam energy. Relationships between the film microstructure, mechanical properties and residual stresses in IBAD AlN films will be discussed.

2:30 PM 1.5.4 DYNAMIC OBSERVATION OF DISLOCATION FREE PLASTIC DEFORMATION IN FCC METAL THIN FILMS. Yosikiko Mitakusa, Kazunari Yamauchi, Masato Komatsu, Michio Kitami, Advanced Materials Frontier Research Center, AIST-Ultra-high plastic deformation, Hiroshima Institute of Technology, Hiroshima, JAPAN.

In the fractured tip of ductile fcc metal films anomalous high density of point defect clusters has been confirmed to be produced by plastic deformation without dislocations. To clarify the mechanism whereby such unusual microstructure is introduced, a dynamic observation of the microstructural evolution during deformation has been carried out in a transmission electron microscope with a video recording system, at 298K with a crosshead speed of 0.01 μm/s. Specimens were ribbons of fcc metals (Au, Al, Ni) whose thickness was 10μm. The initial thickness was too thick for 200kV transmission electron microscopy, but decreased during elongation, and finally became thin enough for observation. In the thinned part a high density of point defect clusters had already existed, which were used as markers to measure subsequent local deformation. With further deformation some new point defect clusters were produced in local heavy deforming part; however, during this heavy deformation no dislocation movement was confirmed, despite observed under two-beam dark field image condition, in which dislocations should be observed if any. The heavy deformed part without dislocation finally ruptured, and the ruptured edge shrunk, which indicated some elastic deformation had been involved in the heavy deformation. The elastic deformation detected was 13%, which corresponds to an internal stress of about 10 GPa. These results indicate that deformation takes place without dislocations in extremely high internal stress condition. The dislocation free plastic deformation accompanies with both production and annihilation of point defect clusters. During these events a characteristic diffraction image contrast change was confirmed; equal thickness fringes became faint temporarily. The disappearance of equal thickness fringes indicates a temporal disturbance of the crystal structure, which leads to a statement that a stability of crystal structure under extremely high internal stress will be a key for the dislocation free plastic deformation.

2:45 PM 1.5.5 VOID INTERGRANULAR MOTION UNDER THE ACTION OF ELECTROMIGRATION FORCES IN THIN FILM INTERCONNECTS WITH BAMBOO STRUCTURE. Ersin Emre Ozen, Turk Omer Ogurcanli, Middle East Technical University, Dept. of Metallurgical and Materials Engineering, Ankara, TURKEY.

In these studies the rigorous formulation of the internal entropy production, and the generalized forces and conjugate fluxes associated with the virtual displacement of a triple junction are developed. A well-posed moving boundary value problem describing the kinetics of interphaseal layers and surfaces is obtained for the ordinary points as well as for the triple junction. Extensive computer simulations are performed on the void configurational evolution during the intergranular motion, under the actions of capillary and electromigration forces in thin film metallic interconnects with bamboo structure. Very rich, and also novel void grain-boundary interaction morphologies such as penetration and detropping, fragmentation and daughter void formation are observed, at high normalized electromigration intensities. Singular point associated with a triple junction is treated rather rigorously by using microdiscrete (straight) interfaceal elements as convenient mathematical tools as proposed by Ogurcanli. The generalized forces and conjugate fluxes associated with
the triple junction are obtained in terms of the asymmetric dihedral angles and the specific Gibbs free energies related to the void surface energy and the void interfacial energy. The velocities of the triple junction, longitudinal, and transverse are also included as a by-product of this treatment. Mathematical model of the void shape evolution dynamics in the presence of the void-grain boundary interaction is developed by utilizing the results of our theory on the common points at the void surface layer.

3:20 PM L5.6
MICROMECHANICAL TESTING OF FREE-STANDING THIN FILMS FOR MEMS APPLICATIONS
John C. Brawner, Ping Zhang, Stanford University, Stanford, CA; Hoon-Jeong Lee, Agee, Inc., Reading, PA.

In Micro Electro Mechanical Systems (MEMS) devices, many of the active components exist in the form of free-standing thin films. Such components are constantly in motion under varying actuation conditions and the mechanical properties of free-standing thin films are therefore important for the design of MEMS devices, as well as for predicting their mechanical performance and reliability. The mechanical behavior of a free-standing thin film is expected to be different from that of bulk material or a conventional thin film on substrate. Although thin films on substrates have been extensively studied, knowledge of free-standing thin films was generally not available until the advent of micromachining techniques and is yet to be further explored. We have developed a dedicated simple fabrication process to produce free-standing thin films of different materials and different thicknesses. We have built a custom-designed micromechanical test apparatus with a load resolution of 10 nN and a displacement resolution of approximately 10 nm. We use Transmission Electron Microscopy (TEM) to reveal the microstructural features of the samples both before and after testing to expand our understanding of the mechanical properties of the thin film materials under investigation. We study the stress-strain response of pure Al and Al-Ti alloy free-standing thin films for both monotonic loading and preliminary cyclic loading. Monotonic tests include micromechanical tests and stress relaxation tests. From strength tests, we examine the results with respect to those of bulk materials and thin films adhered to substrates. From relaxation tests, we study the effect of straining in conjunction with a proposed anelastic model. The unique features of our free-standing Al thin films include high yield strength, small ductility, and a negative slope in the flow stress. These are found to be very sensitive to grain size and uniformity. From TEM analyses of deformed specimens, we also find evidence of inhomogeneous deformation, including the development of localized thinning, which we believe accounts for the negative slope and the small ductility.

4:00 PM L5.7
ELASTIC PROPERTIES OF THIN FILM MATERIALS FOR COPPER INTERCONNECT TECHNOLOGY CHARACTERIZED WITH OPTOACOUSTIC TECHNIQUE
A.A. Menashy, M. Gosten, P. Kravetz and R. Botolotsky, Philips Analytical, Natick, MA.

The continuous drive for increased speed and packing density of integrated circuitry with metal interconnection technologies is in metal interconnect fabrication technology, where copper and low-k dielectrics are replacing traditional Al/SiO₂ structures. Knowledge of the mechanical properties of materials comprising an interconnect structure is becoming increasingly important for the new generation of metal interconnects. While significant advances with chemical mechanical polishing (CMP) techniques have lessened the modulus than SiO₂ must provide enough structural support for heavy Cu wires during critical process steps such as chemical mechanical polishing. Elastic properties are also of prime importance for laser/acoustic techniques introduced for interconnect fabrication process control. In this work, a laser optoacoustic technique called Impulsive Stimulated Thermal Scattering (ISTTS) implemented in a commercially available instrument was used to characterize elastic properties (i.e. elastic modulus or, alternatively, acoustic velocities) of dielectrics (both traditional such as thermally grown and CVD SiO₂ and new prospective low-k ones), barrier materials such as Ta/TaN and copper thin films. Acoustic wave propagation in complex multi-layer structures is simulated in order to use the optoacoustic instrument for Cu thickness control on product wafers. Related issues such as effective elastic constants of dense Cu/dielectric structures and the applications of the technique to the control of the mechanical integrity of such structures are also discussed.

4:15 PM L5.8
SPHERICAL INDENTATION TESTING OF POLYMERIC SURFACES AT VARIOUS TEMPERATURES
Vincent Jardret, MTS Nano Instruments Innovation Center, Oak Ridge, TN; Pierre Morel, Nicola Conte, University of Tennessee, Knoxville, TN.

Contact mechanics for indentation testing with spherical indenters is very attractive. Numerous projects have established equations to define strain and stress distribution in order to obtain stress-strain relationship from a single indentation experiment. Also a large number of metal indentation experiments having objective of estimating the yield point. The subject of this work is to analyze the behavior of various polymeric materials during spherical indentation testing at various temperatures in order to observe the relationship between the indentation behavior and temperature. The evaluation of stress-strain behavior of the same materials as a function of temperature. Thermal effects on the indentation data are used to understand the actual effects of the mechanical properties on the indentation behavior. In addition to the load displacement and frequency specific stiffness information, topographic analysis of the residual indent test is used to accurately estimate the contact area. Therefore validate the indentation models for contact depth calculations using spherical indentation. Results presented in this article include spherical indentation data obtained on PMMA and Polycarbonate over a range of temperature from 5°C to 108°C.

4:30 PM L5.9
MECHANICS OF INDUSTRON-OXIDE FILMS ON POLYMER SUBSTRATES WITH ORGANIC BUFFER LAYER
Sung Kyu Park, Jeong In Han, Chan Jee Lee, Won Keun Kim and Min Gi Korok, Korea Electronics Technology Institute, Pyungtiak, Kyunggi, KOREA.

The characteristics such as electro-mechanical and optical properties of indium tin oxide films deposited on polymer substrates were examined. The materials of substrates are polyethylene naphthalate (PEN), polycarbonate (PC) and polyethylene terephthalate (PET), which have glass barrier layer and anti-ghost coating for organic electron devices. The experiments were performed with ellipsometric sputtering using a special instrument and novel stepped heating process to reduce thermal stress of the substrates. Furthermore, we simulated the mechanical behavior of the organic buffer layer with respect to Stoney formula and experienced the changes of strains as the material of buffer layers and substrate thickness. X-ray diffraction observations, measurement of electrical resistivity, scanning electron microscope observations and nano-indentation measurement were performed for the investigation of micro-structure and electro-mechanical properties. In addition to the investigation, we discussed the conduction mechanism of ITO films related to the residual stress. Consequently, based on the investigation of the electro-mechanical characteristics and organic buffer layer, more reliable ITO films were deposited on polymer substrates with sheet resistance of 25-28 Ω/square, transmittance of 85% and enhanced mechanical properties.

4:45 PM L5.10
TRIBOLOGICAL BEHAVIOR OF NANOCOMPOSITE DIAMONDLIKE CARBON FILMS
Erjia Liu, Nanyang Technological University, School of Mechanical and Production Engineering, Centre for Mechanics of Microsystems, SINGAPORE; Beng Kang Tay, Nanyang Technological University, School of Electrical and Electronic Engineering, SINGAPORE; Jie Hong Shi, Data Storage Institute, SINGAPORE; Xu Shi, Nanyang Technological University, School of Electrical and Electronic Engineering, SINGAPORE.

Tetrahedral Amorphous Carbon (ta-C) contains a large percentage of sp³ carbon bonding. Filtered cathodic vacuum arc (FCVA) technique is an efficient method of producing high quality ta-C films. However, a relatively high internal stress develops in ta-C films with a high sp³ content, which limits their applications for wear protection. In this study, Al containing ta-C films are produced using FCVA. The tribological behavior of the films is measured using ball-on-disk tribometer and micro-scratch tester. The wear rate, friction coefficient, and critical load are determined correspondingly. In the ball-on-disk testing, different loads are applied to the counterface which is a sapphire ball. All the tests are performed at controlled relative humidity and room temperature (about 35°C). It was noted that the friction coefficient of ta-C/Al films increases before the beginning of the test reaching a peak value of about 0.25. After the peak, the friction coefficient drops until reaching a steady state value. The test shows that the steady state friction coefficient maintains until the film is worn through and the friction coefficient suddenly increases to a high value up to 0.46. The original surface roughness of the counterface, surface smoothing due to successive wear, and wear debris produced during the testing are all responsible for the tribological behavior of ta-C/Al films. The thickness of the ta-C films is determined with bending plate method. The internal stress is related to the structural properties of the films. The pure carbon ta-C films generally contain a relatively high residual compressive stress, which is related to its sp³ content, which limits their applications. For the Al containing ta-C film, the stress reduction is significant with increase of aluminum content in the film. A decrease of the mechanical properties of the ta-C/Al nanocomposite films has been noticed with the decrease of the internal stress in the films.
DETERMINING THIN FILM PLASTIC PROPERTIES BY THE BULGE TEST. Z.L. Zhuang, SINTEF Materials Technology, Trondheim, NORWAY; J. Vissers, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

The bulge test has been commonly applied to determine the elastic properties of thin films. However, relative few studies have been carried out on determining thin film plastic properties. Understanding and determining the plastic properties including yield strength, hardening modulus, strain for the metal films are very important for modern technological applications. In this paper, we have analyzed various effects that influence the relation between the plastic stress-strain curve of a material and the pressure-displacement curve measured in a bulge test. The effects include sample aspect ratio, the hardening exponent of the material, sample compliance, and residual stress. Residual stress up to initial yielding has been considered. It has been found that the average true stress strain curve determined from the pressure-displacement curve is in good agreement with the stress-strain curve of the material as long as the thickness change is taken into account, especially when the true strain is larger than about 1%. For rectangular thin films, an instability point, which marks the shift from linear to non-linear stretching, occurs at a strain close to its hardening exponent. This strain is in general much smaller than the necking strain from classical analyses. A closed formula for calculating strain hardening is given. The results of this study have been applied to determine the plastic properties of Cu and Al thin films on silicon substrates which will be presented in a separate study.

STRESS EVOLUTION AND PHASE FORMATION DURING DEPOSITION AND DURING ANNEALING OF Ti$_2$Al$_2$O$_7$ ALLOY FILMS. Stefan Lackner and Reinhold Abelnann, Institute of Physical Chemistry, University of Innsbruck, AUSTRIA.

In continuation of earlier work, the growth stress of Ti$_2$Al$_2$O$_7$-alloy films was investigated under UHV-conditions using a channel beam technique. The alloy films were deposited on 10 mm thick Al$_2$O$_3$-substrate films and the deposition temperature was varied from RT to 500°C. The evolution of the microstructure and the phase formation was investigated by TEM/EDS and XRD-experiments. Alloy films with Al-content below 50% show stable stress versus thickness curves comparable to one component systems. Films with high Al-content, however, exhibit an average formation of growth stress during their deposition. The origin of these irregularities, which become more pronounced with increasing substrate temperature, was investigated in detail. For this purpose, we have performed annealing experiments of 150 nm thick Ti$_2$Al$_2$O$_7$-alloy films, previously deposited under UHV-conditions using a channel beam technique. The alloy films were deposited on 10 mm thick Al$_2$O$_3$-substrate films and the deposition temperature was varied from RT to 500°C. The evolution of the microstructure and the phase formation was investigated by TEM/EDS and XRD-experiments. In order to clarify the origin of the above mentioned irregularities in the growth stress, we have also measured the growth stress of a 150nm thick Ti/Al multilayer film deposited at 600°C. This multilayer experiment shows large tensile stress contributions during deposition and compressive stress contributions of comparable magnitude during the Al on Ti deposition. The stress at interdiffusion and for delayed Al deposition is assumed to be the reason for these growth irregularities. The average stress during deposition of 24 Ti/Al-multilayers is compressive and its magnitude is larger than that built up in a film with the same composition but deposited by simultaneous deposition of the alloy components at this substrate temperature.

ON VISCOS CREEP IN METALLIC WIRES AT ELEVATED TEMPERATURES AND LOW STRESSES. Piersale Floha, Brno University of Technology, Faculty of Chemistry, Brno, CZECH REPUBLIC; Libor Kloc, Václav Klenička, Institute of Physics of Materials CAS, Brno, CZECH REPUBLIC.

Vicious creep behaviour of several metals and alloys was investigated at the temperatures close to one half of the absolute melting point and very low stresses using the technique of helicoidal specimens. Due to extremely high sensitivity the technique provides a unique tool for measurement of very low creep strains in acceptable time of the experiment. Helicoid spring specimens were made of wires of either circular or square cross section. The stress distribution along the wire radius (caused by shear stress loading) and threshold stresses were taken into account, as well as the influence of the surface layer loaded by minimum stress. The experimental results were interpreted as combinations of Hall-Petch creep and/or creep-related diffusion. Some data are in very good agreement with Coble theory especially those obtained on some fine grained materials. This dependence is related by large data scattering for coarse grained materials. Some authors dispute the role or even the very existence of diffusional creep and offer other explanations. There are many theories trying to describe the dependence creep mechanism, but none of them is capable to explain all observed properties. The observed effects which cannot be explained by the current theories are discussed (large scatter of creep rates obtained for coarse grain materials, creep rates much higher than those predicted by the diffusional creep law in some materials, variation stage duration independent from the total creep strain dependent on temperature). Despite the problems in theoretical description, the experiment show clearly that the viscous creep regime must be considered as an important behavior of structural materials at conditions of engineering practice.

A STUDY ON THE BEHAVIOR OF RESIDUAL STRESS WITH WATER ABSORPTION OF S OF THIN FILMS DEPOSITED BY ELECTRON CYCLOTRON RESONANCE PLASMA ENHANCED CHEMICAL VAPOR DEPOSITION METHOD. S.P. Kim, S.K. Choi, Korea Advanced Institute of Science and Technology, Department of METE, Daejon, KOREA; Yongsoon Park, Bubh Chang, Samsung Advanced Institute of Technology, Materials and Device Laboratory, Suwon, KOREA.

Fluorinated silicon oxide (SiOF) films were deposited by electron cyclotron resonance plasma enhanced chemical vapor deposition (ECR-PECVD). The behavior of residual stress was studied with water absorption. SiOF film showed compressive stress after deposition. The compressive stress increased after the exposure to room air. Fourier transformed infrared (FTIR) spectroscopy analysis was carried out to study the water absorption. However, the change of chemical bonding structure was not observed in this study. After the exposure to room air, SiOF films were kept in dry nitrogen. The residual stress results from the initial value after 1 week. Considering the results of the residual stress and FTIR analysis, it is supposed that the water absorption in this study occurs entirely by physical adsorption of H$_2$O molecules to Si-F bonds on the surface.

A NEW FORCE-INDENTATION MODEL FOR CONTINUOUS INDENTATION OF BI-LAYER MATERIALS. Ali Nagehi, Rechdi El Ahbi, Gérard Maussion, Olivier Bartier, Rennes F University, LARMAR, Rennes, FRANCE.

An elastic-plastic spherical micro-indentation study of bilayer (film-substrate) materials is made by experimental and finite element methods. Indentation simulations are done with a sphere of 1.5875 mm radius. Various surface-substrate film yield stress ratios $\beta$ (1 $< \beta < 6$) and various film hardening exponents $n (1 < n < 5)$ have been considered in modeling. The thin film thicknesses vary from 50 to 400 μm. Based on finite element results, a mixture law is proposed for bilayer materials indentation. This law takes into account the influence of film and substrate mechanical properties and on the applied force. Finite element results show that this parameter depends on dissipated plastic energy induced by indentation in the film and in the substrate of the bilayer. Uniaxialized plastic strain and dissipated plastic energy in a thin film and in the substrate, a new applied force-indentation depth of bilayers is obtained. Comparison of this new relation with experimental results, shows the capabilities of the model for simulating the behavior of bilayer materials under indentation. The results from this model are compared with those of experiment to show the capability of the model.

DEPOSITION OF TiN THIN FILMS ON Si(100) BY UNBALANCE MAGNETRON SPUTTERING. Wei-Jun Chou, Ge-Ping Yu, and Jia-Hong Huang, Department of Engineering System Science, National Taiwan Ocean University, Hsin Chu, TAIWAN.

Titanium nitride (TiN) films were deposited on Si(100) substrate using an unbalance magnetron sputtering (UMS) of stress and the thickness of the TiN film was controlled and, from our previous study, the nitrogen partial pressure was selected as the variable parameter.
The purpose of this study is to investigate the effect of nitrogen partial pressure on the structure and properties of TiN films. After deposition, the film's structure was characterized by X-ray diffraction (XRD), cross-sectional transmission electron microscopy (XTEM), and field-emission-gun scanning electron microscopy (FEG-SEM). Nitrogen content in the thin films was determined using nitrogen content using X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectrometry (RBS). The resistivity of the TiN film was measured by a four-point-probe. The hardness of the thin films was determined using nanoindentation tests. An atomic force microscope (AFM) was used to measure the roughness of the thin films. The results showed that (111) was the dominant preferred orientation in the TiN films for most of the deposition conditions. Hardness values were about 30 GPa. Hardness can be correlated to the (111) preferred orientation of the TiN film. The lowest resistivity of the TiN film was about 60 cm.

L6.7 ORIGIN OF THE Ag/Ni (111) INTERFACE STRESS.
Dillon D. Feng, Harvard Univ. Division of Engineering and Applied Sciences, Cambridge, MA; Dennis B. McWhan, National Synchrotron Light Source, Upton, NY; Frans Spaepen, Harvard Univ. Division of Engineering and Applied Sciences, Cambridge, MA.

Ag/Ni multilayer films with bilayer thicknesses between 3.2 nm and 22.8 nm were grown by ion beam sputtering at near liquid-nitrogen temperatures. X-ray measurements of the in-plane and out-of-plane lattice parameters in substrate-supported films were made at the National Synchrotron Light Source, and the elastic strains were calculated using the bulk lattice parameters of Ag and Ni. The Ag/Ni (111) interface stress was fit based on the dependence of the difference between the substrate-supported stress and the average volume stress on the number of interfaces per unit volume. Between the bilayer thicknesses of 3.2 nm and 11.3 nm, the multilayers have an interface stress of \( \sigma = 2.17 \pm 0.15 \) Nm, which is in close agreement with previous results. The origin of this compressive interface stress is studied using a systematic method of determining the interfacial structure based on the fitting of low and high-angle x-ray spectra, as well as the analysis of Rutherford backscattering data.

L6.8 CONSTITUTIVE RESPONSE OF PASSIVATED COPPER FILMS: EXPERIMENTS, ANALYSES AND IMPLICATIONS.
Y.-L. Shen, Univ of New Mexico, Dept of Mechanical Engineering, Albuquerque, NM; U. Ramamurty, Indian Institute of Science, Dept of Metallurgy, Bangalore, India.

The constitutive behavior of passivated copper films is studied. Stresses in copper films of thicknesses 1000 nm, 400 nm and 40 nm, passivated with silicon oxide on a quartz or silicon substrate, were measured using the curvature method. The thermal cyclic sapphire a temperature range from 196 to 600°C. It is seen that the stress relaxation at high temperatures normally found in unpassivated films is not present in passivated films. The copper film did not show any rate-dependent effect over a range of heating/cooling rates from 5 to 25°C/min. Further analysis showed that the significant strain hardening exists during the course of thermal loading. In particular, the measured stress-strain response can only be fitted with a kinematic hardening model, if a simple constitutive law within the continuum plasticity framework is to be used. This is drastically different from the unpassivated and passivated aluminum films and unpassivated copper films. Implications to stress modeling of copper interconnects in actual devices will be presented, and the possible microstructural mechanisms responsible for this unique feature will be discussed. Isothermal stress relaxation at fixed temperatures between 300 and 400°C was also measured. Without the influence of continuous thermal loading and strain hardening, the relaxation is shown to follow a power law-type behavior with high stress exponent and activation energy, compared to bulk polycrystalline copper. The possible causes will be addressed.

L6.9 IN SITU MEASUREMENTS OF THE EFFECT OF THE DEPOSITION RATE ON THE EVOLUTION OF STRESS IN METALLIC THIN FILMS.
Andrea Del Vecchio, Frans Spaepen, Harvard Univ. Division of Engineering and Applied Sciences, Cambridge, MA.

In situ stress measurements on polycrystalline metallic thin films show a characteristic evolution of the stress. For materials with a high surface mobility, such as copper and silver at room temperature, there are three successive regimes: (i) an initial compressive stress that results from a Laplace-type pressure in isolated islands, induced by the surface stress; (ii) a tensile increase that results from densification, or "rippling" up of surfaces into grain boundaries upon coalescence of islands; and (iii) an asymptotic compressive stress that is as yet unexplained. In addition, there is a post-deposition change in the stress of the film that is always tensile and reversible. In this study, possible effects of the deposition rate on the location and magnitude of the tensile maximum, on the asymptotic compressive slope, and on the magnitude of the post-deposition stress change have been investigated using an in-situ wafer curvature apparatus during deposition in ultrahigh vacuum. Although deposition rate was varied, no post-deposition rate was found. This investigation provides a test of the models for the components of the intrinsic stress.

L6.10 FORMATION OF INTERMETALLIC PHASES AT THE METAL FILM-SILICON INTERFACES.
Abuz Ozenbas, Huseyin Guler, Dept. of Metallurgical and Materials Eng., Middle East Technical University, Ankara, Turkey.

This study presents the formation mechanisms of intermetallics which formed at the metal thin film/silicon substrate interfaces. In the experimental part of the work, six systems, Al/Si, Al/5%Fe/Si, Al/5%Cr/Si, Al/5%Ni/Si, Cu/Si, and Cu/5%Ni/Si were studied. After vacuum vapor deposition of the films, they were annealed at different temperatures and time intervals. X-ray, SEM and EDS studies showed that several intermetallics at the interfaces formed sequentially on the contrary of intermetallics which formed synchronous in bulk materials. Using this data, a model will be given about the formation of intermetallic phases obtained. These studies are important to ensure the reliability of these surface coatings on the long term, especially in electronics industry. No intermetallics were observed in Al/Si system. Silicides, Fe2Al5Si2O4 and Fe2Al5SiO4 were detected in Al/5%Fe/Si system. In spite of the presence of two intermetallic phases, Ni4Al5Si3O8 and [AlSi]Ni5C, in Al/5%Ni/Si system, only one intermetallic phase (Cu2SiAl3)4Si was formed in Al/5%Ni/Cr/Si system. Cu/5%Si system, CuSiAl3Si2, CuSiAl2Si were observed. Finally in Cu/5%Ni/Ni system, Cu2Al5Si3, CuAl5Si were formed. In most cases, t1/2 (annealing time) dependence of the phase growth, parabolic growth, has been found, indicating a diffusion limited growth. In some cases a linear dependence on time was found, suggesting an interface rate-controlled interaction. As a model, growth kinetics of Cu2Al5Si3 was investigated with and without Ni incorporation to understand the impingement. It was concluded that reaction rate constants increased and activation energy decreased with impurity content and growth rate was diffusion limited which obeys the parabolic growth rate.

L6.11 MECHANICAL BEHAVIOR OF INTERCONNECT ALUMINUM ALLOY PRODUCED IN A COMMERCIAL FABRICATION FACILITY.
David T. Read, Yu-Wen Cheng and J. David McEvoy, National Institute of Standards and Technology, Materials Reliability Division, Boulder, CO.

Mechanical characterization of many thin film materials has been reported. The vast majority of these films were produced in laboratories rather than in commercial production facilities. Here, we report tensile test results for material produced in a commercial fabrication facility, obtained through the MDSH program. Samples of two interconnect levels, designated M1 and M2. Because of restrictions inherent in the commercial process, the specimens tested were M2 and M1/M2 composite. The as-received chips had M1 and M2 interconnects patterned in the shape of tensile specimens deposited directly on silicon substrate. Silicon beneath and near the tensile specimen was removed to a depth of about 50 μm by etching with XeF2. Tensile tests were performed using a recently developed force probe technique. The specimen thicknesses were 1.5 and 2.4 μm for the M2 and M1/M2 specimens, respectively. The specimen gauge sections were 10 μm wide and 180 μm long. On one chip, the average values for 12 specimens of M2 for yield strength and ultimate strength were 67 and 81 MPa, with an elongation of 1.4%. The five M1/M2 specimens had an average yield strength and ultimate strength of 63 and 71 MPa, with an average elongation of 1.8%. For both types, the standard deviations of the strength values were low, but the elongations had a high standard deviation. The strength and ductility of both the M2 and the M1/M2 specimens were low compared to results for pure material. Similar strength values were observed in tests of three specimens on a second chip from the same production run, but repeated loading-relaxing runs on this set of specimens did not allow extraction of yield strength and elongation values. The apparent Young’s modulus was equal to or higher than the bulk polycrystalline value.

L6.12 Abstract Withdrawn.

L6.13 Abstract Withdrawn.

The residual stress induced in a P-aluminide bond coat on a single crystal super-alloy has been measured. Absent the ability to use diffraction approaches, since the lattice parameters are unknown, the ‘wafer’ curvature method was used. It has been confirmed that the substrate thickness is systematically reduced by mechanical thinning. It was also required that curvature measurements be made with the bond coat present and after it had been removed by thinning. This approach revealed that the bond coat is in residual tension, 140 MPa, consistent with its thermal expansion coefficient, relative to that of the substrate.

L6.15 MECHANISTIC UNDERSTANDING OF THE STRESS-INDUCED FAILURE OF Si3N4 METAL-INSULATOR-METAL CAPACITORS. Dongwook Shih, Dongki Moon, Kye-Hwan Shin and Jin-Yeong Kang, Compound Semiconductor Research Department, Microelectronics Technology Laboratory, Electronics and Telecommunications Research Institute, Taejon, KOREA.

We investigated the failure of Si3N4 metal-insulator-metal (MIM) capacitors at a microstructural and mechanistic point of view using cross-sectional transmission electron microscopy (XTEM) and stress analysis of the interface, respectively. The Si3N4 layers with both 300 nm (Specimen-A), 500 nm (Specimen-B), and 700 mm-thick (Specimen-C) were deposited by plasma enhanced chemical vapor deposition (PECVD) at 400°C, corresponding to the area-normalized capacitance of 2.2, 1.3, and 0.9 pF/mm², respectively. The metal electrodes of TiN/Ti were deposited by reactive sputtering at 400°C with TiN diffusion barriers, finally having a multilayer structure of Al/TiN/Si3N4/Ti/TiN/Al in bottom-up direction on thermally grown SiO2 over p-type silicon wafers. It was found that microvoids had occurred at the Si3N4 layer and then propagated into the adjacent metal layers in Specimen-A whereas no microvoid was observed in the others. In consistence with the microstructural difference, Specimen-A showed very low breakdown voltage of 3 Volts comparing with Specimen-B and -C of which breakdown voltages were 14 and 24 volts, respectively. The microvoids of the MIM capacitor was caused by thermal-induced residual stress embedded into the constant layers during the cooling down of the thin films from the deposition temperature to room temperature. According to the stress analysis, the stress state of Specimen-A was relaxed from compressive stress of 300 MPa to tensile one of 390 MPa across the boundary of the SiN4 and Ti layers. Thus such stress state comes to exert large bending moment on the entire stacking structure of the MIM capacitor and in turn to initiate microvoids in the brittle Si3N4 layer provided the stress goes beyond the threshold value. It was noted that the thickness of the interface between the constant layers should be taken into account for the design of MIM capacitors.

L6.16 RELATIONSHIP BETWEEN ELASTIC MODULUS AND PORE FORMATION MODE IN LOW K INTERMEDIATE DIELECTRICS FOR ULSI APPLICATION BY A FINITE ELEMENT METHOD. Nobuaki Aji, Tomoya Fukuoka, Hiroshi Yasumoto, and Hiroshi Yamazaki, Electronics and Telecommunications Research Institute, Semiconductor Technology Research Department, Association of Super-Advanced Electronics Technologies, Yokohama, JAPAN.

With shrinking of device size of ULSI, degradarion of device performances are caused by an increase of RC decay due to intradie capacitance. In order to reduce capacitance between metal lines of ULSI multilevel interconnects, it is indispensable to introduce low-k intermetal dielectric for the uppermost interconnect layer. Low-k dielectric material is in a porous material. A pore inclusion into a dielectric film decreases its density, and reduces its dielectric constant drastically. However it causes its poor mechanical strength simultaneously. Poor mechanical strength causes difficulties in interconnect formation due to high shrink stress in the film. We investigated low-k dielectric multilevel interconnect due to many kinds of stresses induced by chip fabrication, such as metal CMPs, thermal cycles, and wafer bonding, etc. Then, we believe that a degradation of mechanical strength restricts lowering dielectric constant of films. In this paper, we focus the relation between elastic modulus of low-k films and pore formation mode. We evaluated elastic modulus of various porous and nonporous intermetal dielectric films by nanoindentation method. The density and thickness of films were obtained by means of GIXS. Elastic modulus of various low-k films shows a linear dependence on the film density for porous and nonporous inorganic low-k materials, respectively. We have studied the effect of pore aggregation on elastic modulus of thin films by a finite element method (FEM) using the 3-dimensional random pore generation model. FEM results of elastic modulus, which were calculated for 3-dimensional random pore model, extremely well fit with the experimental data obtained by a nanoindentation method. From our model, we defined the pore aggregation ratio (Apor), defined by area ratio of generated pore to unit pore, can be obtained. Logarithmic values of Apor linearly depend on logarithmic values of relative density of low-k dielectrics. And elastic modulus of low-k dielectrics exhibits strong dependence upon pore aggregation mode. From the results, it is indicated that the dependency of elastic modulus upon density can be described by a function of Apor. In order to develop some robust low-k dielectrics by using this quantification method, it is required that controlling pore aggregation mode is essential. This work was performed under the management of ASET in New Energy and Industrial Technology Development Organization (NEDO).

L6.17 STRESS MEASUREMENT IN Pt THIN FILMS. B.J. Park, Youngman Kim, Chonnam National Univ., Dept. of Materials Science and Engineering, Gwangju, KOREA.

Any type of multi-layer structure results in the difference in thermal expansion coefficient and elastic modulus between layers due to the artificial bonding of dissimilar materials. Inevitably the thermal stress and residual stress between layers arise from the bonding. In turn the stresses may cause dimensional instability of parts, such as twisting and bending, which can trouble the engineering products during fabrication and even in service. In this study, platinum films have been prepared by RF magnetron sputtering at different processing variables, such as sputtering pressure and RF power. The influence of processing variables on the microstructures and residual stresses in the films has been studied. Relations between microstructures and residual stress changes in Pt thin films were also investigated in terms of heat treatment.

L6.18 INTRINSIC STRESS IN SUPERIONIC α-PhSn4 THIN LAYERS. AND ITS EVOLUTION THROUGH THE α → β-PhSn4 PHASE TRANSITION. Georges Desal, J.M. Gervais and M. Cesbron, Universite Concordia, Dept. of Chemistry and Biochemistry, Laboratory of Solid State Chemistry and Microscopy, Laboratories for Inorganic Materials, Montreal, Quebec, CANADA.

PhSn4 is the high performance fluorode-ionic conductor. The exceptionally high mobility of the fluoride ions in α-PhSn4 has been ascribed to the disturbance brought about by the replacement of half the Pt in the Pt-hexagonal structure of α-PhSn4 by covalently bonded Sn(II), and to the presence of a vacancy fluorode-ionic layer between two adjacent tin layers. However, tin(II) lone pairs, that are strongly stereoreactive, occupy the vacant anionic plane and keep the number of interstitial sites reasonably low. The tin(II) lone pairs being clustered in planes, create highly efficient cleavage planes parallel to one another in polycrystalline samples. PhSn4 can be prepared by several methods, and it undergoes a large number of order/disorder phase transitions, making it one of the most complex materials known. When prepared by the addition of lead(II) nitrate to a solution of SnF2, α-PhSn4 precipitates, and when recrystallized, the size and quality of the membranes-like crystals is improved. In addition, the thin sheets are the subject of an intense intrinsic stress generated by the ferromagnetic properties of the material. α-PhSn4 has a tetragonal unit-cell, however, just a slight modification of the synthetic procedure produces an orthorhombic distortion, with a peculiar variation of the intrinsic stress.


Metal oxides are known to absorb reversibly a large quantity of protons or hydroxyl ions (protons, Li ions) and this property is widely used in applications such as rechargeable Li-ion batteries, electronic devices, and electrochemical capacitors. The intercalated ions cause undesirable volume changes in the host oxide matrix and related internal mechanical stresses that can lead to internal damage of the oxide and device failure. Depending on the sign of the film stress, a film-substrate interfacial bond can fail either by peeling or buckling. Compressive stresses appear in metal oxide films after ion intercalation, and failure may occur via decohesion or delamination.

L6.20 AN ANALYTICAL MODEL FOR INTRINSIC STRESS EFFECT ON OUT-OF-PLANE DEFLECTION OF A CHEMICAL VAPOR DEPOSITED THICK FILMS. Jeung-Jyun Jeong, Dongil Kwon, School of Materials Science and Engineering, Seoul National University, Seoul, KOREA; Yong-Joon Bisk, Korea Institute of Science and Technology, Thin Film Technology Research Center, Seoul, KOREA.
Polycrystalline diamond wafers for these thick film applications such as heat sinks, SAW, and HF windows are being developed using plasma assisted chemical vapor deposition (CVD). Several commercial processes are successfully used for this diamond wafer fabrication. In the development of a diamond wafer, however, CVD processes often cause out-of-plane deformation in film when film is released from the substrate. In this study, a theoretical model showing how residual stress influences the out-of-plane deformation in a free-standing thick diamond film, which is referred to as bowing phenomenon, is presented. The model is based on residual stress distribution at the film and is believed to be a cause of bowing. In this study, the stress variation is assumed to be produced by a gradual increase in substrate deformation resulting from many layer-by-layer deposition of the film. The model was developed using in situ thermal stress theory, considering the two deformation modes of contraction or expansion and bowing. To verify the suggested model, diamond films were fabricated on Si, Mo and V substrates of varying thicknesses by microwave plasma assisted chemical vapor deposition. The models predictions on bowing, based on the intrinsic stress value measured by the curvature method, were in good agreement with the bowing curvature of the a-released films measured by a profilometer. This result makes sure that the bowing of thick films depends on the intrinsic stress variation of the film associated with a gradual increase in substrate deformation. A method of eliminating bowing is suggested through depositing different layers with different intrinsic stresses.


Hardness measurements of thin films have to face the problem of the influence of the substrate hardness on the measurement result. Also the hardness measurements method has an important part in the obtained experimental data. Some of the models, which take into account such behaviour, are presented. We describe here many of our experimental results of the hardness measurements by Vickers test. There are investigated the nitride layers (CrN, TiN, NbN), superhard layers composed of these nitrides and hard metal films deposited either on the monocrystalline Si or metallic substrates as steel or copper. We propose here a model for superhard hardness anisotropy superhard modulus explained on Feruni surface. Brillouin zone interaction theory similar to HKS procedure for the galvanoelastic properties. We present also the measurements of magnetoresistance effect on the superhard layers, which consist of the multilayers T/NiN deposited on Si substrates. There are investigated the layers of different thickness of TiN and NiN obtained by the reactive sputtering method in different superhard layers. The composition (superlattice on Si) reaches 800GPa and MR value in the best cases was ~80% (beginning from some Ga to kGs). When the supporting thin metallic layer is present, then its influence on the composite hardness was also observed. This work is supported by KBN - Poland under Grant No. 7 T08C 001 16.


As a coating solidifies by drying, it tends to shrink. In early stages it is liquid enough that any shrinkage stress is rapidly relieved by viscous flow. In later stages it becomes solid enough to support elastic stress, which results from shrinkage induced by adherence to the substrate. Stress can relax by viscous creep in the coating. Thus the stress level is an outcome of competing shrinkage and relaxation. A theoretical model of diffusion and mass transfer, large shrinkage-induced deformation and stress, together with yield and post-yielding viscous deformation was developed to predict stress evolution in drying of polymer coatings after solidification. The coupled equation for evaporation and solidification and stress development by the Galerkin/finite element method. In polymer coatings this model is used to study the effect of a grooved substrate and embedded particles to the stress and drying, and to simulate cantilever deflection method that is being used to measure stress experimentally.

L6.23 GOLD/NIOBIUM THIN FILM METALLIZATIONS FOR GaAs DEVICES AND CIRCUITS. Robert Eason, Naval Research Laboratory, Washington, DC, and A. Christo, Dept. of Materials and Nuclear Engineering, University of Maryland, College Park, MD.

A niobium/gold thin film system for first level metallization on GaAs is proposed and examined. Niobium displays good adhesion to other semiconductor substrates such as Si, SiO2, and diamond. It is corrosion resistant. Niobium has the third lowest resistance of the refractory metals. Finally, the melting point of TiN is believed to be a cause of bowing. In this study, the stress variation is assumed to be produced by a gradual increase in substrate deformation resulting from many layer-by-layer deposition of the film. The model was developed using in situ thermal stress theory, considering the two deformation modes of contraction or expansion and bowing. To verify the suggested model, diamond films were fabricated on Si, Mo and V substrates of varying thicknesses by microwave plasma assisted chemical vapor deposition. The models predictions on bowing, based on the intrinsic stress value measured by the curvature method, were in good agreement with the bowing curvature of the a-released films measured by a profilometer. This result makes sure that the bowing of thick films depends on the intrinsic stress variation of the film associated with a gradual increase in substrate deformation. A method of eliminating bowing is suggested through depositing different layers with different intrinsic stresses.

L6.24 RELIABILITY OF METAL INTERCONNECTS. Young Bae Park, Young Ah Cho, Hyuk Hyun Ryu, Won Gyu Lee, System IC & E Division, Hynix Semiconductor Inc., Cheongju, KOREA.

Mechanical stress in metal interconnect line is very important in large scale device integration because the metal line is mostly under a triaxial tensile stress state that is unfavorable for void formation which is a major concern for the reliability of multilevel metallization. Direct X-ray diffraction determination of the mechanical stress state in Al-Cu interconnect lines was used to investigate the effects of various passivation dielectric film and metal line width on the effect of measured stress on electromigration (EM) reliability of the metal interconnect was investigated. Also, reservoir length dependencies of EM lifetime for Al-Cu lines with W via were compared for various passivation dielectrics. The passivation layers used in this study are PETEOS (Diamon enhanced Tetra Ethyl Ortho Silicate), FOX (Fluorolene Oxide), HDP FSG (High Density Plasma Fluorinated Silicate Glass), SiN (Silicon Nitride), FOX/TEOS stack, and FSG/TEOS stack. Also, various metal line widths (0.25, 0.5, 1, 10μm) and via reservoir lengths (0.04, 0.08, 0.12, 0.30μm) were used for stress measurement and EM test of metal interconnect. We tried to experimentally reveal the relationship among EM lifetime, stress dependencies on the passivation layers. Results showed that stress in metal line and passivation was not a direct relevant variable for EM lifetime. And, indeed, the stresses in passivated lines were highest in narrow lines, and a longer reservoir more improved the EM lifetime. Possible mechanisms for reservoir length dependencies of stress and EM reliability of metal interconnect will be discussed through FIB (Focused Ion Beam) failure analysis and FEM (Finite Element Method) stress analysis.

L6.25 MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SILICA BASED LOW-K DIELECTRIC FILMS. Joe Vella, Qiuanghua Xie, Ginger Edwards, Joe Kulk, Motorola Process and Materials Characterization Laboratory, Mesa, AZ, Kurt Junker, Motorola Advanced Products Research and Development Laboratory, Austin, TX.

Low-k material integration issues that continue to plague the microelectronics industry include the compromise in mechanical properties that one incurs in abandoning traditional dense silicon dielectrics. An understanding of the mechanism of plastic deformation in these porous and amorphous materials is critical for modeling the material's device performance. Typical elastic moduli of silica base low-k dielectric films are 1.10 GPa with corresponding hardnesses of 0.5 to 1.5 GPa. In the present study, the hardness and elastic modulus properties measured by nano-indentation of porous silica based low-k films are correlated with the density and conformation of matrix and void transmission electron microscopy (TEM). Young's and Poisson's elastic compliances are used to determine material density, porosity, size, and orientation. Shifts in the electron energy loss spectra may be correlated with the porosity measured by spectroscopic ellipsometry to determine crystallinity on the nanoparticle level and the termination of the silica bond network. Deformation mechanisms such as pore collapse and film cracking are explored via cross sectional
TEM across indentation profiles and their corresponding plastic zones. These mechanisms of yield will be discussed in light of the compensating in the silicon network due to organic (methyl, ethyl) inclusions.

**L6.26**
Abstract withdrawn.

**L6.27**
MAGNETOMECHANICAL PROPERTIES IN SOFT MAGNETIC MATERIALS. Ch. Arocain, A. Kurini, Department of Physics, Swiss Federal Institute of Technology (EPFL), SWITZERLAND.

Soft magnetic materials such as Fe-Cr(Al, Mo) yield to magnetomechanical interactions due to the irreversible movement of magnetic domain walls when subjected to an applied cycling stress. The magnitude of these interactions depends mainly on the domain walls mobility, which is basically controlled by magnetic properties (coercivity, remanence, magnetic relaxation, relative permittivity), and crystalline imperfections (point defects, dislocations, impurities). One consequence of this irreversible motion of domain walls is a change in elastic modulus (ΔE) effect and a dissipation of vibrational energy (magnetomechanical damping) which is associated to elastic deformation domain. In addition, one can expect interactions between domain walls and deformation microstructures which could occur during plastic straining of sample. To study these mechanisms a cantilever beam device and a nanoincider were used. Both experiments were conducted under variable magnetic field (between zero to saturation magnetization), in order to control dynamics of domain walls. The ΔE - effect and the magnetomechanical damping were measured by performing vibration tests and modal analysis of flat bars. Mechanical properties such as elastic modulus and hardness, as well as creep rate, have been measured by nanoindentation. The influence of thermal treatment and chemical composition on the mobility of magnetic domain walls, estimated from the damping capacity and ΔE - effect, were highlighted with respect to the magnetostatics effect and residual stresses. Creep under compressive stress has been found to increase with application of a magnetic field. In this paper we discuss these interactions in terms of magnetic domain structures as observed using AFM equipped with a magnetic force microscopy imaging option (MFM).

**L6.28**
Abstract withdrawn.

**L6.29**
TEXTURE AND NANOINDENTATION STUDIES OF YSZ ELECTROLYTE THIN FILMS PREPARED BY COMBUSTION CVD. Zhonging Xu, Qiuming Wei, Serpey Yarmolenko, Jag Sankar, NSF Center for Advanced Materials and Smart Structures, North Carolina A&T State University, Greensboro, NC.

Yttria stabilized cubic phase zirconia (YSZ) has been used as electrolyte in solid oxide fuel cells (SOFCs) because of its high oxygen ion conductivity over wide range of temperature and oxygen pressure. Thin film is preferred for the YSZ electrolyte in order to minimize the current path in the fuel cell. The fuel cells work at high temperatures (about 1000°C) and during the working cycles, the film will suffer from thermal stresses. A fundamental understanding of hardness and modulus of the film and knowledge towards the influence of the substrate interface on the overall behavior of the electrolyte is very important for the fuel cell application. The YSZ thin films have been synthesized with atmospheric combustion chemical vapor deposition (ACCVD) technique with liquid fuel. The so-grown films are characterized with X-ray diffraction, scanning electronic microscopy and transmission electronic microscopy to identify their microstructures. The mechanical properties are characterized by nanoindentation. The effect of processing parameters, such as substrate temperature, ratio of fuel to oxygen and concentrations of metal reagents, and substrate materials on the microstructure and nanohardness are investigated. The effects of different metal-organic chemical reagents on the microstructures are also compared. The relationship between micro-mechanical properties and microstructures are studied.

**SESSION L7. ADHESION AND FRACTURE I**
Chair: Huaian Guo and Sheldie P. Baker
Wednesday, Morning, November 28, 2011
Room 304 (Hynes)

**8:30 AM #L7.1**
RATCHETING INDUCED CRACKS IN THIN FILM STRUCTURES. Z. Song, M. Huang, Mechanical and Aerospace Engineering Department and Materials Institute, Princeton University, University, Princeton, NJ. Q. Ma, Intel Corporation, Santa Clara, CA. J. He, Intel Corporation, Components Research, Hillsboro, OR.

In the microelectronic and photonic industries, temperature cycling has long been used as a quality control method to detect microstructural and material properties of small feature sizes. The test is time consuming and has been a bottleneck for innovation. Tremendous needs exist to understand various failure modes in such structures caused by cyclic temperature. This paper presents an example of a failure mechanism discovered by the authors recently. In a thin film structure comprising both ductile and brittle materials, the temperature change can cause the ductile material to undergo plastic deformation in every cycle. Under certain circumstances, the plastic deformation rates, namely, accumulates in the same direction as the temperature cycles. The ratcheting deformation may build up stress in the brittle material, leading to fracture. This paper describes the experimental observations and introduces an analogy between ratcheting and viscous flow. An analytical model is developed, which explains the experimental observations, and allows one to design the structure to meet this failure mode. Concepts presented herein are generic to related phenomena in thin film structures. PDF files of the presentation can be downloaded from www.princeton.edu/~xuo, Publication 118.

**9:00 AM L7.2**
THIN FILM FRACTURE DURING NANOINDENTATION OF HARD FILM - SOFT SUBSTRATE SYSTEMS. M. Peng, K.D. Weaver, and D.F. Baur, Mechanical and Materials Engineering, Washington State University, Pullman, WA.

Nanoindentation testing of metal/metal oxide systems exhibit permanent deformation prior to a yield excursion, indicating that the occurrence of this sudden discontinuity is predominantly controlled by oxide film cracking rather than dislocation nucleation and multiplication. In a previous paper, a model was developed to predict the mechanical response prior to oxide fracture for the case of a hard coating on a soft substrate. This model predicted the failure load and the onset of the model to passivation films on copper will be discussed.

**9:15 AM L7.3**
PLASTIC ENERGY DISSIPATION IN THIN POLYMER FILMS: IMPLICATIONS FOR INTERFACIAL ADHESION. Christopher S. Letekene and Reinhold H. Dauskardt, Department of Materials Science and Engineering, Stanford University, Stanford, CA.

The development of new organic low-k materials has provided an opportunity to characterize the plastic energy dissipation in thin polymer films by studying the adhesive properties of thin film structures containing polymer layers. Specifically, macroscopic adhesion values in thin film structures containing thin polymer, metal, and ceramic layers were determined by measuring the critical strain energy release rate, Gc, for debonding of a selected interface. The significant contribution to Gc values from local plastic energy dissipation in the polymer layer was examined while manipulating the elastic constraint of the polymer layer. This was achieved by varying the thickness of the polymer layer, the thickness of intermediate layers, and by producing patterned polymer lines of varying aspect ratio. The significant effect of plastic energy dissipation in such polymer materials is often not observed for thin metal layers such as Al and Cu due to thin film strengthening mechanisms that occur in crystalline metals. Results will be presented which demonstrate that polymer yield properties are not sensitive to layer thickness and other geometrical features over typical feature length scale. Thin film properties relevant to microelectronic device structures will be discussed in terms of the prevailing plastic deformation mechanism.

**9:30 AM L7.4**
RATCHETING AS A FAILURE MECHANISM IN A CLASS OF THERMAL BARRIER SYSTEMS. Annette M. Kirkland, Anthony G. Evans, Princeton Materials Institute, Princeton University, Princeton, NJ.

A thermal barrier system is a multi-layer system consisting of (i) the super alloy substrate, (ii) the bond coat, (iii) the thermally grown oxide (TGO), and (iv) the thermal barrier coating (TBC). This system continuously evolves during its lifetime, where the TGO is a reaction product that forms by oxidation of the bond coat. Even though the TGO is thin compared to the other constituents, it is nano-connected with many of the features observed in the thermal barrier systems. In this study, we investigate NiCoCrAlY bond coats with
EB-PVD 2x02. The failure mechanisms in this class of thermal barrier systems are intrinsically linked to the morphological features of the TBC/substrate interface that develops during coating deposition. While these features assume a critical size, they act as nucleation sites for cracks and are eventually associated with large scale buckling of the TBC. The geometric instabilities of morphological features are caused by several factors. A primary factor is the thermal mismatch between bond coat and TGO, causing a large compressive stress in the TGO at room temperature (3-6 GPa). Furthermore, the oxidation of the TGO leads to a growth stress in the TGO. In addition, a necessary factor for transforming the initial small imperfection to a critical yielding and reversing yield of the bond coat and the TGO. Results are presented to illustrate how the development of stresses and strain during thermal cycling results in a shape change in the layered structure.

9:45 AM L7.5

Adhesion is a critical factor in the performance of hybrid microcircuits where the diffusion of copper from leads during processing and cooling can alter the adhesive strength and thereby influence the performance of the devices. High-temperature diffusion bonding techniques have been used to bond copper substrates to copper microstructures. We have therefore begun to study the interfacial fracture susceptibility of gold-gold, gold-gold-on-copper, and gold-2x02-copper films. Nanindentation showed that the adhesion of thin and hard-film gold and gold-on-copper films were similar but less than corresponding values for the gold-gold alloy film. A tungsten overlayer was then used to deposit onto the film systems to apply a uniform compressive stress. Then combined with nanindentation, these stress-strain-deformations deduced interfacial delamination and blister formation from which fracture energies and interfacial bond strengths were obtained using mechanics-based models. The results show that both the gold-gold alloy and gold-on-copper metal films adhere better than single-layer gold films. In this presentation, these results will be discussed and linked to the roles of composition and structure on interfacial fracture. This work is supported by U.S. DOE Contract DE-AC04-94AL85000.

10:30 AM L7.6
ADHESION AND COALESCENCE OF DUCTILE NANO-PARTICLES. Jacobs Israelenchik, Chad Park, Norma Almenar, Joan-M. Pue, Department of Chemical Engineering and Material Science, University of California, Santa Barbara, CA.

Much is known about the fundamental mechanisms of material failure, such as fracture toughness and propagation. It is less clear how these mechanisms interact with the disperse state of materials. This work uses the reverse effect of how bulk material form or coalesce via the coalescence of their constituent nanoparticles or surfaces, e.g., during material processing. Using the Surface Forces Apparatus (SFA) technique, it is possible to measure forces between thin films as well as to image their interface. We have studied how bulk gold and platinum films form at the nanoscale when two initially rough surfaces are pressed together. We find that coalescence of these ductile materials occurs abruptly, like a phase transition, once a critical local pressure or interparticle separation is reached. Simple thermodynamic reasons are given for this apparent general effect, which suggest that it may also occur in other systems and situations.

11:00 AM L7.7

As the critical size feature in microelectronic devices continues to shrink below 100 nm, it may not be feasible to deposit diffusion barrier/adhesion promoter layers by conventional physical and chemical vapor deposition (PVD and CVD) routes. As a result, new methods must be developed to address this problem. A possible solution is the replacement of traditional metal barriers with self-assembled monolayers (SAMs). Such a solution will also be compatible with the fabrication of newly emerging micro and nano device systems. Here we provide recent demonstration of the wetting properties of SAMs with an amorphous terminal functional group and a silane group. In order for these SAMs to be used in applications, their role in improving interfacial adhesion to SiO2 or Si3N4-based layers has been studied. We have recently demonstrated the existence of interfacial adhesion in Cu/SAM/SiO2 thin film structures by a mixed-mode delamination technique. The details of interfacial failure are revealed by surface analysis of delaminated structures using X-ray photoelectron spectroscopy and high resolution transmission electron microscopy. The adhesion results are correlated with the chemical structure of the SAMs, and discussed in the context of their diffusion barrier properties. A. Krishnamoorthy, K. Chand, S.P. Murarka, J.G. Ryan, and G. Ramamurthy, "Self-assembled monolayer thin-film adhesion to Cu", Applied Physics Letters 78, 2467 (2001).

11:15 AM L7.8
NANOINDENTATION INDUCED FRACTURE IN HARD MULTILAYER FILMS. D. Bethemuth, Y. Wang, A. Karimi, Department of Physics, Swiss Federal Institute of Technology (EPFL), Lausanne, SWITZERLAND.

Mechanical properties and fracture behavior of multicomponent titanium aluminum carbide thin films were investigated using nanoindentation measurement methods. The films were prepared as a single layer, multilayers, and nanocomposites and deposited onto the WC-Co substrates. Although depth sensing nanoindentation of the samples provided a measure of hardness and modulus versus microstructures and deposition parameters, but normal loading, unloading cycle did not show the formation of any detectable thickness changes in such film-substrate system. To activate different failure modes and assess fracture toughness and adhesion parameters of nanostructured films, microscratch tests under a constant or continuously increasing load were performed. Scratch damage appeared first by a visco-elastic deformation at the initial stage of the contact before the occurrence of plastic deformation and plowing mechanism leading to ductile character of scratch groove. When the applied load reached a critical value, periodic microcracks appear regularly on both sides of the contact area due to tensile stress ahead of the indenter. Further displacement of indenter into the film develops a compressive stress ahead of the tip and results in the formation of periodic through thickness lateral cracks and material removal by flaking. The fracture events are strongly influenced by the frequent presence of brittle submicron titanium rich particles randomly distributed within the films because of re-deposition technique. These structural inhomogeneities yield microcracking much earlier than the overall film, modify median scratch crack, and develop a complex stress field ahead of the indenter which causes the propagation of lateral internal cracks and thereby surfacing of the film adjacent to the indenter. Some of these cracks give signature on the nanoindentation and scratch curves from which fracture properties and interfacial strength were determined. This paper deals with fracture mechanisms of nanostructured titanium aluminum carbides and attempts to highlight the relationship between microstructures, fracture modes and mechanical properties.

11:30 AM L7.9
ANALYSIS OF Ti ADHESION TO <100> Si SUBSTRATES WITH VARYING SURFACE PREPARATION CONDITIONS. N. Barrosa, III, R.P. Vanc, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA. R. Bagley, S. C.H. Sone, R.S. Dwyer, T. Greb, Fairchild Semiconductor, Mountaintop, PA.

The desire for improved process yield has motivated the power semiconductor industry to better understand the effects of process variation on reliability. Wafer probes are often thinned to reduce the through thickness resistance of their end product devices. Processes involving wafer thinning can affect the adhesion of the subsequently deposited thin films. The relationships among surface character, film adhesion, and film stress are investigated through variation in sample preparation for sputter deposited Ti thin films on a <100> Si substrate. Surface roughness values (RMS) ranging from 25 nm to 200 nm were obtained through the use of various etching techniques [wet chemical etching, plasma etching], and measured with an atomic force microscope (AFM). The Ti film strength was varied through changes in temperature, substrate, and through the use of an in-situ optical film stress measurement tool. Scratch testing and the modified - Edge Lift off Technique (m-ELT) were utilized to show the effect of Si surface quality on Ti film adhesion.

11:45 AM L7.10
SAMPLE GEOMETRY EFFECTS IN THIN FILM BILTER DELAMINATION TESTS. Z.L. Zhang, SINTEF Materials Technology, Trondheim, NORWAY. J. Vlasak, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

The bilster test has certain advantages for measuring thin film adhesion toughness. Equations for energy release rate in the bilster test are available in the literature. The energy release rate was found to be based on either the estimated bilster area, calculated bilster area, or non-linear plane solution combined with the layer crack model by Suo.
and Hutchinson. In all cases, however, the substrate was assumed to be perfect and infinite, and the effect of the hole in the substrate used for pressure sensing was neglected. However, irradiation and analyses we have shown in this paper that the energy release rate for the blister test will be significantly affected by the normalized hole size, geometry and mechanical mismatch between the film and substrate. Both elastic and plastic thin cases have been considered. The energy release rate increases steadily with delamination. The general observation is that a delamination over a distance of about 100 times the film thickness is necessary before the energy release rate increases. Observations from tomography, as well as tests, can be applied. This observation is particularly important for practical applications where in many cases the extent of delamination is considerably smaller than this limit. The experimental work that we have presented here is an important step toward the development of new methods for the use of compliant substrates to further increase the delamination range that invalidates the energy release rate equation. Several other critical issues including the mode mixity during loading and unloading (delamination) and the possibility of reaching a maximum pressure after the initiation of delamination have been explored.

SESSION L8: THIN FILM APPLICATIONS IN MEMS
Chairs: Eric H. Chason and David J. Srolovitz
Wednesday Afternoon, November 28, 2001
Room 304 (Hynes)

1:30 P.M. L8.1
MECHANICAL STABILITY AND CHARACTERIZATION OF DIELECTRIC THIN FILMS. Robert F. Cook, Jeremy Thurn, Dylan J. Morris, Yeete A. Tokoh and Michael F. Higby, University of Minnesota, Department of Chemical Engineering and Materials Science, Minneapolis, MN.

Dielectric thin films are used in a wide variety of advanced technology devices including microelectronic, photonic, magnetic storage and micro-electromechanical systems (MEMS). In all cases, the dielectric performs a (frequently primary) role as a structural element in the device, in addition to providing appropriate insulating, optical or permeability characteristics. The mechanical stability of dielectric thin films is then of crucial importance for advanced device design especially as most dielectrics are brittle and prone to fracture. This talk will describe a variety of techniques used to characterize the mechanical properties of dielectric thin films, leading to an understanding of fracture behavior: wafer curvature for film stress, stability and thermal expansion coefficient; depth-sensing indentation for modulus and hardness; indentation fracture for toughness and stress-corrosion susceptibility; and indentation four-point bend for adhesion. Particular attention will be given to new methods of probe calibration for nanoscale hardness, modulus and toughness estimation and to implementation of a dual-substrate method for thermal expansion coefficient determination. The behavior of a wide variety of oxide, nitride, cyanide, carbide, silicon and hybrid organic-inorganic films will be described, for microelectronic, magnetic and MEMS applications. A theme of the presentation will be that the mechanical properties of dielectric films is as interesting and changeable as those of the more-traditionally studied metallic films.

2:00 P.M. L8.2

With the development of advanced fabrication techniques, it is becoming possible to produce structures and Micro Electromechanical Systems (MEMS) devices with lengths approaching the nanometer scale (e.g. Nano-Electromechanical Systems, NEMS). The performance of such devices is expected to be highly dependent on their electrical and mechanical properties. However, evaluating the mechanical properties of such devices is challenging due to their small size and the resulting difficulty in positioning indentation burdens accurately. Furthermore, as the length scale of the indentation approaches the scale of the nanostructure, the mechanical response can deviate markedly from that of a semi-infinite surface due to lack of constraint during deformation. The motivation for the present study is to develop an understanding of how size and shape of a structure, as well as lack of constraint, influence the measured mechanical response. A hybrid nanoindenter, scanned probe imaging and depth-sensing indentation capabilities, was used to examine patterns of nanostructures [nanostructures] as well as single metal wires and dots on silicon. The experimental results demonstrated that the mechanical response (load-displacement and contact stiffness) was affected by the lack of constraint. From these experiments, we show that the true materials properties of the nanostructures can be evaluated by correcting the contact stiffness to compensate for the lack of constraint.

2:15 P.M. L8.3
A NOVEL EXPERIMENTAL TECHNIQUE FOR MECHANICALLY TESTING THIN FILMS AND MEMS MATERIALS. Horacio Espinosa, Birt Proctor, Rong Wang, Max Fisher, Northwestern University, Department of Mechanical Engineering, Evanston, IL.

We have developed a membrane deflection experiment particularly suited for the investigation of sub-micron thin films that directly and independently measures actual load and strain elements of the test. This experiment consists of a fixed-fixed beam with a line load that is applied to the middle of the span with a nano-indentor column. A Mirau microscope interferometer is conveniently aligned with the nano-indentor to directly measure strain. This is non-destructively through a specified manufactured wafer containing a window to expose the bottom surface of the membrane. The sample stage incorporates the interferometer to allow continuous monitoring of the membrane deflection during both loading and unloading. By this method stresses and strains are measured directly and independently without the need for mathematical assumptions to obtain the parameters describing material response. We will present stress-strain signatures obtained on thin gold films 300, 500 and 1000 nm thick. Elastic modulus was consistently measured in the regime of 23-27 GPa which is considerably lower than the bulk value of 78 GPa. Although, values of thin films have been reported as low as 34 GPa (Nix, 1989). Rupture stress was measured between 110 and 190 MPa with corresponding rupture strain of 1.7 to 2.8%. We will also present data on the effect of membrane size and accumulated plastic strain as well as surface passivation with 30 nm SO2 layers.

2:30 P.M. L8.4
FRAC TURE BEHAVIOR OF MICRO-SIZED SPECIMENS PREPARED FROM AN AMORPHOUS ALLOY THIN FILM AT A MIDDEN AND ELEVATED TEMPERATURES. Ramski Tokuhama, Ryoji Terumi, Yukihi Higo, Tokyo Institute of Technology, Pet Lab., Yokohama, JAPAN.

MEMS devices are usually fabricated from a thin film deposited on a substrate by a suitable surface micromaching technique, and micro-sized elements prepared from a thin film layer are used as mechanical components. Amorphous alloy thin films prepared by deposition or sputtering are expected to be potential candidate materials for MEMS, because of their excellent mechanical properties and corrosion resistance. Therefore, the evaluation of the mechanical properties for amorphous thin films is important for practical applications of MEMS devices. In particular, fracture properties including fracture toughness is essential for designing reliable MEMS devices. In addition, the temperature of substrate is considered to increase up to 500 K during service. Amorphous alloy is non-equilibrium state at room temperature, and structural relaxation may occur at elevated temperature, even if lower than its crystallization temperature. Therefore, it is also important to investigate fracture properties at elevated temperature. In this investigation, fracture behavior has been investigated for microscale cantilever beam type specimens prepared from an electroless-deposited Ni-P amorphous alloy thin film at room and elevated temperatures. Cantilever beam type specimens with dimensions of 10 x 2 x 500 μm² were prepared by a Ni-P amorphous alloy thin film, and notches were introduced by focused ion beam machining. Fatigue pre-cracks were introduced ahead of the notches. The temperature of the specimen was controlled from room temperature to 473 K. Compared with the room temperature, fracture toughness increased approximately 36% at 373 K but decreased 16% at 473 K. The increase of fracture toughness at 373 K is considered to be related with precipitation of nano-scaled crystals and the decrease of fracture toughness at 473 K is considered to be due to crystallization of the amorphous. It is required to consider the fracture behavior obtained in this investigation when designing actual MEMS devices using electroless-deposited amorphous films.

2:45 P.M. L8.5
STRESS RELAXATION MECHANISMS IN TETRAHEDRAL-AMORPHOUS CARBON FOR MEMS APPLICATIONS. T.A. Friedmann, T.M. Schneid, R.V. Ellis, M. Mitchell Sandia National Laboratories, Albuquerque, NM.

Tetrahedral amorphous carbon (ta-C) deposited by pulsed laser deposition, colloquially known as amorphous-Diamond (a-D), can be fully stressed relieved by thermal annealing. We have studied the stress relaxation kinetics using in situ technique to measure real-time changes in wafer curvature with annealing time and temperature. The results reveal the process to be thermally activated. Two mechanisms for stress relief in these materials have been postulated, each involving stress-relieving transformations from the tetrahedral to amorphous phase. Recently, we have made fully 13C enriched films by ablating from a 13C (99%) target. Nuclear magnetic resonance (NMR) magic-angle
spinning measurements of these enriched films have been made to quantify the changes in structure with annealing in an effort to validate the proposed models. Results of these measurements will be presented along with Ramman, SEM, TEM, and cross-section EELS experiments.

The low stresses that are achievable in 3D makes this material suitable for sensors. Micromechanical systems (MEMS). We have demonstrated several MEMS structures from this material. One set of structures includes simple one-element cantilever beams and tensile pull tabs designed to evaluate the micromechanical properties of the film. The tensile test results obtained by using a nanomanipulator to pull laterally on the samples until fracture occurred. The 3D fracture strength was found to be dependent upon the gauge volume in a manner consistent with simple statistical mechanics.

3:30 PM #18.6

The use of metals is becoming increasingly important in microelectronic thin film device applications such as microswitches and radio frequency switches. However, when thin film metals are used as a structural material, fatigue and creep are of particular concern. Using micromachining techniques, we introduce a new method for preparing optically patterned test structures to study the mechanical properties. The test structure is a fixed-fixed beam suspended five microns above a substrate. It is wide at the support posts, but narrows to a ligament, greatly amplifying stress. Actuating the structure out-of-plane by electrostatic voltage enables further stress application up to the glass transition temperature, thereby inducing plastic deformation. Layout geometry allows the degree of stretching versus bending stress in the film to be controlled. Formation of nearly rigid support posts is accomplished by electroplating. A metal film is then deposited and defined to span the posts by lithography. Here, we investigate Al films with and without Cu alloying. Testing is accomplished in two ways. First, deflections are monitored by interferometry while applying voltage to actuation pads under the beam. By matching the deflection data to a 3D electromechanics model, we sensitively determine the residual and applied stresses in the ligament, allowing us to obtain fatigue and creep data. Second, we etch a hole through the back side of the substrate. The test structure is then mounted in situ and microscopically imaged by transmission electron microscopy while the film is actively loaded to a controlled stress level. This work leads to an improved understanding of the reliability of metal films to be used for structural applications. Acknowledgment: Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

4:00 PM #18.7
IN SITU MECHANICAL CHARACTERIZATION OF A FREE-STANDING 100 NANO-METER THICK ALUMINUM FILM IN SEM USING MEMS SENSORS. M.A. Hogue and M.T.A. Saif, Dept. of MSE, University of Illinois at Urbana-Champaign, Urbana, IL.

We present the uniaxial stress-strain response of a 100 nanometer thick, 8,8 microns wide, and 276 microns long freestanding 99.95% pure sputtered Aluminum film with grain size about 60 nanometers, tested in situ inside an SEM chamber. The specimen is fabricated with MEMS techniques to include the following unique features, 1. Ability to measure shear stress in the specimen, 2. Reduction of any force misalignment by five orders of magnitude, 3. Elimination of any gripping mechanism, 4. Small sample size to fit SEM and TEM chambers, and 5. Ability to measure creep activation energy. The MEMS force sensor is calibrated with a nanomanipulator. In the first loading cycle, the specimen behaves linearly up to 460 MPa. The elastic modulus value was found to be 96 GPa. The specimen then showed a nonlinear deformation. In the second loading cycle, it started to show yielding at about 655 MPa. At 655 MPa, the specimen showed hardening. In the third loading cycle, the specimen showed no yielding. After about 10 minutes at 460 MPa, the specimen showed hardening. The yield strength for the specimen was 675 MPa. The specimen was then strained to 675 MPa stress without failure. At this point, the specimen with mean diameter 300 nanometers were seen on the specimen. The experimental results strongly suggest that 1. The grains deform elastically up to 675 MPa, 2. The specimen produces the yielding effect. 2. There is no strain hardening at this size scale, which indirectly suggests the creep mechanism is not the dominant mechanism.

4:15 PM #18.8
ABSORPTION INDUCED FAILURE MODES OF THIN FILM RESONATORS. Robert Knorrnm, Jeff Mollinger, Andre Bossele. Deft Univ. of Technology. Dept. of Information Technology and Systems, Delft, THE NETHERLANDS.

The further downsizing of mechanical micromachined devices lead to emphasized surface related problems. The change of the surface properties severely affects the mechanical properties and thus the operation of MEMS devices. In several applications the mechanical components are in direct contact with the environment. The adsorbs atoms and molecules from the surrounding media. The adsorbates change the surface tension and the mass of the micro-machined structures. This is typically a problem in case of devices based on resonant structures, because the resonance frequency of the resonator is changed due to the adsorption. This phenomenon is exploited in applications like functionalized probe tips, but special care is required to eliminate the adsorption of undesired components. We studied silicon nitride and silicon carbide cantilevers, and the effect of various environments on their mechanical and resonant behavior. The measurement results were compared to analytical calculations and FEA simulations. The resonance frequency increased logarithmically in time in ambient air. The silicon nitride samples exposed to air for several weeks suffered 2% spring constant increase due to a surface stress change of 0.0181 nm. This corresponds to a compressive stress of 30 MPa in the surface film. Oxygen exposure on the surface increases the stability of the resonators. The shock response of the structures was studied in various environments. The resonance frequency abruptly drops up to 5% due to cracking of the absorbed and/or oxide layer, then recovers logarithmically. The initial drop and the recovery rate is environment dependent. Humidity increased, while argon and nitrogen rich environments weakened the effects. These results offer cheap atmospheric packaging solution for resonators. The silicon carbide resonators are less sensitive to the environmental effects. No substantial oxide formation was observed, though adsorption slightly increased the stiffness of the structure. Silicon carbide proved to be a promising candidate for structural components in resonant MEMS devices.

4:30 PM #18.9
LOW TEMPERATURE SILICON DIOXIDE THIN FILMS DEPOSITED USING TETRAMETHYLSILANE FOR STRESS CONTROL AND COVERAGE APPLICATIONS. Xin Lin, Motorola, SPS, Mesa, AZ; Stephen Fonash, The Pennsylvania State University, University Park, PA.

Low temperature silicon dioxide thin films have been prepared by plasma-enhanced chemical vapor deposition (PECVD) using Tetramethylsilane (TMOS) at 100-250 C in the pressure range of 2-8 Torr. PECVD TMOS oxide thin films deposited at these temperatures and pressures exhibit adjustable stress. The type of stress, from tensile stress, to essentially zero stress, to compressive stress, as well as the stress level can be tailored as desired by changing the deposition conditions and film thickness. In addition, the conformality of PECVD TMOS oxide thin films can be made to vary significantly by adjusting the deposition conditions. Conformality improves when the deposition pressure is raised and the substrate temperature is reduced. The mechanisms for the variations of stress and conformality with respect to deposition conditions were discussed in this study. The adjustable stress and conformality of the PECVD TMOS oxide makes it a promising material for many low temperature applications, such as inter-level dielectric, micro-electro-mechanical systems (MEMS), microfluidics, and large area electronics.

4:45 PM #18.10
THE DESIGN OF MULTILAYERED POLYSILICON FOR MOEMS APPLICATIONS. D. Sherman*, H. Kahn*, S. Phillips*, R. Balaram, and A. H. Tozer**, Case Western Reserve University, Cleveland, OH. "Department of Mechanical Engineering and Computer Science". "Department of Civil Engineering.

A rigorous analysis of a multilayered polysilicon laminated system, constructed by alternating low-temperature plasma enhanced chemical vapor deposition (LPCVD) polysilicon thin films at two different temperatures is presented. Different residual deformation fields are generated in these polysilicon thin films as a function of fabrication temperatures due to differences in crystalization rates. The combination of the two layers, however, enables precise control of the radius of curvature of released structures, provided the material properties are well defined. We
describe a new method, which combines experimental and numerical procedures, to define the material properties, which responsible for the residual strain function of thin films. The procedure to design the desired curvature of a multilayer device. A linear deformation field is assumed. It is shown that the precise design of the thicknesses of the individual layer is a prerequisite for controlled curvature. Therefore, we have determined the curvature of single and multilayered polysilicon systems with good precision.

SESSION L9: ADHESION AND FRACTURE II

Chair: Neville R. Moody and Cheung-Him Chiu
Wednesday Evening, November 28, 2001
Room 304 (Hynes)

8:00 PM L9.1
THIN FILM PRESSURE SENSITIVE ADEHESIVES
APPLICATIONS IN TRANSDERMAL DRUG DELIVERY.
Richard H. Dadswell, Marc Taub and Kenneth Wu, Department of Materials Science and Engineering, Stanford University, Stanford, CA.

Pressure sensitive adhesives (PSAs) are used extensively in the form of thin-films to adhere devices for transdermal drug delivery to the dermis. PSAs are unique in that they offer desirable properties of good initial and long-term adhesion, clean removability, and skin and drug compatibility. The development and implementation of successful transdermal devices for drug delivery requires an understanding of the adhesion occurring between the device and the soft dermal layer. The trend towards increasingly complex and novel patch designs further necessitates the development of a systematic approach to quantify adhesion. However, understanding of the adhesion of PSAs in complicated bio-organic systems is not well understood. As such, there is almost no reproducible test methods or quantitative adhesion data available for this class of materials. In this presentation, a mechanics approach to quantify the adhesion properties of representative PSAs is described. Examination of PSAs is accomplished by: 1) quantifying the PSA and the formation of an extensive cohesive zone behind the deformed tip. The presence of such an adhesive layer, large-scale bridging adds additional energy dissipation and increased resistance to delamination. The experimental results are modeled to account for both the work of adhesion as well as the viscoelastic constitutive behavior of the soft adhesive layer. The effects of varying the chemistry of the adhesives through modified interactions and adhesion will be discussed. The effects of environmental conditions (temperature, humidity, and aqueous environment) will be presented as they relate to cavitation behavior and adhesive performance. Finally, experiments to characterize the mechanical and fracture properties of the soft dermal layers are described, particularly as they relate to the mechanical integrity of thin-film transdermal drug delivery device applications.

8:30 PM L9.2
REAL-TIME IN SITU IMAGING OF THE DELAMINATION OF THIN Ta FILMS ON Si(100) SUBSTRATES VIA A SYNCHROTRON RADIATION TECHNIQUE. B.L. French and J.C. Bille, Center for National Synchrotron Science, Department of MSCE, University of Michigan, Ann Arbor, MI.

The thermal-stress induced delamination of 500nm polycrystalline Ta coatings on Si(100) substrates was studied via a recently developed real-time in situ imaging technique. This method used white beam synchrotron (on SSRL station 2-2) Laue transmission diffraction topography coupled with simultaneous direct radiography to record the thermomechanical failure modes of the Ta films as a function of temperature. The observations were made using a novel experimental apparatus, consisting of a 600°C heating device/sample holder and a portable CCD array imaging/stORAGE system with ~20μm resolution. The system is capable of recording 36 images/sec; hence, moderate strain rates not only was the morphology of failure evident, but detailed study of the dynamics of delamination was also possible. The mode of failure of adhesion strength are correlated with the physical-chemical nature of the containing substrate interface, and also with the degree of in-plane/out-of-plane texture of the Ta films. These observations should be capable of extension to the study of a variety of adhesion and cyclic failure issues in this class. This work was supported, in part, by the ARO under Grant #DAAG 55-98-1-0382, and the authors also thank USDEE for support for work done at SSRL.

8:45 PM L9.3
CRITICAL THICKNESS FOR CRACKING OF Pt/ZrO2/TiO2 (PZT) THIN FILMS DEPOSITED ON Pt/Ti/Si(100) SUBSTRATES. Ran Fu, Ming-Hao Zhuo, Desin Liu, and Tao-Xi Zeng, Hong Kong Univ of Science and Technology, HONG KONG.

This paper studied the critical thickness for cracking of Pt/ZrO2/TiO2 (PZT) thin films deposited on Pt/Ti/Si(100) substrates. The Pt/Ti layers with thicknesses of 150nm and 300nm, respectively, were deposited on the substrates by the RF sputtering. The Pt/ZrO2/TiO2 (PZT) thin films were deposited by solgel technique, baked at 110°C for 3 minutes, pyrolyzed at 350°C for 10 minutes and finally annealed at 650°C for 60 minutes. The experiment results show the existence of a critical film thickness of about 0.88μm for cracking of the PZT films. No cracking was observed if the film thickness was less than the critical value. When the film thickness was larger than the critical thickness, the crack density, defined as the total crack length per unit area, increased rapidly with increasing film thickness. An elastic-plastic model is developed to explain the experimental results. This model predicts the critical thickness for film cracking and the crack density as a function of the surface energy of the film, the film thickness, the residual stress in the film, and the yield strength of the metal layers. Comparing the theoretical results with the experimental results indicates that the model captures the main characteristics of the film/substrate system.

9:00 PM L9.4

Positional Beam Analysis provides a non-contact and non-destructive tool to monitor early stages of changes in the stress state of thin films during mechanical testing. In this study a four point bending apparatus has been mounted in the analysis chamber of a variable energy position beam. This configuration allows studying stress development and delamination phenomena of different coating/substrate systems. Varying the position implantation energy, the coating layer or the coating/substrate interface can be studied separately. A typical experiment consists of monitoring the position strain contours while the applied load is increased, either in compression or in tension mode for selected depths. Depending on the residual stress of the as-prepared coating, stress can be released or increased, e.g., from an initial compressive to a tensile state. Porous have proven to be very well capable of observing the transition from compressive to tensile stress in thin quenched Cr films on silicon substrates. This transition is detected by the increase of open volume when the stress state changes from negative to positive (tensile). Regarding adhesion, the very first defects associated with delamination can be observed. Examples of this novel analysis technique will be discussed on PVD TiN (4μm and 1μm thick) and WC/C (2.5μm) layers on tool steel and low carbon steel substrates.

9:15 PM L9.5
WEAR AND ADHESION OPTIMIZATION OF THIN TiAlN, CoN, AND C FILMS BY STRUCTURE DESIGN. G. Knoke, E. Liska-Moscher, K. Boltz, O. Leyendecker, T. Mayer, Institut für Materialwissenschaftliches Meßtechnik, University of Technology Aachen, GERMANY; CemCon GmbH, GERMANY.

The structure of TiAlN, CoN, and the other thin films depends on coating conditions, their parameters variation and the after coating treatment. Reactive sputter and evaporation process influence structure formation as well as the plasma existed chemical reaction on a substrate. The crystal formation and the agglomeration depends on the energy level and flow in the coating system, the kind and number of reactive partners, temperature on coating system specific parameters. The after coating treatment influences the stress formation and the residual stress but not the crystallization of different phases in the beginning. In some cases the spray structure is to be preferred in other the distribution of the hard phases in the matrix had to be optimized in size, amount and spatial depositing using plasma spraying. The use of different targets and a continuous change of the reactive gas mixture can influence these structure variation during a coating process for a special application.

9:30 PM L9.6
THE ROLE OF CHEMICAL COMPOSITION FOR THE DUCTILITY AND MICROSTRUCTURE OF THIN NiAl FILMS. Peter Ström, Oliver Kraft, and Eduard Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

NiAl thin films can be used as protective coatings for structural applications against oxidation and corrosion at high temperatures. For such applications, the ductility of NiAl films plays an important role since a lack of ductility may lead to the formation of cracks in the coating during temperature changes. In order to study the ductility of NiAl films with different compositions (5.0 to 50.0 at. % Al) and thicknesses (0.4 to 3 μm) were deposited by magnetron co-sputtering
Mechanical properties of thin film materials for the 2D numerical analysis of deep submicron CMOS technologies. Vinicius Sene, IEMN-ISEN, UM3 CNRS 8520, Villeneuve d’Ascq, France; Giovanni Carlotti, INFM and Dipartimento di Fisica dell’Università, Perugia, Italy; Ingrid de Wolf, IMEC, Leuven, Belgium; Roberto Balboni, CNR-Istituto LAMEL, Bolzano, Italy; Alessandro Benedotti, Department of Electronic and Electrical Engineering, University of Sheffield, Sheffield, United Kingdom; Donato Piccolo and Gianfranco Marraschini, STM Microsystems, Agrate Brianza, Italy.

In order to shrink the development time of state-of-the-art technologies, two- and three-dimensional (2D and 3D) process induced stress simulation capabilities are of prime interest. The difficulty in the numerical modelling of silicon technologies arises from the necessity to ensure a wide prediction capability (i.e., a high modelling accuracy) that requires to take into account all the sources of mechanical failures and to characterize the stress distribution in thin film properties. This paper presents a methodology to determine the Young Modulus, Poisson ratio and coefficient of thermal expansion by the combination of two complementary techniques, namely Brillouin Light Scattering (BLS) and Indentation (IN). This method has been used to characterize TEOS and HDP silicon dioxide, LPCVD silicon nitride, sputtered titanium and C49/C54 T182 which are largely used in the front end processes of modern deep submicron CMOS technologies. Subsequently, we have used a new method IMPACT that allows to take into account the stresses induced by complex physical phenomena like material growth (e.g. SiO2, silicides), water absorption/evaporation (e.g. in TEOS oxide or BPSG) and phase transformation (e.g. TiSi2). This capability represents a clear innovation with respect to conventional finite element methods. For the validation of the methodology, test structures have been designed and processed which consist in shallow trench isolations (STI) with different processes where the active region has been silicided using titanium. The corresponding process flow has been simulated with IMPACT and the calculated 2D stress/strain distributions have shown good agreement with the measurements performed locally in the test structures by means of confocal laser micro-Raman spectroscopy and the Central Backlash Diffraction Technique (CBED) for extremely compact structures (i.e., quarter micron) and by micro-Raman spectroscopy for larger structures (i.e., a few microns). This work has been performed in the framework of a European project called STREAM.
constructed as the sum of the total deformation energies over all atoms in an atomic lattice. In particular, our energy can be regarded as a generalization of the Kesting model. The energy can be related directly to the continuum elastic energy wherever in the material continuum theory gives a reasonable description of the strain. But the atomic nature of the energy allows it to be tailored to those local geometries where continuum theory does not apply, for instance at atomically sharp corners, defects, interfaces, etc. We derive the analytical conditions for minimizing the energy, which are similar to discretized continuum equations, and are suitable for numerical solutions. Some specific features we are able to incorporate are the latice mismatch between film and substrate, and numerical boundary conditions at sharp corners. Applications to specific epitaxial systems will be presented.

9:45 AM L10.5
ELASTIC INTERACTION OF STEPS ON A ROUGH SURFACE: MOVING DEVIATIONS IN HALF-SPACE PARADIGM
Robert V. Kistler, State University of New York, Dept of Mechanical Engineering, Stony Brook, NY.

The elastic interaction between atomic steps determines an important part of the thermoelastic force that drives surface evolution during crystal growth. The standard approximation of the step-induced elastic field as a line force and line dipole on a halfspace is accurate only for very flat surfaces. A revised model is presented for steps on a rough surface, which accounts for the actual surface shape by explicitly enforcing the traction-free boundary condition on terraces between steps. Analytical expressions are obtained. The model displays remarkable agreement with atomistic calculations. It is shown that the interaction energy between two steps exhibits a distinct intermediate-range behavior that extends out to separation distances of 100 step-heights or larger in some cases. Energies of multiple-step configurations are discussed where differences between the revised model and the halfspace model are found to be even more substantial. Novel features of the model are: (1) the elastic field cannot be represented by a line dipole on a halfspace, (2) the elastic field depends on step-step interactions and is not simply the superposition of individual step fields, and (3) the energy depends on threefold step interactions in addition to standard pair interactions.

10:30 AM *L10.6
SELF-ASSEMBLY OF HETEROEPITAXIAL ISLANDS.
Chengmin Chiu, Institute of Materials Research and Engineering, Singapore.

The self-assembly of nano-size heteroepitaxial islands on a crystal surface is a remarkable phenomenon with many potential applications in semiconductor devices. In this talk, we explore the morphologies and the structures of the nano-crystal islands that develop during the process, by employing both the energy analyses for the island structures and the numerical simulation for the morphological evolution of the islands. Of particular interest are the island structures formed in the initial stage of island formation as well as the stability of the islands against Ostwald ripening. The island formation is studied by reproducing in our simulation the different formation patterns observed in experiments such as surface roughening, development of a network of elevated islands, cooperative nucleation, and nucleation. Comparing the material parameters of the results reveals a clear picture about the driving forces and the kinetic mechanism that control the formation of islands. Our discussion then turns to the stability of the islands against coarsening, a crucial issue in device applications of the self-assembly technique. We first use the energy analyses to show that under strong film-substrate interaction and favorable surface energy anisotropy, the islands can evolve toward a steady state in which is stable against coarsening. The stability is further demonstrated by simulation for a model system that can develop pyramidal and dome islands. The results suggest that the stable island morphology can be pyramids, domes, or a mixture of pyramids and domes, depending on the details of the system. The results on growing a uniform island array is then discussed.

11:00 AM L10.7
PHASE FIELD AND DISCRETE DISLOCATION MODELING OF DEFORMATION IN MULTILAYER THIN FILMS.
Chen Shen, Qihen Li, Yunhui Wang, Peter M. Anderson, Ohio State University, Dept of Materials Science and Engineering, Columbus, OH, Tina Frecke, National Institute of Standards and Technology, Materials Division, Gaithersburg, MD.

Observations of dislocation motion in multilayer thin films of Cu/Ni indicate that the strength may be controlled by the percolation of dislocation loops through interfaces which may be decorated with obstacles in the form of misfit dislocations. This paper will present the results of two computational approaches to understand the process of dislocation motion. The first is the phase field model, which formulates the energy of a dislocated material in terms of a slip parameter that are mapped to a regular 3D array of nodes. The second is a discrete dislocation model which formulates the elastic energies of a dislocated material in terms of a sum of interaction energies of discrete dislocation line segments. The models then update dislocation positions according to a path of steepest descent in energy according to a Monte Carlo process. Each of these models will be used to simulate the motion of confined dislocation propagation within layers and the transmission of slip across interfaces with and without misfit dislocation content.
130 PM *L11.1* ANALYSIS OF NUCLEATION OF DISLOCATION LOOPS FROM THE SURFACE IN THE PIEZOELECTRIC-EFFECT framework. Guanghui Xu, Department of Mechanical Engineering, University of California at Riverside, Riverside, CA.

Nucleation of dislocation loops from the crystal surface under stress is analyzed using a variational boundary integral method in the Piezoelectric framework. The stress-dependent activation energies that are required to activate dislocation loops from their stable to unstable saddle point configurations are determined. Our findings are consistent with the previous analyses of this problem using the continuum dislocation theory, this analysis provides a more definitive solution because it eliminates the uncertain core cutoff parameter and the presumption that the dislocation loops are in the semicircular shape. Moreover, the influence of defects such as cracks and steps at the surface on the energetics of dislocation nucleation can also be readily studied using the presented methodology.

2:00 PM L11.2 Cu DEPOSITION ON Mo[110]: THE MAKING OF A MICRO-STRUCTURE. Bouke S. Burmik and Harrie J. Thijssse, Delft University of Technology, Dept of Materials Science, Center for Research on Solid-Solid Interactions and Surfaces (CRISS), Delft, NETHERLANDS.

Large-scale Molecular Dynamics simulations have been performed to investigate the structural development of Cu films of up to 8 nm thickness on Mo[110] substrates. Thermal evaporation as well as Ar-ion assisted growth (100 eV per atom) were studied, in order to separate thermal from ion-beam effects. We find that the heterodimeric structure of the system gives rise to a complex evolutionary behavior. The first Cu monolayer grows as a perfect polymorphic bcc planar, after which island growth sets in. The lateral island size is approximately 5 nm. Maximum roughness occurs after 0.7 nm nominal thickness. The islands grow together and after 1.2 nm the Cu film is flat again. In this stage the majority of Cu atoms still have a bcc symmetric coordination polyhedron. At 1.6 nm a massive transition takes place, in which most bcc Cu atoms become face or top coordinated. Therefore the growth continues steadily in a mosaic for[111]/hcp[111] surface orientation (i.e. not fully close-packed), while the roughness decreases slowly, only 0.02 nm/str. From atomistic dislocations and intrinsic stacking faults are the main crystal defects. They define the evolving microstructure. During the entire growth process the film as a whole continues to change. <111> crowns nucleate in the first Cu monolayer plane, and then tilted planes form grain boundaries. Ar-ion ballistic effects mainly influence interface sharpness (i.e. adhesion), island growth, and grain boundaries. The results will be illustrated by animated simulation movies.

2:15 PM L11.3 ISLAND SIZE EFFECTS ON INTRINSIC STRESS EVOLUTION IN IR-VISIBLE THIN FILMS. Aashish Agrawal, B. E. Sheldon, Brown University, Division of Engineering, Providence, RI.

AIN and GaN films were grown on patterned substrates to obtain well controlled, periodic domain sizes. In situ measurements of intrinsic stress typically show a small initial compressive stress followed by tensile stress during island coalescence, with further growth beyond coalescence also results in compression. The intrinsic compression is attributed to surface stress effects, while the tension is generated during both initial coalescence and during subsequent growth of the islands into each other. A finite element model is used to calculate stress distribution in the island before, during, and after coalescence. Some stress relaxation mechanisms are incorporated into the FEM model and their effects on stress generation will be detailed. The modeling results have made it possible to predict film stress and stress distribution between film growth conditions such as island size, precursor super saturation, and host surface properties.

2:30 PM L11.4 ANALYSIS OF INELASTIC DEFORMATION IN POLYCRYSTALLINE METAL FILMS ON SUBSTRATES. Yoonjoon Choi and Subra Sharek, Dept of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, MA.

The stress-temperature relation of thin polycrystalline metal films on substrates due to thermal strain has been modeled based on interaction energy between dislocations. The extent of strain relaxation at a given thermal strain is estimated by examining minimum energy configurations. The relaxed strain energy is stored in the form of sets of dislocation loops and the generation of dislocation loops is assumed to be restricted within the grain of thin films. By accounting for the interaction energy between dislocation loops, the equilibrium strain after relaxation from a given thermal strain as well as the corresponding stress are calculated. While thermally activated dislocation glide process or diffusional creep can play a role at room temperature, this work specifically examines those systems in which time-independent plasticity arises from dislocation interactions. The model predictions are compared extensively with available experimental data for unpassivated Au and passivated Cu films on Si substrates. It is shown that the predictions of the yield behavior of thin films as a function of temperature match experimental observations much more closely than those based on previous models. The model successfully describes the effect of two important size parameters, such as the thickness and grain size, on the mechanical properties of thin films and correctly predicted the development of stress states during thermal excursions. The dislocation density predicted by the analysis also rationalizes the high strain hardening phenomena seen in thin polycrystalline films.

2:45 PM L11.5 THE CORRELATION OF INDENTATION SIZE EFFECT EXPERIMENTS WITH PYRAMIDAL AND SPHERICAL INDENTERS. J. G. Szwedowski, Los Alamos National Laboratory, Los Alamos, NM, E.P. George, Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN; G. M. Pharr, Dept of MEIE, Univ Tennessee, Knoxville, TN and Oak Ridge National Laboratory, Oak Ridge, TN.

Based on the concept of geometrically necessary dislocations, the indentation size effect for pyramidal and spherical indenters can be correlated. Experiments were conducted using pyramidal and spherical indenters over a wide range of load. For a pyramidal indenter, the hardness measured at increasing loads decreases with increasing depth of penetration. However, for spherical indenters, hardness was not affected by the depth of penetration, but increased with decreasing sphere radius. After accounting for work hardening, the experimental results show that the number of geometrically necessary dislocations generated by indentation gives a correlation of the indentation size effect measured with pyramidal and spherical indenters. Material length scales were found to be approximately 200 micrometers in annealed FCC metals. *Oak Ridge National Laboratory Shar User Facility was sponsored by the Office of Space Power Systems and by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under contract DE-AC05-84OR22750 with UT-Battelle, LLC.*

SESSION L12: FILM DEPOSITION, MICROSTRUCTURE, EVOLUTION AND INTRINSIC STRESS I: Chair: Franz Spangen

Thursday, November 29, 2001
Room 304 (Hyene)

3:30 PM L12.1 EFFECTS OF GROWTH KINETICS ON RESIDUAL STRESS DEVELOPMENT IN POLYCRYSTALLINE THIN FILMS. E. Chien, B. Sheldon, L.B. Freund, R. Rajaraman, Brown University, Division of Engineering, Providence, RI; J.A. Floro, S.J. Hearne, Sandia National Laboratories, Albuquerque, NM.

In many polycrystalline thin films, residual stress develops in a similar way during deposition. In the initial stage of growth, when the film consists of individual islands, the stress is compressive. As the islands coalesce into a continuous film, the stress becomes tensile. At larger thicknesses, the stress often becomes compressive again. In this talk, we will discuss effects of the thin film growth kinetics on determining the stress evolution. In order to understand the tensile stress generation, we have developed a simulation that combines finite element calculations with a kinetic model for the formation of the grain boundary between two islands during growth. For the compressive regime, we have developed a model in which atoms are driven into the grain boundary by the higher chemical potential on the surface due to the grain flux. Both these models are compared with measurements of stress evolution obtained from wafer curvature experiments. Portions of this work were performed at Sandia National Laboratory and supported by the United States Department of Energy under contract DE-AC04-94AL8500.

4:00 PM L12.2 STRESSED STATES AND SELF-ORGANIZED STRUCTURING OF W/C MULTILAYERS. Dirk C. Meyer, Anke Klingen, Peter Paufler, Institute for Crystallography and Solid State Physics, Dresden University of Technology; Thomas Holz, Reiner Dietz, Fraunhofer Institute for Material and Beam Technology, Dresden, GERMANY.

Characterization and quantitative analysis of stressed states of a series of W/C multilayers (10-40 periods prepared by pulsed laser deposition on Si (111) substrates of different thicknesses) were carried
out by means of X-ray reflectometry, wide angle diffraction, and a novel laser mapping device. As the W/C multilayers were dedicated to technical application to X-ray monochromators and substrate optimization of stacking parameters (thickness and number of layers) for a long term (mechanical) stability also further investigations will be discussed. Comparison of water distillation as evaluated by laser scanning and strain of the W layer as deduced from X-ray diffraction let us conclude that W layers are under compressive and C layers under tensile stress. Moreover, we report on a self-organized process of structuring of the multilayers under investigation, which might be of interest also in a technical point of view. This technique is based on the 100 percent of the surface area (2 mm diameter 2 inches) could be converted from the smooth (as-deposited) to a structurized (refracted) state at room temperature. Investigation by optical and electron microscopy showed that the topology of the surfaces consists of a mountain range where the valleys are on the level of the as-deposited non-debonded surface and that long wrinkled ridges of about some height run along arbitrary directions. The structure thus obtained had characteristics of the AFM roughness of 30 nm. The maximum height of the ridges was found to be between 2 and 3micrometer. A wrinkle will grow if the release rate is equal to the debonding surface energy. The constant width of wrinkles enables the bonding energy of the layer to be estimated. Part of the ridges could be made to disappear or to rearrange by applying external strain. This in combination with quantitative results for stress states within the W and C layers will enable the pattern to be controlled.

4:15 PM L12.3
HIGHLY ORGANIZED NTI-CU SHAPE MEMORY THIN FILMS GROWN BY AR-ION BEAM PLASMA ENHANCED DEPOSITION. R. Hadzioannou, J. Feydt, R. Pascual, S. Thienhaus, T. Sterzl, B. Weitz, M. Moser Research Center Ceska, Bonn, GERMANY.

Shape memory alloys constitute a special materials category due to an inner structural transformation occurring under changing external parameters like temperature, strain, or mechanical forces which leads to a recovery of macroscopic deformation and to superelastic behavior. To date, bulk metals are employed - mainly the classical NiTi-based alloys - for applications in medical technology like tooth braces, cardiovascular stents, microgippers, or endoscopes. The martensitic transformation occurring in these materials is, however, of further practical interest, e.g., in miniaturized devices like electric or thermal switches. Therefore, thin films are currently under development opening up the possibility for lithographic microstructuring. In this context, we present a study demonstrating the capability for controlled shape memory film growth using molecular beam epitaxy for film deposition on up to four-inch substrates. In order to compare with previous results known for, e.g., sputtered NiTi-based films, NiTiCu alloy films were grown which are known to exhibit a martensitic transformation well above room temperature. After deposition, the films are amorphous and they crystallize upon in-situ heating at around 450°C as observed by RHEED. On subsequent cooling, the transformation into the metastable martensite structure occurs which is studied in detail by ex-situ through reversible thermal processing in a stress apparatus using a capacitance bending beam technique and by X-ray diffraction in combination with a specially designed heating stage capable to use a temperature detector. Remarkably, using molecular beam epitaxy technique the microstructure is very different compared to conventionally sputtered polycrystalline films: the crystallites are highly oriented with the austenite-orientation (001) orientation as well as the martensite-orientation (222)/(002) orientations as seen in the film normal (23°). The splitting of the martensite-orientation (222) peak indicates the presence of only two martensite variants. Stress measurements reveal a high ratio of recoverable stress even for films below 500 nm thickness. In situ STM images show a pattern of plateaus of distinct heights. The special orientation properties observed give rise to expect novel properties in such thin films, especially in regard to their superelastic behavior.

4:30 PM L12.4
DEPTH PROFILE OF STRUCTURE, STRAIN, AND COMPOSITION IN AN ANNEALED Al-Cu-Fe-Cr QUASICRYSTALLINE ALLOY. M.I. Damodaran, Center for Nanomaterials Science, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI. D. King, Technology Assessment and Transfer, Ann Arbor, MI. J. S. Zachariah, Air Force Research Laboratory, Wright-Patterson Air Force Base, OH. J. C. Bilello, Center for Nanomaterials Science, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI.

Quasicrystalline films were formed by RF sputtering from a powder composite target onto Inconel substrates, which produces a polynorphic nanoquasicrystalline grain structure, ± 2.5 - 10.0 nm. Subsequent annealing at 500°C for 4 hours, at base pressures of below 5x10^-7 Torr, fully devolved the quasicrystalline structure with decagonal phase predominating, except near the termination surface. Analysis by XPS indicated extensive oxygen incorporation at the surface. These results are correlated with structure and strain analysis via x-ray synchrotron grazing incidence x-ray scattering [GIXS]. By varying the incident angle, hence the x-ray penetration depth, the evolution of a crystalline second phase at the surface of the film has been detected. This layer is 30 nanometers thick. Residual strain analysis shows that this second phase induces a compressive residual stress of the order of 0.1%. From x-ray diffraction measurements of the full scan, a residual strain of ± 0.1% as measured from displacement of the major quasicrystalline diffraction peaks in the surface layers of the film. Authors thank both the USAFOSR for support, in part, of this work under a DARPA contract and USDOE for facilities used at Stanford Synchrotron Radiation Laboratory.

4:45 PM L12.5
DEPOSITION OF DIAMONDLIKE CARBON BY MAGNETIC ENHANCED INDUCTIVELY COUPLED PLASMA T. Mendez, P. Colpo, C. Ceccone, P. Leray, P.N. Gibson, D. Simma, and M. Rossi, European Commission-Joint Research Institute for Health and Consumer Protection, Ispra (VA), ITALY, P. Ranson, GREM-ESPEO, Universit d’Orl ans/CNRS, Orleans, FRANCE.

A novel inductively coupled plasma source (the Magnetic Plasma Enhanced ICP or MPE-ICP) designed and characterized in our laboratory was used for diamondlike carbon (x-CH) deposition. The MPE-ICP uses a magnetic field to concentrate the magnetic flux on the load (i.e., plasma) and hence very interesting features like high plasma density, good plasma uniformity and wide pressure range. The ICP sources are particularly interesting for the deposition of amorphous carbon since they offer the possibility to control independently the ion energy and/or the ion flux bombarding the substrate, in contrast to capacitively coupled discharge. Diamondlike carbon coatings were deposited with this source from CH4 and C2H2 precursors. The effect of precursor dilution by noble gases (He, Ne, Ar, Kr, Xe) as well as the effect of process parameters (power, pressure, etc.) were investigated. Langmuir probe measurements, optical emission spectroscopy and microwave interferometry were carried out in order to characterize the discharge. Mass spectrometry including ion density and ion energy distribution measurements have been carried out at the substrate holder surface in order to identify the impinging species on the growing film. Conting characterization including FTR spectroscopy, Raman spectroscopy, and X-ray reflectivity (XRR) were undertaken for various process parameters and the results were related to the films properties like hardness and intrinsic stress.

SESSION L13: FILM DEPOSITION, MICROSTRUCTURE, EVOLUTION AND INTRINSIC STRESS II
Chair: Jost Vielkind and Douglas Dougan
Friday, November 30, 2001
Room 304 (Hyne)

8:30 AM L13.1
EVOLUTION OF INTRINSIC STRESS DURING DEPOSITION OF POLYCRYSTALLINE AND AMORPHOUS THIN FILMS Jerold A. Flexo and Senn J. Henne, Sandia National Laboratories, Albuquerque, NM. Eric Chason, Brown University, Div. of Engineering, Providence, RI.

Understanding the fundamental origins of intrinsic stress has been an outstanding problem in non-epitaxial thin films for many decades. The problem is made difficult by the complex evolution of microstructure associated with the Volmer-Weber film growth mode. This talk will focus on stress evolution after film continuity is achieved. Polycrystalline films frequently exhibit compressive stresses due to capillary forces when deposition is initiated, followed by tensile stress generation during island coalescence to continuity, followed again by a long-term compressive stress after continuity, whose origins are still controversial. Using in situ wafer curvature measurement, we have observed additional stress evolution during deposition of thicker films, wherein film stress again reverses from compression back into tension. This has been observed in amorphous Si films, as well as in polycrystalline films of Ag and Ti. The origins of this tensile regime are also unclear. While consensus wisdom has held that grain growth can be responsible for tensile stress generation in continuous films, our experiments suggest that dynamic processes such as grain growth and densification are not contributing to the measured stresses. We will discuss the temperature dependence of stress generation, as well as the possibility of additional sources of stress generation. Multilayer growths of amorphous Si / amorphous Ge will also be discussed, as these shed additional light on the origins of intrinsic stress in continuous thin film systems. The work was performed at a laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.
9:00 AM L13.2
STRESSES DUE TO CHEMICAL POTENTIAL GRADIENTS IN NON-STOICHIOMETRIC OXIDE FILMS. Ramaswamy Krishnamurthy and Brian W. Shakleton, Division of Engineering, Brown University, Providence, RI.

Oxygen potential gradients across non-stoichiometric oxides can lead to compositional mismatch strains and residual stresses in the oxide. These strains will in turn affect diffusion mechanisms in the film. A general treatment of this problem is presented, and applied to relate mean curvature measurements to oxygen evolution from titania. A second, more complex example is doped ceria, which is used as an electrolyte in solid oxide fuel cells (SOFC). Large oxygen potential gradients occur across the electrolyte, from the anode side to the cathode side. The resulting residual stresses can cause mechanical stability problems in the PEN (positive cathode - electrolyte - negative anode) structure. Stress distributions at different operating temperatures and doping conditions and results for the evolution of the mean curvature are presented. Different mechanical constraint conditions are imposed on the PEN structure, and their effect on stress distributions in the electrolyte are addressed. A maximum operating temperature for mechanical stability is identified, and the effect of dopant content on this stability is assessed.

9:15 AM L13.3
TOWARDS A BETTER UNDERSTANDING OF TENSILE STRESS EVOLUTION AND STRESS GRADIENTS DURING THE GROWTH OF POLYCRYSTALLINE FILMS. Brian W. Shakleton, K.H.A. Lou, and Ashok Rajamani, Brown University, Division of Engineering, Providence, RI.

Previous treatments of tensile, intrinsic growth stresses in polycrystalline films have been based on the stress-free state assumed when neighboring islands first coalesce. Subsequent tensile stress is then assumed to arise when additional atoms are templated onto the atomic positions defined by the underlying strained lattice. Experimental data contradicts this picture will be presented. This work examines several different vapor-deposited films with low mobility carriers (e.g., carbides and nitrides), where significant tensile stresses are generated long after individual islands have coalesced into a continuous film. Film stresses were measured by wafer curvature. In some cases, free-standing films were also obtained by removing the substrate, where severe curvature away from the original substrate indicates substantial stress gradients, with the growth side in considerable compression relative to the interface side. A mechanical analysis was used to evaluate stress gradients in these films, based on the observed curvature measurements. These results were then analyzed with a kinetic model that describes tensile stress evolution during growth. In addition to explaining the experimental observations, this model also suggests a number of strategies that can be used to control stress gradients during the growth of polycrystalline films.

9:30 AM L13.4
PLANAR OXIDATION OF STRAINED SILICON SUBSTRATES. M.T. Lin, R.J. Jacobson, and T.J. Dolph, Dept. of Mechanical Engineering and Materials Science, Lehigh University, Bethlehem, PA.

We report here on a series of experiments in which relatively low levels of in-plane bending strain were applied to oxidizing silicon substrates. The experiments involved increasing oxide thickness in the ultrathin oxide regime. Both tensile and compressive bending resulted in roughly the same degree of thickness retardation, although compressive bending typically led to somewhat thinner oxides than did tensile bending. An explanation of the data indicates that the principal effect seems to occur in the very early stages of oxidation, with only minor effects on subsequent oxide growth. We hypothesize that the observed oxide thickness retardation is related to straining of the underlying silicon lattice at the oxidation front.

9:45 AM L13.5
PRESSURE ASSISTED CRYSTALLIZATION OF MoAl Thin FILMS. Gregory Fischer, Army Research Laboratory, Adelphi, Md., M. Leu Rudee, Graduate Program in Materials Science/Center for Magnetic Recording Research, U.C.S.D., La Jolla, CA; Vitali F. Nesterenko, Sastri Indranantra, Graduate Program in Materials Science/Department of Mechanical and Aeronace Engineering, U.C.S.D., La Jolla, CA.

The effect of hot isostatic pressure processing (HIP) on MoAl films has been compared to vacuum annealing for the purpose of obtaining substantial amounts of two-phase MoAl films under room conditions. Films were deposited by dc sputtering from both MoAlNc and MoAl targets. As-deposited films were nearly amorphous. Post deposition annealing in vacuum produced only small amounts of the ferromagnetic two-phase in films thinner than 200 nm. In all instances, regardless of substrate and sputtering target, the use of HIP in place of vacuum annealing increased the degree of crystallinity of the samples when compared to those annealed in vacuum. For the 100 nm samples deposited from the MoAlNc target, these changes in crystallinity were accompanied by changes in the Mo-H loops of the samples. MoAlNc HIP samples had improved magnetic properties compared to those of equal thickness annealed in vacuum. The 100 nm Hip sample sputtered from the MoAl target also showed an increase in moment, though the changes were not as dramatic as seen in the samples sputtered from the MoAlNc target. The 50 nm films from both targets also showed a change in crystallinity when compared to vacuum annealed samples. These films, unlike the 100 nm films, had ferromagnetic properties that were no better than those of the vacuum annealed samples. This suggests that while the 2 kbar of pressure used in this study assists in the formation of two-phase in 100 nm films, the appropriate pressure for forming two-phase in 50 nm films is yet to be determined.

10:30 AM L13.6
THERMOMECHANICAL BEHAVIOR OF THIN Ag FILMS ON SUBSTRATES. Shepard P. Baker, Krishnamoorthy Saha, and Jonathan Shu, Cornell University, Department of Materials Science and Engineering, Ithaca, NY.

Thin Ag films were deposited on Si substrates with various diffusion barrier and passivation layers. The thermomechanical behavior was studied using substrate curvature stress measurement methods during thermal cycles between room temperature and 450°C, and was found to be very sensitive to interface chemistry. Films exposed to nitride barrier and passivation layers formed voids, hillocks, and blisters, and exhibited unusual stress-temperature behavior during cycling that includes compressive plastic deformation under applied tensile stress, extension of the pre-existing voids and hillocks, and increasing stress with increasing temperature. Addition of a thin layer of Ti between the Ag film and the nitride encapsulant layers eliminated the voids, hillocks, and blisters, and generated the open stress-temperature hysteresis loops that are typical of other passivated thin metal films. Stress relaxation measurements were conducted at different temperatures in order to identify plastic deformation mechanisms. Adhesion and dislocation- and diffusion-mediated deformation mechanisms and their dependence on interface conditions are discussed. Thermomechanical behavior is modeled using a deformation mechanism map approach and the behavior of Ag films is compared to the behavior of other refractory metal films. All are seen to show large variations in plastic deformation behavior with small changes in interface conditions.

11:00 AM L13.7
IN-SITU MEASUREMENTS OF GROWTH STRESSES IN ALUMINA FILMS USING SYNCHROTRON RADIATION. E.D. Specht, P.E. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN; P. Zarchin, University of Illinois at Urbana-Champaign, APS-UNICAT, Argonne, IL; B.A. Plet, K.L. More, Oak Ridge National Laboratory, Oak Ridge, TN.

While growth stresses in oxide films formed by high-temperature oxidation have long been discussed and modeled, there has been considerable disagreement regarding their magnitude and importance relative to the stresses generated during temperature changes by the differences in the coefficients of thermal expansion between the substrate and oxide product. The present work reports the use of focused monochromatic synchrotron radiation to measure stresses in growing alumina films during high-temperature air exposures of Fe and Ni-base alloys at temperatures in excess of 1000°C. The excellent time resolution afforded by the use of synchrotron undulator radiation to measure strains in the oxide revealed, in some cases, rapid changes in stress caused by film growth and relaxation processes. Research sponsored by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U. S. Department of Energy (DOE), under contract DE-AC05-10OR22725 with UT-Battelle, LLC. The UNICAT facility at the Advanced Photon Source (APS) is supported by the Univer of Illinois at Urbana-Champaign, Materials Research Laboratory (U.S. DOE), the State of Illinois-Illinois-HEW (NSF), the Oak Ridge National Laboratory (U.S. DOE), the National Institute of Standards and Technology (U.S. Department of Commerce) and UOP LLC. The APS is supported by the U. S. DOE, Basic Energy Sciences, Office of Science under contract No. W-31-109-ENG-38.

11:15 AM L13.8
CONTROLLED STRESS REFRACTORY METALLIZATIONS. Jim Golecki and Margaret Eagan, Honeywell International Inc. (formerly AlliedSignal Inc.), Corporate Materials Laboratory, Morristown, NJ.
Refractory metals, such as iridium and rhodium, are highly electrically conductive and can be used as current-carrying thin-film metallic lines. Their chemical inertness further enables their use at relatively high temperatures. However, due to the high elastic modulus of such metals, a residual tensile stress of 300 to over 1,000 MPa is measured in evaporated thin films. We present novel results evidencing complete control over both the magnitude and the sign of the residual stress in such refractory thin films. The metallic layers are deposited by means of ion-beam-enhanced physical vapor deposition and both electrical resistivity and stress are controlled. Controlling the stress in this manner enables achieving thicker films. Films with near-zero residual stress have been produced.

11:30 AM L13.9
THermal STABILITY OF SELF-SUPPORTED METALLIC MULTILAYERED THIN FILMS. Amir Mossa, Harriet Kung and Richard G. Hoglund, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Metallic multilayers, composed of alternating layers of soft metals, typically possess strengths approaching the theoretical limit when the bilayer periods are on the order of a few nanometers. The morphological stability and strength retention following elevated temperature exposure or thermal cycling will be crucial in exploiting the unusual properties of these nanoscale materials in advanced engineering applications. We have investigated the effects of elevated temperature vacuum annealing on the morphological stability and mechanical properties of sputter deposited Cu-Nb multilayered thin films with bilayer periods ranging from 30 to 600 nm. Both as-deposited and cold-rolled self-supported films were annealed at temperatures in the range of 500 to 800°C. Even after 800°C / 1 hour anneal, the continuity of the layered structure is maintained and the bilayer periods are unchanged for all samples, except the 30 nm bilayer period multilayer that is stable only at temperatures ≤ 600°C. For bilayer periods > 30 nm, long-term stability is studied at 700°C. The in-plane grain sizes in both Cu and Nb layers coarsened but were anchored at triple points preventing further growth. For a constant bilayer period, the effect of increasing the in-plane grain size on the multilayer hardness is studied. After annealing, the layers are observed to be offset by shear along a vertical plane at the triple point junctions that have equilibrium groove angles aligned in a zig-zag pattern. The mechanism that drives this morphological evolution is discussed. This research is funded by DOE-OBES.

11:45 AM L13.10
PLASTICITY ON A SUBMICRON SCALE: HOW DO INDIVIDUAL GRAINS INTERACT IN A POLYCRYSTALLINE THIN FILM? R. Spolenak1,*, N. Tanuma2, B. C. Valek3, M. D. Morris4, W. L. Brown5, A. A. MacDowell6, R. S. Celestre7, H. A. Padmore8, J. C. Braiman9, B. W. Bitterman9,10 and J. R. Patel9,10, 9Arete Systems (formerly of Bell Laboratories, Lucent Technologies), Murray Hill, NJ, 1Colorado State University, Boulder, CO, 2Dept. Materials Science & Engineering, Stanford University, Stanford, CA, 3SHRI/SLAC, Stanford University, Stanford, CA.

When the grain size in thin polycrystalline metallic films is roughly of the order of the film thickness, the stress state in one grain strongly influences its neighboring grains; the constraint of the substrate does not dominate. In this paper we report measurement of this interaction at a submicron level. A quasi-uniaxial bulge test on a rectangular membrane was performed in-situ at the microdiffraction beamline at the Advanced Light Source, Berkeley. The submicron focus of the wide synchrotron allowed for the accurate determination of the 3D deviatoric strain tensor of every single grain. In this way the plastic behavior of a one micron thick sputtered Cu film could be investigated up to an average total strain of 0.15% at room temperature. As the film was already at yield stress in tension prior to bulging due to a thermal anneal after film deposition, plastic behavior was observed from the beginning of bulging. The distribution of stresses among the grains ranged even into the compressive regime although the film as a whole was in tension. The width of the distribution that was established by the pre-bulging heat treatment was retained throughout the bulge experiment. The actual yield stress of every single grain was found to depend on its crystallographic orientation, its size and the interaction with its neighbors.