SYMPOSIUM L

Thin Films-Stresses and Mechanical Properties IX

November 26 – 30, 2001

Chairs

Cengiz Sinan Ozkan
Mechanical Engineering Dept
Univ of California-Riverside
Bourns Hall, Rm A305
Riverside, CA 92521
909-787-2417

L. B. Freund
Brown Univ
Box D
Providence, RI 02912-9104
401-863-1476

Robert C. Cammarata
Dept. of Materials Science & Engineering
Johns Hopkins Univ
102 Maryland Hall
Baltimore, MD 21218-2689
410-516-5462

Huajian Gao
Max Planck Inst for Metals Research
Stuttgart, D-70174 GERMANY
49-711-2095-519

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SESSION L1: STRAIN RELAXATION AND STRENGTHENING MECHANISMS

Chairs: Robert C. Cammarata and Guanshui Xu
Monday Morning, November 26, 2001
Room 304 (Hynes)

8:30 AM *L1.1

SOLID SOLUTION STRENGTHENING IN Pt-Ru THIN FILMS.

Richard P. Vinci, Lehigh University, Dept of Materials Science and Engineering, Bethelehem, PA.

The general effects of solid solution alloying on microstructure development and strength are well known for bulk metals. However, the mechanical properties of solid solution alloy thin films have not been heavily 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 a 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 wt% 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. The results should also be relevant for other FCC materials such as Cu and Al.

9:00 AM L1.2

X-RAY DIFFRACTION ANALYSIS OF STRAIN INDUCED DURING THERMAL CYCLING OF A THIN ALUMINUM BI-CRYSTAL FILM.

D.E. Nowak, S.P. Baker, Cornell University, Department of Materials Science and Engineering, Ithaca, NY; K. Finkelstein, Cornell University, Cornell High Energy Synchrotron Source, Ithaca, NY; E.A. Stach, K. Balzuweit and U. Dahmen, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA.

Heteroepitaxial films of aluminum bi-crystals grown on silicon provide a model system in which to study thin film plasticity of polycrystalline metals. 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 $\sin^2(\psi)$ 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 discussed explicitly.

9:15 AM L1.3

DIRECTION DEPENDENT GRAIN INTERACTION MODELS FOR THE DIFFRACTION STRESS ANALYSIS OF THIN FILMS.

Udo Welzel, Peter Lamparter, Eric J. Mittemeijer, Max Planck Institute for Metals Research, Stuttgart, GERMANY; Matteo Leoni, Universita 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 stresses) are known. Usually, the diffraction elastic constants are calculated from single crystal elastic data 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, Neerfeld-Hill and Eshelby-Kroener, can be inappropriate for the analysis of strain data 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 Vook & 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 stress in 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). Traditional models are shown to extract erroneous stress values from diffraction strain data (errors as large as 50%) when the grain interaction in the thin film is direction-dependent. [1] M. van Leeuwen, J.-D. Kamminga, E.J. Mittemeijer, J. Appl. Phys. 86 (4), 1904-1914 (1999). [2] M. Leoni, U. Welzel, P. Lamparter, E.J. Mittemeijer, Phil. Mag. A. 81 (3), 597-623 (2001). [3] R.W. Vook, F. Witt, J. Appl. Phys. 36 (7), 2169-2171 (1965).

9:30 AM L1.4

MECHANISM OF CATASTROPHIC STRESS RELIEF IN AlGaN/GaN.

K.E. Waldrip, J.A. Floro, D.M. Follstaedt, S.R. Lee, J.A. Hunter, S.J. Hearne, D.D. Koleske, A.A. Allerman, J.J. Figiel, T.M. Kerley, Sandia National Laboratories, Albuquerque, NM.

The ability to produce short wavelength optoelectronic devices based on III-nitrides has been severely limited due to the residual tensile stres of AlGaN grown on GaN. Tensile-strained (0001)-oriented wurtzite heterosystems 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 cracking in AlGaN occurs prior to the generation of misfit dislocations, which subsequently nucleate at crack tips and propagate throughout the film. Early in situ stress measurements revealed that up to 100% of the tensile stress was relieved in approximately 3 seconds during the growth of AlGaN (at 0.9um/hr) at a thickness of 350-400nm. Cracking could only account for $\sim$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 $Al_{0.2}Ga_{0.8}N$ were grown on top of 1.5um of GaN by metalorganic chemical vapor deposition on sapphire substrates. The stress evolution of AlGaN was monitored in situ via a wafer curvature laser deflectometry technique. AlGaN films of varying stages of stress relaxation (or, thickness) were studied. Stages investigated include immediately pre- and post-catastrophic relaxation, and at earlier stages of relaxation. The density and types of dislocations generated through each stage of relaxation have been analyzed 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 L1.5

INTERNAL STRESS ENHANCEMENT IN POLYTETRAFLUOROETHYLENE/Al NANO-MULTILAYERS.

Eiji Kusano, Naoto Kikuchi, Akira Kinbara, Advanced Materials Science Ctr. Kanazawa Inst. of Technol., Matto, JAPAN.

By multi-layering polytetrafluoroethylene (PTFE), which demonstrates a low surface energy of about 18mJ/m^2, with metal or metal compound, a film with high interface energy can be fabricated. In this paper, PTFE/Al nano-multilayers have been prepared to study 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 multilayers 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 0.07mm 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 also enhanced with decreasing modulation period. The interface free energy accumulated in a multilayer is estimated to be about 50-100 J/m^2. This equates 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. Shu, Shefford 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 (thermomechanical behavior seen in one cycle can persist into subsequent cycles to higher temperatures). The details of this set of unusual behaviors are yet unknown, but we have proposed a mechanism whereby dislocations stored in the film during one part of the temperature cycle are recovered in another, which can qualitatively account for many of the observed behaviors. We have recently developed a UHV system in which Cu films can be prepared and thermomechanical stresses 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.W. Yu, Frans Spaepen, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

Relaxation of the stress in free-standing metallic thin films was investigated using a microtensile tester with in-situ strain measurement by optical diffraction [1,2]. Testing of free-standing thin films has the advantage of eliminating possible effects of the substrate on dislocation motion, as well as allowing variation of the stress indepent of temperature. The relaxation behavior in copper films at room temperature can be described by obstacle-controlled dislocation glide. The results will be compared with those from measurements of copper films on a silicon substrate. Measurements at different temperatures and on multilayers are underway. [1] J.A. Ruud, D. Josell, F. Spaepen and A.L. Greer, J. Mat. Res. 8, 112 (1993). [2] H. Huang and F. Spaepen, Acta Mat. 48, 3261 (2000).

11:00 AM L1.8

RESIDUAL STRAIN/STRESS DETERMINATION IN TEXTURED $La_{0.67}Sr_{0.33}MnO_3$ THIN FILMS. Lamartine Meda, Saleh Hayek, Hamid Garmestani, FAMU-FSU College of Engineering and MARTECH National High Magnetic Field Laboratory/CeNNas Tallahassee, FL.

X-ray diffraction (XRD) technique was used to evaluate the residual strain/stress in textured $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) thin films. The films were deposited by liquid delivery metal-organic chemical vapor deposition (LD-MOCVD) on single crystal substrates. The conventional $\sin^2\psi$ and 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 bi-axial state of stress. ϵ_Ψ vs. $\sin^2$ plots resulted in either a curve or an oscillation. A correlation between residual strain/stress, lattice mismatch strain, and microstructure is observed.

11:15 AM L1.9

MICROSCOPIC PHENOMENOLOGY OF PLASTIC DEFORMATION IN POLYMER-METAL LAMINATES. Ruchi Rastogi, Willem-Pier Vellinga, Han Meijer, 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 those mechanisms in a laminate. As plastic deformation may involve localisation 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, results of which will be presented. These involve in-situ measurements during uniaxial tensile deformation of PET-steel and PP-steel laminates using optical microscopical techniques, mainly refelction birefringence microscopy, as well as ex-situ CLSM and AFM results. Dependence of 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 MULTI-LEVEL METAL INTERCONNECT STRUCTURES ON Si SUBSTRATES. Tae-Soon Park, Ming Dao, Subra Suresh, Massachusetts Institute of Technology, Dept of Materials Science and Engineering, Cambridge, MA; Daniel Pantuso, Sadasivan Shankar, 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 and dielectric line behaves like an individual thin film in equibiaxial plane stress state. As the line aspect ratio increases, on the other hand, the line structure is homogenized into a single composite layer with different effective thermoelastic properties along and across lines due to geometrical anisotropy. Effects of passivation geometry and stiffness on the stress evolution in the lines are 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 Eshelby-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 metalization levels and for analyzing effect of the upper level line arrangements involving aligned and mis-aligned 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, Mesa, 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 adhesion-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 across the wafer 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 Hysitron Triboindenter and the continuous stiffness option (CSM) of the Nanoindenter 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

Chairs: Zhigang Suo and Jacob N. Israelachvili
Monday Afternoon, November 26, 2001
Room 304 (Hynes)

1:30 PM *L2.1

FLOW STRESSES AND DISLOCATION MECHANISMS IN EPITAXIAL AND POLYCRYSTALLINE THIN METAL FILMS. G. Dehm, H. Edongue, Max-Planck Institut für Metallforschung, Stuttgart, GERMANY; B.J. Inkson, Department of Materials, University of Oxford, Oxford, UNITED KINGDOM.

Flow stresses of thin metal films (thickness $\leq 1\mu$m) 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_2O_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_2O_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 Nix-Freund model. In contrast, the TEM results imply that this mechanism cannot account for high flow stresses in polycrystalline metal films on diffusion-barrier-coated Si substrates, because we observed that interfacial dislocations disappeared at crystalline/amorphous interfaces.

2:00 PM L2.2
ACOUSTIC EMISSION MONITORED PLASTICITY INITIATION UNDER CONTACT LOADING. Natalia Tymiak, Antanas Daugela, Thomas Wyrobek, Hysitron, Inc., Mineapolis, MN; and William Gerberich, Dept of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN.

The present study utilizes AE monitoring for the evaluation of yield point phenomena on (100) W surfaces with several nm of native oxide and freshly cleaved (100) MgO. For MgO, previous studies suggest dislocation nucleation onset at the start of the yield excursion. In contrast, a more complex 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 indenter tip eliminates sample size effects and provides a greatly enhanced detection resolution for loads below 1 mN (Hysitron, Inc.). Synchronization error between load displacement and AE data acquisition less than 10 microseconds enables a precise time scale correlation between AE and indentation curves. AE transients precede yield excursions by time intervals ranging 0.01-0.09 seconds for both evaluated materials. For indentation depths prior to excursion ranging from 6 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 cracking or oxide film delamination for W. While 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 $\Sigma = 3$ BOUNDARIES IN EPITAXIAL GOLD THIN FILMS. Gene Lucadamo 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 *insitu* hot stage transmission electron microscopy (TEM), we have studied dislocations at the junctions between orthogonal $\Sigma = 3$ {111} and $\Sigma = 3$ {112} faceted grain boundaries. Dislocation arrays were observed with $\mathbf{b} = 1/6 < 112 >$ 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 (SGBDs) and that the motion of the emitted dislocations is coupled with the migration of the {112} facet junctions. The SGBDs are present to accommodate a rotational misorientation between adjacent crystallographic domains. This interpretation was corroborated by measurements showing that the domains in the vicinity of the facet junctions deviate slightly ($< 1^\circ$) from the exact $\Sigma = 3$ coincident-site-lattice (CSL) orientation. We propose that the driving forces for this motion are provided primarily by the climb stress between the SGBDs arising from the discontinuous 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-AC04-94-AL85000.

2:30 PM L2.4
FUNDAMENTAL CRITERIA FOR THE PROPAGATION OF TELEPHONE CORD BUCKLES BENEATH DLC FILMS ON GLASS SUBSTRATES. Myoung-Woon Moon, Kyu 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 Chung, Kwang-Ryeol Lee, Korea 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 Focussed Ion Beam (FIB). Using AFM with 2nm resolution, 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 have been related to differences in local 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 Dunn, Katharine Dovidenko, Anna W. Topol and Alain E. Kaloyeros, U Albany Institute for Materials, The University at Albany-SUNY, Albany, NY.

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 250°C-500°C range on an AlTiO/InSnO/glass stack. Selected samples were then subjected to a post-deposition anneal in H_2S/Ar 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 films consisted of columnar grains whose column axis was parallel to the growth direction, and which widened laterally through the thickness of the films. For the as-deposited 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 microtwins, 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 Bragg-Brentano X-ray Diffraction scans. The correlation between the defect microstructure and the electrical and mechanical behavior of these phosphor films is discussed.

3:30 PM L2.6
HREM OBSERVATIONS OF THE RELAXATION OF GRAIN BOUNDARIES IN {110} GOLD MAZED BICRYSTAL THIN FILMS. Tamara Radetic and Urlich Dahmen, NCEM, Lawrence Berkeley National Lab., Berkeley, CA.

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 orientations. All grain boundaries are of the type $\Sigma 99$ 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 fcc 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 $\Sigma 99$ grain boundaries in Au are reported and discussed in terms of mechanisms of formation and anisotropy 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-ACO3-76SFOOO98.

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

The stresses that exist in thin film metallizations can be significantly higher than those at 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 thermo-mechanical 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 directly 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 room 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 volumes correspond to apparent dislocation pinning distances of less than 8% of the film thickness. Thermal cycling in both 200 keV and 1 MeV TEMs revealed that dislocations move rather smoothly during the initial stages of cooling from 500°C. However, below approximately 200°C, the dislocations tangle and constrain one another, resulting in sporadic motion. Moreover, unexpected dislocation activity on a plane parallel to 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 persistent 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[a,b], H. Liu[c], Z.Q. Zeng[d], C.Y. Li[b], Joseph Xie[a,b]; [a]Advanced Materials, Singapore-MIT Alliance, SINGAPORE; [b]DISC Dept, Institute of Microelectronics, SINGAPORE; [c]Rodolph Technologies, Inc., USA; [d]School of Materials Engineering, Nanyang Tech. Univ., SINGAPORE.

Effects of the microstructure and stress status of Tantalum (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 as-deposited Cu/Ta/Si samples were annealed in N_2 ambient at 500°C, 550°C, 600°C and 650°C, respectively, for 30 minutes to evaluate the barrier performance. Microstructures and residual stress of the barriers were investigated using MetaPULSE, Film stress measurement (FSM) and Transmission electron microscopy (TEM). Four-point probe (FPP), X-ray diffractometry (XRD) and Rutherford backscattering spectrometry (RBS) were used to study the failure behavior and mechanism. Under different growth conditions, microstructure of the Ta barriers would vary, i.e., barrier layers with different amount of 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 sample with more interfaces was higher than that of the other samples. And according to XRD spectra, the failure could be confirmed by the appearance of new compound - Cu_3Si. 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 hold up the diffusion through 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 SPUTTERED THIN COPPER FILMS. Brando Okolo, Udo Welzel, Peter Lamparter, Thomas Wagner, Eric J. Mittemeijer, 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 and silicon nitride, at a temperature of 373K. 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 characterised by atomic force microscopy (AFM) and X-ray reflectivity measurements. The (mean square) roughness of the substrate as determined by AFM measurements was found to be 0.43nm for the silicon oxide surface and 0.36nm for the silicon nitride surface. Texture analysis, performed by X-ray diffraction pole figure measurements, showed that the layers have a {111}-fibre texture with minor additional texture components. In particular, a {511}-fibre texture component resulting from twinning was observed. Texture sharpness increased with decrease in substrate roughness. The twin fault 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 100MPa was observed, by employing the X-ray diffraction $sin^2\psi$-method, in the film on the smoother substrates whereas the stress was 200MPa 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 correlation 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. Zhaohua Feng, Edward G. Lovell, Roxann L. Engelstad, University of Wisconsin, Computational Mechanics Center, Mechanical Engineering Department, Madison, WI; 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 Mathews-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. Axisymmetric 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 stresses were simulated. Other 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 L3: POSTER SESSION
Chairs: Cengiz Sinan Ozkan and Robert C. Cammarata
Monday Evening, November 26, 2001
8:00 PM
Exhibition Hall D (Hynes)

L3.1
ADAPTIVE PROTOCOL FOR ROBUST ESTIMATES OF COATINGS PROPERTIES BY NANOINDENTATION. Nigel M. Jennett[a] and Andrew J. Bushby[b]; [a]National Physical Laboratory, Materials Centre, Teddington, Middlesex, UNITED KINGDOM; [b]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 indentation 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 NANOINDENTATION STUDY OF DROSOPHILA MELANOGASTER DEVELOPMENT. Antanas Daugela, Hiroshi Kutomi, Hysitron Inc; Michael Kohane, Durance Co; Jerzy T. Wyrobek, Hysitron Inc, Minneapolis, MN.

A nanometer scale quantitative quasi-static indentation technique has a potential to provide a simple mechanical characterization of soft biological tissues, helping to bridge a gap between destructive spectrometric analysis and model based diagnostics. This is a challenging task for the existing instrumentation. A Scanning Probe Microscopy (SPM) based nanoscale indentation yields errors associated with contact area changes due to the cantilever bending. On the other hand, quantitative nanomechanical test instruments have difficulties with matching the sample contact stiffness as well as incomplete data evaluation algorithms. Classical quantitative nanoindentation algorithms are suited to fit elasto-plastic contact phenomena while soft biological samples exhibit adhesive visco-elastic contact behavior. In this study a quantitative nanoindentation technique was used to monitor developmental changes of live Drosophila melanogaster samples. Drosophila melanogaster has been the object of the study for many years by geneticists and is a standard sample for biologists. A commercially available nanomechanical test instrument interfaced with SPM scanner was used to derive reduced elastic modulus from experimental loading-unloading curves. Extrapolation of loading-unloading curves was curried out in order to compensate surface approach. Adhesion of the contact was compensated using the JKR model based data fitting. The model based fitting was enhanced by two-point angular contact measurement. A case study was carried in-vivo on etherized Drosophila melanogaster samples for two lifecycle stages, i.e. larvae and pupae. Measurements were taken at three locations (posterior, mid and anterior regions) for (1) ether-immobilized larvae and non-etherized pupae and (2) for the ether-immobilized larvae, over time, during larval development. Discrete ranges of reduced elastic modulus, complex elastic modulus and viscous energy were monitored for larvae and pupae at the completion of metamorphosis. Variations in reduced elastic modulus (0.2 - 0.6MPa) were correlated to the different stages of metamorphosis.

L3.3

NANOSCRATCH AND NANOWEAR CHARACTERISTICS OF ULTRA-THIN DLC FILMS. Dehua Yang, Tony Anderson, Richard Nay, and Thomas Wyrobek, Nanomechanics Research Laboratory, Hysitron, Inc., Minneapolis, MN.

Increasing the recording density of hard drives demands reduction of the flying height of magnetic recording heads and reduction of the thickness of the protecting DLC layers. This requires improved design of air bearings. Also, the reduced thickness of the DLC layer exposes new challenges in evaluating the ability of the layer to protect underlying magnetic materials. Nanoindentation had played a vital role in characterization of DLC films over the past several years when film thickness was in the range of a few tens of nanometers. However, the recent trend of using less than five nanometer protecting layers has caused problems with interpreting the nanoindentation testing results. As an alternative, nanoscratch and nanowear have drawn more attention from researchers in this field. The first reason is that nanoscratch and nanowear can be used as a direct simulation of what is happening in the processes of head slider start, stop, and crashing. The second reason is that the difficulty in interpreting nanoindentation data can be avoided. In this paper, the experimental and analytical results of nanoscratch and nanowear applied onto 3-30 nm DLC thin films will be reported. The applicability and suitability of nanoscratch and nanowear tests to the characterization of DLC thin films will be discussed, based on the experimental results and data on wear rate, friction coefficient, and critical load of interfacial failure. Emphasis has been given not only to the thin film itself, but also to the interface with the underlying layer; therefore, the effects of multi-layer structure, as well as lubricant will be covered in this paper. Further, in-situ images with nanometer resolution of the scratched and worn surfaces are presented for the purposes of studying the damage and failure mechanisms at film surfaces and interfaces.

L3.4

POSTBUCKLED SQUARE THIN FILM MEMBRANES UNDER DIFFERENTIAL PRESSURE. Torsten Kramer, Thilo Brenner, Oliver Paul University of Freiburg, IMTEK, Microsystem Materials Laboratory, GERMANY.

We report on the extraction of mechanical material parameters from the load-deflection response of postbuckled square membranes under differential pressure. We investigated micromachined PECVD silicon nitride (SiN) membranes with side lengths a between 101 μm and 3050 μm, thickness $h = 1.9\ \mu$m, and prestrain $\epsilon_0 = -1.6 \times 10^{-3}$. As a function of the pressure P, the deflection profiles $w(x,y)$ of large membranes undergo various symmetry transitions. From these we extracted Young's modulus $E = 150$ GPa of the membrane material. Under sufficiently large loads, the deflection profiles of all membranes show the same symmetries under reflection and rotation as a square. If the pressure is reduced below a critical value, some symmetries disappear. As an example, membranes with $a = 1040\ \mu$m lose their reflection symmetries at $P =$ 250mbar and settle into a postbuckling profile symmetrical only under rotation by $\pi/2$. In contrast, larger membranes with $a = 2060\ \mu$m settle into a butterfly-like state with two remaining reflection symmetries and no rotation symmetry. A small negative differential pressure drives these membranes into a state with rotational symmetries only and without reflection symmetries. This state is identical to that found immediately after the micromachining, 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 ϵ_0 and E of the investigated SiN thin film. Secondly, it was found that the membrane response is universal in the sense that w/h depends only on the reduced dimensionless coordinates x/a, y/a, Poisson's ratio ν, reduced prestrain $\epsilon_0 a^2/h^2$ and reduced pressure $Pa^4(1-\nu^2)/Eh^4$. The method and results can therefore be carried over to the characterization of all compressively prestressed thin films.

L3.5

SCRATCH RESISTANCE AND MECHANICAL PROPERTIES OF POLYMER FILMS. C.D. Eisenbach, E. Klinke, 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 tester. 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 (relaxation) after relief. The combination of the surface viscoeleastic 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 gloss loss of a multi-scratched coatings surface. The implication of the correlation of the optical appearance of the surface of an organic coating with its mechanical and viscoelastic properties for the evaluation of the coatings performance will be discussed. This work was supported by the German Federation of Industrial Cooperative Research Associations “Otto von Guericke” e.V. (AiF) (Grant No. 11806) and the companies Audi AG, DaimlerChrysler 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. Stahley, and C.G. Pantano, Materials Research Institute & Department of Materials Science and Engineering, The Pennsylvania State University, University Park, PA; D.H. Yang, T. Anderson and L. Kuhn, Hysitron, Inc., Minneapolis, MN.

Tin oxide thin films on glass are used in a variety of applications ranging from transparent conductors to coupling layers to wear resistant 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 composition 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 Hystrion 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 ramping load scratch test. Critical loads were determined by the ramp scratch test and multi-slide nanowear tests. Below the critical load no significant wear is observed. Above the critical load however, the wear increases sharply. Discussion on the thickness dependence of and the effect of surface chemistry on the mechanical properties and were made.

L3.7

ON THE RELATION BETWEEN MORPHOLOGY AND ELASTIC PROPERTIES IN AMORPHOUS COLUMNAR THIN FILMS. Thomas J. Yurick, Jr., Pedro C. Andia, Charles Randow, Francesco Costanzo, 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,

- there is an increasing interest in model-based simulation (MBS) for the design of engineering structures (including nanostructures), and
- increasing computer speeds are beginning to make MBS an effective design tool capable of bridging the nanoscale with the continuum scale,

has made it increasingly important to understand how the nanostructure of a thin film impacts its properties at all length scales. The authors have developed the capability to determine the mechanical properties of thin films with amorphous nanostructure by combining molecular dynamics, *i.e.*, position of particles (*e.g.*, atoms or molecules) and their interatomic potential(s), with continuum mechanics principles. This work concerns the application of this capability to determine mechanical properties, such as stability and distributions of elastic moduli and residual stresses, to thin films grown via physical vapor deposition (PVD) under conditions in which the deposition angle of the incident species is *not* normal to the substrate. Deposition in this manner results in an amorphous thin film with a porous, columnar nanostructure, whose morphology depends primarily on the deposition angle. In this presentation, we will focus on the exploration of the relationship between the deposition angle in PVD and the resulting mechanical properties of the film for important species such as MgF_2 and GaN. The films are created via molecular dynamics simulations using potentials available in the literature. The resulting mechanical properties are then obtained by an approach that bridges length scales from the discrete to the continuum.

L3.8

THE SCALE-SENSITIVE MECHANICAL RESPONSE OF SiC-ON-Si COATINGS STUDIED BY NANOINDENTATION TECHNIQUES.

R. Eva Gutierrez, Isabel Arce Garcia, Trevor F. Page, University of Newcastle, Materials Division (MMME), Newcastle upon Tyne, UNITED KINGDOM.

As part of our aim of determining how nanoindentation techniques can optimally be used to determine a wide-range of mechanical property data and mechanical response mechanisms of coated systems, we have continued our study of very thin (<200nm)coatings on glass and also studied a range of epitaxial 3C-SiC coatings on (100) and (111) Si. These coatings range from 10 nm to 1000 nm in thickness. Besides calculating the variation of E and H with indenter displacement in these systems, detailed analysis has been made of load-displacement (P-δ) curves to identify the detailed effects of load support by the coating. Thus, the effect of coating thickness and deformation scale on the maximum displacement achieved (compared to the substrate alone), the amount of elastic recovery observed (compared to the substrate alone) and the critical load at which the reverse undensification transition (pop-out) in silicon occurs. In addition, analysis of P vs. δ^2 and P vs. S^2 have been made in order to look at transitions in deformation responses between the coating and substrate with increasing scale, and also to explore the detailed effects of the variation of both E and H with displacement. We have also used the new “stiffness ratio” technique to compare E and H data with that obtained by the usual Oliver & Pharr technique. A Hysitron has been used to explore responses at very low loads. High-resolution SEM techniques, together with SEM-SACP techniques have been used to explore the deformation of fracture modes observed and to give further insight into the nanoindentation observations. Overall, our observations provide a very detailed investigation of this particular coated system.

L3.9

STRESS HYSTERESIS AND MECHANICAL CHARACTERIZATION OF PLASMA-ENHANCED CHEMICAL VAPOR DEPOSITED DIELECTRICS.

Jeremy Thurn, Robert F. Cook, University of Minnesota, Dept of Chemical Engineering and Materials Science, MN; Mallika Kamarajugadda, Laura C. Stearns, Seagate Technology, Bloomington, MN.

Three plasma-enhanced chemical vapor deposited (PECVD) dielectric films pertinent to microelectronic-based applications were examined for thermo-mechanical stability. All three films—silicon oxide, silicon nitride, and silicon oxy-nitride—showed significant permanent non-equilibrium changes in film stress on thermal cycling and annealing. The linear relationship between stress and temperature changed after the films were annealed at 300°C, representing a structural change in the film resulting in a change in coefficient of thermal expansion and/or biaxial modulus. A double-substrate method was used to deduce both properties before and after the anneal of selected films and the results compared with the modulus deconvoluted from the load-displacement data from small-scale depth-sensing indentation experiments. Infrared spectroscopy (IR) and Rutherford backscattering spectrometry (RBS) were used to study the bonding nature and composition of the films and correlated with the film stress and mechanical properties.

L3.10

EFFECT OF PRESSURE ON THE STRUCTURAL AND ELECTRONIC PROPERTIES OF THIN FILMS OF CARBON-BASED MATERIALS.

S.C. Sharma, B. Ha, J.H. Rhee and Y. Li, Dept of Physics and Materials Science & Engineering Program, University of Texas at Arlington, Arlington, TX.

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 and uv photoemission spectroscopies. In addition to thin films, we also present data on powder as well as on 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 by using x-ray diffraction, Raman scattering, photoemission spectroscopy, and diamond anvil cell. We discuss 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-FG03-00ER45840/A000 and Welch Foundation, Houston, Texas.

L3.11

STRUCTURAL PROPERTIES OF CARBON NITRIDE FILMS DEPOSITED BY REACTIVE PULSED LASER DEPOSITION TECHNIQUE.

A.R. Phani, J.E. Krzanowski, 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 ground pressures of nitrogen. Si(111) substrates are used in the present investigation. Deposited films are uniform and shows good adhesion to the substrates The deposition rates depend on laser fluence, back ground pressure, and target substrate distance. The nitrogen concentration in the deposited films increases with increasing back ground nitrogen gas pressure and laser fluence. Fourier transform infra red 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 revealed that the deposited films have shown oriented microcrystalline structure at relatively high 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 (POLYCIDE) FILM MEASURED BY X-RAY DIFFRACTION.

Joshua Pelleg, Eyal Elish, Ben Gurion University of the Negev, Dept of Materials Engineering, Beer Sheva, ISRAEL.

Stresses in chemical vapor deposited polycide tungsten silicide (poly -Si/WSi_x) were evaluated at each stage of the fabrication. The individual layers of the Si/SiO_2/Poly -Si/WSi_x/Poly -Si multilayer structure, were deposited sequentially on separate wafers and subjected to X-ray diffraction analysis in the as deposited and annealed conditions to determine the changes in strain occurring in WSi_x. Samples cut from wafers containing all the layers were either heat treated at 1193K for 30 min or capped with a 25nm thermal oxide and the strain in the WSi_x film was also analyzed by X-ray diffraction. The change in strain of the WSi_x layer, following each step of the fabrication process, was evaluated by the lattice parameter variation of the c axis. The stress is affected by the layers of the multilayered film. An as deposited poly-Si layer on top of WSi_x, reduces its stress, since it introduces a compressive component, which further decreases upon annealing. It also maintains a Si supply at the poly-Si/SiO_2 interface, thus, eliminating Si outdiffusion during heat treatment in an oxygen containing ambient. Capping the system by a thin oxide layer modifies the stress pattern of the WSi_x which becomes compressive. Average microstrains in WSi_x, 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.

Jozef Keckes, Erich Schmid Inst and Inst of Metal Physics, Leoben, AUSTRIA; Stephan Six, Axel Wenzel, Inst of Physics, Augsburg, GERMANY; Robert Averbeck, Infineon Technologies AG, München, GERMANY; Jürgen. W. Gerlach, Bernd Rauschenbach, Inst für Oberflächenmodifizierung, Leipzig, GERMANY.

X-ray diffraction is used to characterise residual stresses and unstressed lattice parameters in a variety of heteroepitaxial structures (e.g. GaN/Al_2O_3, GaN/AlN//SiC, AlN/Al_2O_3, Al/AlN/Al_2O_3, AlN/GaN/Al_2O_3) at elevated temperatures. The measurements allow to determine temperature-dependent changes of stresses, temperatures of stress free state, annealing-induced relaxation of stresses, temperature regions of yielding and extrapolate intrinsic stresses. The results suggest that the temperature-dependent mismatch of anisotropic thermal expansion coefficients and the deposition temperature predefine 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 nitride 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

RELAXATION OF A STRAINED ELASTIC FILM ON A VISCOUS LAYER.

Rui Huang, Princeton Univ, Dept of Civil and Environmental Engineering, Princeton, NJ; Zhigang Suo, Princeton Univ, Dept of Mechanical and Aerospace Engineering, Princeton, NJ; Haizhou Yin, James C. Sturm, Princeton Univ, Dept of Electrical Engineering, Princeton, NJ.

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 elastic film is modeled 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 plain strain condition. For each fixed wave number less than the critical wave number, there exists a constrained equilibrium state, at which the buckling amplitude reaches an equilibrium value and the stress is partially relaxed. Numerical simulations with a single buckling wave number confirm that the buckling amplitude grows if the wave number is less than the critical wave number and decays otherwise. The numerical simulation goes beyond the range of linear stability analysis and reaches the constrained equilibrium state eventually. While it is relatively quick for a film to reach the constrained equilibrium state, it takes a much longer time to further relax the stress by decreasing the buckling wave number. Starting with a randomly buckled film, the present numerical simulation shows the growth of mixed-mode buckling.

L3.15

NANOINDENTATION ANALYSIS OF VISCOELASTIC COATINGS ON SILICON SUBSTRATES.

Manuel Luis B. Palacio, 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. Using a previously developed elastic-plastic 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 could be attributed to inherent limitations of the elastic-plastic unloading model, such as sensitivity to creep-in effects and the assumption of 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 junction. Using first-principles simulation methods, we have conducted a systematic study of a broad class of interfaces, involving adhesion of Al to selected oxides, carbides, and nitrides and including the effects of surface polarity, alloying agents, and non-stoichiometry. We have thoroughly analyzed the electronic structure of each system to classify the interfacial bonding mechanism. Most importantly, we identify a simple and intuitive trend linking the work of adhesion with the surface energies of the interfaced slabs.

L3.17

TRANSIENT SPUTTERING OF SILICON STUDIED BY MOLECULAR DYNAMICS SIMULATIONS.

Edwin F.C. Haddeman and Barend J. Thijsse, 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 e.g. for interpreting SIMS measurements of shallow 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 81×81Å^2 and contain 60 ML of Si. The samples are exposed to a continued Ar bombardment until the first 5 to 10 ML of Si are removed by sputtering. Even after reaching this depth a steady state has not yet been reached. To our knowledge these are the first MD simulations performed to study the initial stages of sputtering on such a large scale. The Si-Si interactions are modeled with the Modified Embedded Atom Method. Argon ion energies range from 50 eV to 500 eV and the impact angles are varied from 0° to 80°. The most extensive simulations are done at impact angles of 0° and 45°. Results on the change of structural and surface properties of the substrate under ion bombardment are presented as well as a quantitave analysis of the sputtered particles. The incoming ions cause mixing of the topmost surface layers and an amorphous region develops with a width of 10 to 20 Å. The normal incidence Ar implantation profiles compare well to available data. The mixing of the surface region is investigated and the depth of origin of the sputtered particles is determined. We find a total sputtering yield that is in reasonable agreement with experimental values, and that is higher than most other simulation results. Variations in the yield during sputtering are clearly observed. Where possible we compare our results with other simulations and experimental work.

L3.18

STATIC SOFTENING AND DYNAMIC INSTABILITIES IN Al-BASED FOILS USING THE BULGE TEST.

Miroslav Cieslar, Ayatollah Karimi, 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 temperature were observed between RT and 590°C. The contribution of different annealing processes to the softening was established using transmission electron microscopy. It was found that below 200°C only redistribution of dislocations inside dislocation cells and the improvement of the cell structure occurred. Between 200°C and 380°C the formation of a subgrain 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 macroscopic 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

MICROBEAM X-RAY DIFFRACTION FOR 3D INVESTIGATION OF PLASTIC STRAIN AND DEFORMATION IN THIN-FILMS AND SUBSTRATES.

B.C. Larson, Wenge Yang, G.E. Ice, J.Z. Tischler, and J.D. Budai, Oak Ridge National Laboratory, Oak Ridge, TN; and W.L. Lowe, Howard University, Washington, DC.

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 microbeams, differential-aperture diffracted beam depth profiling, and computer automated analysis of the crystallographic orientation and stress/strain in sub-micron resolution 3D voxels in single-crystals, polycrystals, heterostructures, and deformed materials. The basic features of the technique will be described and the results of applications to measurements of 3D elastic and plastic strain distributions will be discussed. The potential for direct comparison of results of such measurements with detailed computer simulations and modeling of deformation microstructure in thin-films, substrates, and bulk materials will be considered.

Research sponsored by the Department of Energy (DOE) Basic Energy Sciences Division of Materials Sciences and performed at Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S.DOE under contract DE-AC05-00OR22725; measurements were performed on the MHATT-CAT beamline at the Advanced Photon Source (APS); the APS is operated by the DOE.

L3.20

DEFECTS AND NANOWIRE FORMATION ON $TiTe_2$ AND VSe_2 LAYERED CRYSTALS. Stefan Hollensteiner, Christel Dieker, Erdmann Spiecker, Wolfgang Jaeger, University of Kiel, Faculty of Engineering, Center for Microanalysis, Kiel, GERMANY; Rainer Adelung, Lutz Kipp, Michael Skibowski, University of Kiel, Institute of Experimental and Applied Physics, Kiel, GERMANY.

The knowledge of the microscopic structure and of the surface structure of layered transition metal dichalcogenides is important for the detailed understanding of their electronic properties and of the processes governing epitaxial growth on these materials. Cleaved crystals and surfaces of $TiTe_2$ and VSe_2 layered crystals have been investigated before and after ultra-high vacuum (UHV) evaporation with metals, such as Rb and Cu, by a combination of transmission electron microscopy (TEM) of plan-view (PV) and cross-section (CS) specimens and by scanning tunneling microscopy (STM). Investigations of PV specimens by TEM and stereo-tilt experiments reveal the presence of layered dislocation arrangements in near-surface regions. In addition, small planar interstitial-type defects (several nanometers in diameter) are present in the layered crystals as could be depicted by high-resolution TEM of CS specimens. Contrast analyses show that the dislocations are perfect dislocations with Burgers vectors $b = a < 100 >$ and $\{001\}$ glide planes. STM characterization of the surface structure before metal deposition shows the presence of linear grooves connected with zones of localized strain and occasionally crack-like defects near the edges of the crystals on the otherwise flat surfaces. Upon UHV metal deposition at room and elevated temperatures networks of metallic nanowires are formed in a self-organized manner on the crystal surfaces. The results suggest that the linear grooves observed by STM on the surfaces before metal evaporation can be attributed to the effect of strain fields induced by crystal dislocations which are located in regions near cleavage surfaces. Furthermore, mesh dimensions of the nanowire network correspond well with the density of the dislocations observed in plan-view specimens of the cleaved crystals by TEM. Linear grooves induced by the near-surface defects are likely to act as nucleation site for the formation of nanowires upon metal evaporation during the early stages of their growth.

L3.21

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

The stress-strain behavior 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 150°C. Films with a thickness and grain size of about 1 mm were deposited by magnetron sputtering onto polyimide 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 ($\sin^2\psi$-method), respectively. 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 can be determined by monitoring the width 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 more than 100 MPa (starting from 300 to 400 MPa) within 2 hours was observed. This 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. Our current experiments focus on the relaxation behavior 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.

L3.22

AE MONITORED PLASTICITY INITIATION UNDER CONTACT LOADING. Natalia Tymiak, Antanas Daugela, Thomas Wyrobek, Hysitron, Inc., Mineapolis, MN; William Gerberich, Dept of Chemical Engineering and Materials Science, University of Minnesota, MN.

Present study utilizes AE monitoring for the evaluation of yield point phenomena for (100) W surfaces with several nm of native oxide and freshly cleaved (100) MgO. For MgO, previous studies suggest dislocation nucleation onset at the start of the yield excursion. In contrast, more complex 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 an additional information towards understanding of the yield point mechanisms as affected by the presence of surface films. Incorporating AE sensor into an indenter tip eliminates sample size effects and provides a greatly enhanced detection resolution for the loads below 1 mN (Hysitron, Inc.). Synchronization error between load displacement and AE data 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 60 nm, relative 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 as 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 excursion lengths. Advanced signal analysis provided an additional information on separation of plasticity and oxide fracture induced contributions of AE waveforms.

L3.23

SELF-FORMED ALUMINUM NITRIDE MICROTUBES THAT EXHIBIT A LARGE BENDING STRESS. Morito Akiyama, Kazuhisa Shobu, Chao-Nan Xu, Kazuhiro Nonaka, National Institute of Advanced Industrial Science and Technology, Saga, JAPAN.

We have investigated the aluminum nitride microtubes made of highly c-axis oriented aluminum nitride thin films. Aluminum nitride thin films deposited on aluminum foils by rf magnetron sputtering form mictrotubes of themselves at room temperature, when the aluminum foils are dissolved in hydrochloric acid solution. The aluminum nitride microtubes exhibit a large bending stress of 110094 megapascals. The bending stress is more than three times larger than the bending strength of aluminum nitride bulk. The diameter of the mictrotubes is proportional to the film thickness. The bending stress is independent of the film thickness and is fixed.

L3.24

IN SITU INVESTIGATION OF THE STRESS EVOLUTION DURING DEPOSITION OF Ti/Al-MULTILAYER FILMS AS A FUNCTION OF LAYER THICKNESS AND SUBSTRATE TEMPERATURE. Stefan Lackner and Reinhard Abermann, Institute of Physical Chemistry, University of Innsbruck, AUSTRIA.

The growth stress of Ti/Al multilayer films, formed by a variable number of double layers of Ti and Al, was measured in situ under UHV-conditions with an apparatus based on the cantilever beam principle. The thickness of the respective Ti/Al-double layer subunit was chosen in such a way as to achive an overall composition of Ti_25Al_75 in a 150 nm thick film. This requires 24, 12 and 6 subunits with doublelayer thicknesses of 6.25 nm, 12.5 nm and 25 nm respectively. These multilayer films were deposited on 10 nm thick Al_2O_3-substrate films at substrate temperatures of 300°C, 400°C and 500°C. Microstructure and phase formation was investigated by TEM/TED- and XRD-analysis. In summary these experiments show a tensile interface stress during the deposition of the first Ti monolayer on Al and a compressive stress during deposition of Al on Ti. The magnitude of both stress contributions strongly depends on layer-thickness and substrate temperature. During deposition of Ti on Al a second and very pronounced tensile stress feature developes at higher Ti-thickness, which is interpreted to indicate alloy formation and grain growth (see also accompanying paper). The influence of the layer thickness and substrate temperature on the respective stress contributions will be discussed. The average film stress built up in the respective multilayer film by superposition of the contributions mentioned above, is also strongly dependent on layer thickness and substrate temperature.

L3.25

NEAR-EDGE X-RAY ABSORPTION FINE STRUCTURE OF HARD CARBON FILM FORMED BY GAS CLUSTER ION BEAM ASSISTED DEPOSITION. Kazuhiro Kanda, Yutaka Shimizugawa, Yuichi Haruyama, Isao Yamada, Shinji Matsui, Himeji Institute for Technology, Laboratory of Advanced Science and Technology for Industry, Hyogo, JAPAN; Teruyuki Kitagawa, Mititaka Terasawa, Harushige Tsubakino, Himeji Institute for Technology, Faculty of

Engineering, Hyogo, JAPAN; Tatsuo Gejo, Masao Kamata, Institute for Molecular Science, Aichi, JAPAN.

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 the electronic 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. Consequently, 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 was studied by Near-Edge X-ray Absorption Fine Structure (NEXAFS) study. The carbon K-edge data show that NEXAFS is sensitive to the local structure around the absorber carbon atom. The resonances from 1s orbital to pi and sigma 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 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. Naruhisa Nagata, Kazuhiro Kusakawa, Hideaki 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 Highly Oriented Pyrolytic Graphite (HOPG) were also prepared. The sp3-C ratio could be estimated by separating the sp3 and sp2 components in the C1s 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 with those mechanical properties. Furthermore, as the sp3-C ratio rose from 0.25 to 0.75,the wave 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 sp2 configuration and are useful indices correlated to the sp3-C ratio.

L3.27
THIN FILM CHARACTERIZATION USING THE POINT-DEFLECTION METHOD. Ning Tang, Roxann L. Engelstad, 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 stresses of freestanding thin films. Compared with the conventional pressure bulge test, it overcomes the difficulties associated with 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 prestressed 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 have been derived based upon classical mechanics. Results indicate that except 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 internal tension. Thus by measuring the transverse deflection 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 PDM 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 frequency tests on the same membrane. Consequently, the PDM has excellent potential for both research applications, and with 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. Mishra, G. Sherfy, T. Buckner, The Univerisity 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)/Cr(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 oxidization process leads to formation of doubled-layered structure of Fcc Co_3O_4 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 fcc regions in the Co layer. The microstructural hardness measured using a nanoindentor as a function of grain size and annealing temperature shows a linear relationship. The transverse magneto-optical kerr performed using HeNe laser (MOKE) shows negligible difference in the coercivity of the film as a function of annealing temperature although difference in magnitude and sign of MOKE signal is noticed

SESSION L4: MECHANICAL PROPERTIES AND NANOSCALE TESTING I

Chairs: Cengiz Sinan Ozkan and Richard P. Vinci
Tuesday Morning, November 27, 2001
Room 304 (Hynes)

8:30 AM *L4.1
THE STIFFNESS OF THIN FILMS. Frans Spaepen, 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 unaxial 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 anelasticity, ...) will be reviewed and applied to the experimental data.

9:00 AM L4.2
EFFECT OF GRAIN SIZE ON ANELASTIC BEHAVIOR OF PURE ALUMINUM AND COPPER MICRO-WIRES. P.A. El-Deiry, 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 anelastic 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 99.99% pure $10\mu m$ diameter Al and Cu as-received (drawn and slight tempered) and annealed micro-wires. Effective elastic moduli of the Al and Cu micro-wires were measured by microtensile tests at various strain rates. Focused-ion beam scanning electron microscopy (FIBSEM) was used for imaging grain sizes. Furthermore, electron-back scatter pattern (EBSP) techniques were used in determining grain orientations. Results from the as-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 Vliet and S. Suresh, MIT, Cambridge, MA.

We present in-situ observations of the effects of crystal orientation, surface roughness, indenter tip geometry and loading conditions on the deformation of surfaces subjected to nanoindentation using the Bragg-Nye bubble raft model. Particular attention is directed at the evolution of defects during indentation loading, and at the effects of atomic level surface roughness on the location and possibility of defect nucleation. In-situ observations of the defect evolution are then directly correlated with discrete deformation phenomena observed during nanoindentation of a variety of face-centered-cubic (FCC) metals. Various aspects of the bubble raft indentation experiments and of the nanoindentation results for FCC metals are then interpreted in conjunction with multi-scale modeling simulations. Surface roughness conditions for which homogeneous defect nucleation occurs during nanoindentation are identified. The presentation will include a series of real-time video images of defect nucleation within the bubble raft during indentation.

9:30 AM L4.4
QUANTITATIVE *IN SITU* NANOINDENTATION OF METALLIC THIN FILMS. A.M. Minor, Dept of Materials Science, UC Berkeley, and Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA; E.A. Stach, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; and J.W. Morris, Jr., Dept of Materials Science, UC Berkeley, and Materials Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA.

A characteristic unique to nanoindentation among mechanical testing techniques is that it measures the mechanical properties of volumes so small that they can be made defect-free. Therefore, the fundamental processes that initiate deformation can be measured and observed directly through a nanoindentation experiment. Typically, these discrete microstrucutal events are examined *ex situ*, and *ex post facto*, in which important details may be missed or lost.
Recently, we have developed a method for quantitative, *in situ* nanoindentation in an electron microscope. The initial application of this new technique will be presented, in which we have studied the onset of deformation during the nanoindentation of aluminum and other metallic thin films. It will be shown that the initial instability during a nanoindentation of Al films corresponds to the first appearance of dislocations in a previously defect-free grain. Plastic deformation then proceeds through the formation and propagation of prismatic loops punched into the material, and half-loops that emanate from the sample surface. Additional results to be touched upon will be direct observations of grain boundary movement during nanoindentation. These results represent the first real time measurements and observations of the discrete microstructural events that occur during nanoindentation.

9:45 AM L4.5
INFRARED PHOTOELASTIC STUDY OF THIN FILM EDGE INDUCED STRESSES IN SILICON SUBSTRATES. H.J. Peng, S.P. Wong, Chinese Univ of Hong Kong, Dept of Electronic Engineering, Hong Kong, CHINA; Shounan Zhao, South China University of Technology, Dept of Applied Physics, Guangzhou, CHINA.

In silicon integrated circuit technologies, the silicon substrate is commonly partially covered with thin films for various applications. When these films contain discontinuities such as edges, large localized stress will be introduced in the silicon substrate near the film edges due to the elastic and thermal mismatch between the substrate and the films. These localized stresses can degrade the device performance by introducing dislocations and defects. In this work, the infrared photoelastic (IRPE) method was employed to study the stress field in silicon substrates under long silicon oxide stripes and windows structures. The IRPE stress fringe patterns in the silicon substrate under these patterned oxide structures were directly observed. From these stress patterns, information about the stress states including the magnitude and the secondary principal stress directions can be obtained. The variations of the stress states with the geometrical parameters of the structure were studied. For example, the transition length that characterizes the range of the film edge induced stress field was found to be about one third of the substrate thickness. There is a local maximum in the stress magnitude in the centerline under the oxide window positioned at a certain depth depending on the window width. Using an analytic solution we obtained recently for the stress field under a thin film edge in isotropic substrates, simulated IRPE stress fringe patterns in these structures were also obtained. The validity of this analytic solution was demonstrated by the good agreement between the simulated and experimental IRPE stress fringe patterns. This work is partially supported by the Research Grants Council of Hong Kong SAR (Ref. No.: CUHK 4155/97E).

10:30 AM *L4.6
IN-SITU ACOUSTIC EMISSION MONITORING OF NANO-INDENTATION. Antanas Daugela, Hiroshi Kutomi, Natalia Tymiak, Thomas J. Wyrobek, Hysitron Inc, Minneapolis, MN.

A newly developed in-situ Acoustic Emission (AE) monitoring technique is a synergy of high bandwidth nanoscale 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 nanoindentation 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 contact wave propagation phenomena is observed starting from a few hundred kHz to several MHz. In this study we discuss progress of the newly developed simultaneous Acoustic Emission (AE) monitoring of quasi-static nanoindentation technique. 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 analyses are used for AE signal decomposition and adaptive filtering. The proposed simultaneous AE monitoring technique targets brittle films and substrates. Experimental results of AE based characterization of W(100), MgO and thin SiC films are presented, where interface cracking, delamination and fracture modes are differentiated by means of the proposed advanced statistical and digital signal processing analyses

11:00 AM L4.7
NANO-CREEP TEST FOR ANATASE POLYCRYSTALLINE FILMS. Hironori Sugata, Shigeo Ohshio, Hidetoshi Saitoh, Nagaoka Univ. Tech., Nagaoka, Niigata, JAPAN.

Nano-indentation 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 Vercobich indenter tip as a function of very light applied force. Resulting force-displacement curve indicates elastic and inelastic deformation behavior of the porycrystalline films. In addition, nano-size cluster flow in the amorphous carbon and carbon nitride films is also detected by this technique. In this study, we formed anatase 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.

11:15 AM L4.8
ACCURATE DETERMINATION OF THE ELASTIC PROPERTIES OF NEAR SURFACE REGIONS AND THIN FILMS USING NANOINDENTATION AND ACOUSTIC MICROSCOPY. Matthew Bamber, Adrian Mann, Brian Derby, Manchester Materials Science Centre, UMIST and the University of Manchester, Manchester, UNITED KINGDOM.

Nanoindentation has been successfully used as a mechanical properties microprobe to characterise the elastic properties of materials. However, in an isotropic material it is not possible to measure the two independent elastic constants by nanoindentation. Normally, a value of Young's modulus is determined using an assumed value for Poisson's ratio. It is also possible to use an acoustic microscope in its z-contrast 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 on the same sample 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 on well-characterised bulk materials (e.g. silica glass and stainless steel) to demonstrate that it produces consistent measurements. It has also been used to characterise thin films of TiN and TiN/AlN multilayers. These results show that, although for thin films there is need to improve the analysis of the mechanics, the combination of nanoindentation and acoustic microscopy shows promise.

11:30 AM L4.9
MEASURING THE MECHANICAL PROPERTIES OF FREE-STANDING ALUMINUM AND COPPER FILMS USING THE BULGE TEST TECHNIQUE. Yong Xiang, Xi Chen, Joost J. Vlassak, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

With the wide application of microelectronic and MEMS devices, accurate measurement of the mechanical properties of thin metal films is becoming more and more important. In this study, the stress-strain curves of freestanding Cu and Al films were determined by measuring the deflection of Si-framed, pressurized membranes. This deflection can be converted into stress-strain data by means of simple analytical formulae the validity of which is shown through finite element 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 membranes out 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 than for bulk materials, a phenomenon that has been observed in microtensile tests as well, and that can be attributed to anelastic stress relaxation in the films.

SESSION L5: MECHANICAL PROPERTIES AND NANOSCALE TESTING II

Chairs: L. B. Freund and Robert F. Cook
Tuesday Afternoon, November 27, 2001
Room 304 (Hynes)

1:30 PM *L5.1
MICROSTRUCTURAL INVESTIGATIONS OF FATIGUE EFFECTS IN THIN Cu FILMS. R. Schwaiger 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 for cyclically deformed bulk metals dislocation structures, such as veins, slip bands, and cell structures evolve, which typically extend over severals micrometers. For (sub)micron dimensions the fatigue behavior is likely to be different than in bulk material as 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 polyimide substrates were fatigued at a total strain amplitude of 0.5%. Focused ion beam 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 fatigued thin film specimens is the formation of voids at the film-substrate interface beneath the extrusions, which is interpreted as a 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 L5.2
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 being 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 due to the thermal-mechanical 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 substarte. 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 tesnile 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 carried out in this study. Both macrotexture and microtexture 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 are calibrated via the simulation of the microtensile tests of free-standing samples. The nanoindentation tests of the gold films on a silicon substarte are finally analyzed to asses the effects of both the film texture and interal stresses on the nanohardness numbers.

2:15 PM L5.3
EFFECT OF MICROSTRUCTURE ON MECHANICAL PROPERTIES OF AlN THIN FILMS. Shuichi Miyabe, Masami Aono, Nobuaki Kitazawa, Yoshihisa Watanabe, National Defense Academy, Dept. of MS&E, Yokosuka, Kanagawa, 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 and the latter yields a granular one. The effect of microstructure on mechanical properties has been studied by using a nanoindentation system equipped with a diamond Berkovich indenter. The maximum force applied to the films was limited to 3 mN. For the columnar structure film of 300 nm in thickness, the ratio of the maximum penetration depth to the film thickness R is about 26% 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 11% and the microhardness is found to be about 14 GPa. These results reveal that the microhardness of the AlN film itself can be evaluate 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 L5.4
DYNAMIC OBSERVATION OF DISLOCATION FREE PLASTIC DEFORMATION IN FCC METAL THIN FILMS. Yoshitaka Matsukawa, Kazufumi Yasunaga, Masao Komatsu, Michio Kiritani, Academic Frontier Research Center for Ultra-high-speed 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 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 0.01μm/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 motion was confirmed, despite observed under two-beam dark field image condition, in which dislocations should be observed if any. The local 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 10Gpa. 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 those events a characteristic diffraction image contrast evolution was confirmed; equal thickness fringes became faint temporally. 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 L5.5
VOID INTERGRANUAL MOTION UNDER THE ACTION OF ELECTROMIGRATION FORCES IN THIN FILM INTER-CONNECTS WITH BAMBOO STRUCTURE. Ersin Emre Oren, Tarik Omer Ogurtani, Middle East Technical Univ, 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 interphasial 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 intergranual motion, under the actions of capillary and electro-migration forces in thin film metallic interconnects with bamboo structure. Very rich, and also unusual void grain-boundary interaction morphologies such as penetration and detrapping, 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 micro-discrete (straight) interfacial elements as convenient mathematical tools as proposed by Ogurtani. 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 layer and the grain boundary interphase, respectively. Displacement velocities of the triple junction, longitudinal and transverse are also deduced 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 ordinary points at the void surface layer.

3:30 PM *L5.6

MICROMECHANICAL TESTING OF FREE-STANDING THIN FILMS FOR MEMS APPLICATIONS. John C. Bravman, Ping Zhang, Stanford University, Stanford, CA; Hoo-Jeong Lee, Agere, 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 various actuation and stimulation. Understanding the mechanical properties of free-standing thin films is 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 on 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 sample fabrication process to produce free-standing thin films of different materials and different thicknesses. We have built a custom-designed micromechanical testing apparatus with a load resolution of 0.2mN and a displacement resolution of approximately 10nm. We use Transmission Electron Microscopy (TEM) to reveal the micro-structural 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 microtensile tests and stress relaxation tests. From microtensile tests, we examine the results with respect to those of bulk materials and thin films adhered to substrates. From stress relaxation tests, we study the effect of alloying 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. Maznev, M. Gostein, P. Krastev and R. Bolotovsky, Philips Analytical, Natick, MA.

The continuous drive for increased speed and packing density of integrated circuits has resulted in rapid changes in metal interconnect fabrication technology, where copper and low-k dielectrics are replacing traditional Al/SiO_2 structures. Knowledge of mechanical properties of materials comprising an interconnect structure is becoming increasingly important for the new generation of metal interconnects. Low-k dielectrics with typically much lower elastic modulus than SiO_2 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 the present work, a laser optoacoustic technique called Impulsive Stimulated Thermal Scattering (ISTS) implemented in a commercially available instrument was used to characterize elastic properties (i.e. elastic moduli or, alternatively, acoustic velocities) of dielectrics (both traditional such as thermally grown and CVD SiO_2 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 application of the optoacoustic 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, Nicolas Conte, University of Tennessee, Knoxville, TN.

Contact mechanics for indentation testing with spherical indenter 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 studies have focused on metallic materials with the 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 temperature in order to observe the relationship between the indentation behavior and compression 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 additional to the load, displacement, and frequency specific stiffness information, topographic analysis of the residual indentation print 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 100°C.

4:30 PM L5.9

MECHANICS OF INDIUM-TIN-OXIDE FILMS ON POLYMER SUBSTRATES WITH ORGANIC BUFFER LAYER. Sung Kyu Park, Jeong In Han, Chan Jae Lee, Won Keun Kim and Min Gi Kwak, Korea Electronics Technology Institute, Pyungtaek, 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 polyethersulfone (PES), polycarbonate (PC) and polyethylene terephthalate (PET), which have gas barrier layer and anti-glare coating for organic electron devices. The experiments were performed with rf-magnetron sputtering using a special instrument and novel stepped heating process to reduce thermal stress of the substrates. Furthermore, we speculated the mechanism of reduced strains resulted from 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-oxygen in polymer matrix structure. Consequently, based on the investigation of electro-mechanical characteristics and organic buffer layer, more reliable ITO films were deposited on polymer substrates with sheet resistance of 20-25 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; Jin Rong 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 sp^3 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 sp^3 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 counterbody which is a sapphire ball. All the tests are performed at controlled relative humidity and room temperature (about 23°C). It was noted that friction coefficient of ta-C:Al films increases at the beginning of the testing before 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 films are worn through and the friction coefficient suddenly increases to a high value up to 0.6. The original surface roughness of the counterfaces, surface smoothening due to successive wear, and wear debris produced during the testing are all responsible for the tribological behavior of ta-C:Al films. The stress of the ta-C films is determined with bending plate method. The internal stress is related to the structural properties of films. The pure carbon ta-C films generally contain a relatively high residual compressive stress, which is related to its sp^3 content, amorphous structure and preparation conditions. 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.

SESSION L6: POSTER SESSION
Chairs: L. B. Freund and Huajian Gao
Tuesday Evening, November 27, 2001
8:00 PM
Exhibition Hall D (Hynes)

L6.1
DETERMINING THIN FILM PLASTIC PROPERTIES BY THE BULGE TEST. Z.L. Zhang, SINTEF Materials Technology, Trondheim, NORWAY; J. Vlassak, 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 and fracture 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-train 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 plane strain deformation to biaxial 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 substrate compliance 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.

L6.2
STRESS EVOLUTION AND PHASE FORMATION DURING DEPOSITION AND DURING ANNEALING OF $Ti_{25}Al_{75}$-ALLOY FILMS. Stefan Lackner and Reinhard Abermann, Institute of Physical Chemistry, University of Innsbruck, AUSTRIA.

In continuation of earlier work, the growth stress of $Ti_{25}Al_{75}$-alloy films was investigated under UHV-conditions using a cantilever beam technique. The alloy films were deposited on 10 nm thick Al_2O_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/TED and XRD-experiments. Alloy films with Al-contents below 50% show stable stress versus thickness curves comparable to one component systems. Films with high Al-content, however, exhibit an irregular 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 first performed annealing experiments of 150 nm thick $Ti_{25}Al_{75}$-alloy films, previously deposited as amorphous films at RT, and measured the irreversible change of the internal stress during heating. By correlating characteristic changes in the stress versus temperature curve with the results of parallel TEM and TED-analysis, a transition of amorphous $Ti_{25}Al_{75}$ to the cubic $L1_2$-$TiAl_3$-alloy phase is deduced. The transition of the cubic $L1_2$-$TiAl_3$-alloy phase to the tetragonal $D0_{23}$-$TiAl_3$-alloy phase, reported in the literature, does not give rise to a significant stress change but has been verified in TEM- and TED-experiments. To further 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 500°C. This multilayer experiment shows large tensile stress contributions during the Ti on Al deposition and compressive stress contributions of comparable magnitude during the Al on Ti deposition. Thus metal interdiffusion and/or delayed alloy phase formation is assumed to be the reason for these growth irregularities. The average stress during deposition of 24 Ti/Al-doublelayers 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.

L6.3
ON VISCOUS CREEP IN METALLIC WIRES AT ELEVATED TEMPERATURES AND LOW STRESSES. Jaroslav Fiala, Brno University of Technology, Faculty of Chemistry, Brno, CZECH REPUBLIC; Luboš Kloc, Václav Sklenička, Institute of Physics of Materials CAS, Brno, CZECH REPUBLIC.

Viscous 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 helicoid 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 infuence of the surface layer loaded by maximum stress. The experimental results were interpreted as Coble diffusional creep and/or Harper-Dorn dislocation creep. Some data are in a very good agreement with Coble theory especially those obtained on some fine grained materials. This dependence is replaced by large data scatering 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 Harper-Dorn 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 theory in some materials, transition stage duration independent of stress and the total transient 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 behaviour of structural materials at conditions of engineering practice.

L6.4
A STUDY ON THE BEHAVIOR OF RESIDUAL STRESS WITH WATER ABSORPTION OF Si 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 MS&E, Daejon, KOREA; Youngsoo Park, Ilsub Chung, Samsung Advanced Institue of Technology, Materials and Device Laboratory, Suwon, KOREA.

Fluorinated silicon oxide (SiOF) films were deposited by electron cyclotron resonance plasma enhanced chemical vapor deposition (ECRPECVD). 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 after the water absorption. However, the change of chemical bonding structure was not observed in this study. After the exposure to room air, the films were kept in dry air. The residual stress returns to 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_2O molecules to Si-F bonds on the surface.

L6.5
A NEW FORCE-DISPLACEMENT MODEL FOR CONTINUOUS INDENTATION OF BILAYER MATERIALS. Ali Nayebi, Rochdi El Abdi, Gérard Mauvoisin, Olivier Bartier, Rennes 1 University, LARMAUR, 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 diameter sphere of 1.5875 mm. Various surface-substrate yield stress ration β ($1< \beta <6$) and strain hardening exponent n ($0.0 < n < 0.5$) of studied materials have been considered in modeling. The thin film thicknesses vary from 50 to 400 μm.

Based on finite element results, a mixture law is propsed for bilayer material indentation. This law takes into account the influence of film and substrate and leads to determine the bilayer indentation depth from film and substrate homogenous materials indentation depths. This influence is modelled by a parameter which depends on the layer and substrate mechanical properties and on the applied force. Finite element results show that this parameter depends on dissipated plastic engery induced by indentation in the film and in the substrate of the bilayer.

Using normalized plastic strain and calculated dissipated plastic energy in a thin film and in the substrate, a new applied force-indentation depth of bilayers is obtained. Comparison this new relation with experimental results, shows the capabilities of the model for simulating the behavior of bilayer materials under indentation. The results form this new method are compared with those of experiment to show the capability of the model.

L6.6
DEPOSITION OF TiN THIN FILMS ON Si(100) BY UNBALANCE MAGNETRON SPUTTERING. Wen-Jun Chou, Ge-Ping Yu, and Jia-Hong Huang, Department of Engineering and System Science, National Tsing Hua University, HsinChu, TAIWAN.

Titanium nitride (TiN) films were deposited on Si(100) substrate using an unbalance magnetron sputtering (UBM) technique. 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 thin film structure was characterized by X-ray diffraction (XRD), cross-sectional transmission electron microscopy (XTEM), and field-emission-gun scanning electron microscopy (FEG-SEM). N/Ti ratios of the thin films were determined using both X-ray photoelectron spectroscopy (XPS) and Rutherford backscattering spectrometry (RBS). The resistivity of the TiN films 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 of TiN films 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. Fong, Harvard Univ, Division of Engineering and Applied Sciences, Cambridge, MA; Denis 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 -2.17 ± 0.15 N/m, 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 cycling spans a temperature range from -196 to 600C. It is seen that the strong relaxation at high temperatures normally found in unpassivated films is nonexistent for passivated films. The copper film did not show any rate-dependent effect over a range of heating/cooling rate from 5 to 25C/min. Further analyses showed that significant strain hardening exists during the course of thermal loading. In particular, the measured stress-temperature response can only be fitted with a kinematically 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 400C 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 succesive 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, most likely due to “zipping up” of surfaces into grain boundaries upon coalescence of the islands (Nix-Hoffman); 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 ultra-high vacuum. Although the window for variation of the deposition rate is narrow, 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. Macit Ozenbas, Husniye Guler, Dept. of Metallurgical and Materials Engr., 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-(5at%)Fe/Si, Al-(5at%)Cr/Si, Al-(5at%)Ni/Si, Cu/Si and Cu-(5at%)Nb/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. Silicates; $Fe_2Al_4Si_5O_18$ and $Fe_3Al_2(SiO_4)_3$ were detected in Al-(5at%)Fe/Si system. In spite of the presence of two intermetallic phases, $[Ni(Al_{0.5}Si_{0.5})]8\underline{C}$ and $[(AlSi)_7Ni_3]\underline{C}$, in Al-(5at%)Ni/Si system, only one intermetallic phase $((Cr_4Si_4Al_13)84\underline{F})$ was formed in Al-(5at%)Cr/Si system. In Cu/Si system; silicides; $Cu_{0.83}Si_{0.17}$, Cu_4Si, [Cu, Si]-ϵ and [Cu, Si]-$\eta\prime$ were observed. Finally in Cu-(5at%)Nb/Si system; $Cu_{0.83}Si_{0.17}$, Cu_4Si, $Nb_5Cu_5Si_4$ and [Cu, Si]-$\eta\prime$ silicides were formed sequentially. In most cases; $t^{1/2}$ (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 $Cu_{0.83}Si_{0.17}$ was investigated with and without Nb impurity to understand the impurity effect. 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, Yi-Wen Cheng and J. David McColskey, 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 MOSIS service. We tested specimens of aluminum alloy with 0.5 mass percent copper on 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 M2 and M1/M2 patterned in the shape of tensile specimens deposited directly on silicon substrate. Silicon beneath and near the tensile specimens 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 gage sections were 10 μm wide and 180 μm long. On one chip, the average values for 12 specimens of M2 for yield and ultimate strengths were 67 and 78 MPa, with an elongation of 1.4%. The five M1/M2 specimens had average yield and ultimate strengths 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 unloading-reloading 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.

L6.14
RESIDUAL STRESS MEASUREMENT IN A Pt-ALUMINIDE BOND COAT.
Makoto Watanabe, Daniel R. Mumm, Stefanie R. Chiras, and Anthony G. Evans, Princeton Univ, Princeton Materials Institute.

The residual stress induced in a Pt-aluminide bond coat formed 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 has been applied. This method required that the substrate thickness be 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, 140MPa, consistent with its thermal expansion coefficient, relative to that for the substrate.

L6.15
MECHANISTIC UNDERSTANDING OF THE STRESS-INDUCED FAILURE OF Si_3N_4 METAL-INSULATOR-METAL CAPACITORS.
Dongwoo Suh, Bongki Mheen, Kyu-Hwan Shim and Jin-Yeong Kang, Compound Semiconductor Research Department, Microelectronics Technology Laboratory, Electronics and Telecommunications Research Institute, Taejon, KOREA.

We investigated the failure of Si_3N_4 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 Si_3N_4 layers with 30(Specimen-A), 50(Specimen-B), and 70 nm-thick (Specimen-C) were deposited by plasma enhanced chemical vapor deposition (PECVD) at 400^oC, corresponding to the area-normalized capacitance of 2.2, 1.3, and 0.9 fF/mm^2, respectively. The metal electrodes of aluminum was deposited by reactive sputtering at 400^oC with TiN diffusion barriers, finally having a multi-stacking structure of Al/TiN/Si_3N_4 /Ti/TiN/Al in bottom-up direction on thermally grown SiO_2 over p-type silicon wafers. It was found that microvoids had occurred at the Si_3N_4 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 thermally-induced residual stress embedded into the constituent 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 reversed from compressive stress of 930 MPa to tensile one of 390 MPa across the boundary of the Si3N4 and the 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 Si_3N_4 layer provided the stress goes beyond the threshold value. It was noted that the residual stress of the dielectric and adjacent metal 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 INTERMETAL DIELECTRICS FOR ULSI APPLICATION BY A FINITE ELEMENT METHOD.
Nobuo Aoi, Takuya Fukuda, Hiroshi Yanazawa, and Hironori Matsunaga, Environmental Process Technology Laboratory, Semiconductor Technology Research Department, Association of Super-Advanced Electronics Technologies, Yokohama, JAPAN.

With shrinking of device size of ULSI, degradation of device performances are caused by an increase of RC delay due to intra-line capacitance. In order to reduce capacitance between metal lines of ULSI multilevel interconnects, it is indispensable to introduce low-k dielectrics. A possible candidate for the future intermetal dielectrics is 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 strengths cause serious issues in integrating low-k dielectrics into ULSI 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 inorganic low-k films by means of a nano-indentation. Density and thickness of films were obtained by means of GIXR. 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 2-dimensional random pore generation model. FEM Results of elastic modulus, which were calculated for 2-dimensional random pore model, extremely well fit with the experimental data obtained by a nano-indentation method. From our model, average pore aggregation ratio (Apore), defined by area ratios of generated pore to unit pore, can be obtained. Logarithmic values of Apore linearly depend on logarithmic values of relative density of low-k dielectrics. And elastic modulus of low-k dielectrics exhibits strong dependency 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 Apore. In order to develop some robust low k dielectrics by a pore inclusion, it is clarified quantitatively by our FEM analyses 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 stress states 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-PbSnF$_4$ THIN LAYERS AND ITS EVOLUTION THROUGH THE $\alpha \rightarrow$ o-PbSnF$_4$ PHASE TRANSITION.
Georges Dénès and M. Cecilia Madamba, Concordia Univ, Dept of Chemistry and Biochemistry, Laboratory of Solid State Chemistry and Mössbauer Spectroscopy, Laboratories for Inorganic Materials, Montreal, Québec, CANADA.

PbSnF$_4$ is the highest performance fluoride-ion conductor. The exceptionally high mobility of the fluoride ions in α-PbSnF$_4$ has been ascribed to the disturbance brought about by the replacement of half the Pb^{2+} ions in the fluorite-type structure of β-PbF$_2$ by covalently bonded Sn(II), and to the presence of a vacant fluoride-ion layer between two adjacent tin layers. However, tin(II) lone pairs, that are strongly stereoactive, occupy the vacant anionic plane and keeps 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. PbSnF$_4$ can be prepared by several methods, and it undergoes a large number of order/disorder phase transitions, making it one of the most complex material known. When prepared by the addition of lead(II) nitrate to a solution of SnF$_2$, α-PbSnF$_4$ precipitates, and when recrystallized, the size and quality of the membrane-like crystallites is improved. In addition, the thin sheets are the subject of an intense intrinsic stress generated by the ferroelastic properties of the material. α-PbSnF$_4$ 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.

L6.19
STRAIN RELAXATION IN AMORPHOUS OXIDE FILMS WITH INTERCALATED IONS.
Igor A. Konovalov, David Kinosky, Sadeg M. Faris, Reveo, Inc., Elmsford, NY.

Metal oxides are known to absorb reversibly a large quantity of positive ions (protons, Li ions, etc.) and this property is widely used in applications such as rechargeable Li-ion batteries, electrochromic 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/delamination.

L6.20
AN ANALYTICAL MODEL FOR INTRINSIC STRESS EFFECT ON OUT-OF-PLANE DEFLECTION OF A CHEMICAL VAPOR DEPOSITED THICK FILMS.
Jeung-hyun Jeong, Dongil Kwon, School of Materials Science and Engineering, Seoul National University, Seoul, KOREA; Young-Joon Baik, 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 IR 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 deflection in film when the film is released from substrate. In this study, a theoretical model showing how residual stress influences the out-of-plane deflection in a free-standing thick diamond films, which is referred to as bowing phenomena, is presented. The variation in residual stress with film thickness 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 layer-by-layer deposition of the film. The model was developed using infinitesimal plate bending theory, considering the two deformation modes of contraction or expansion and bending. To verify the suggested model, diamond films were fabricated on Si, Mo and W 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 as-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 discussed through depositing different layers with different intrinsic stresses.

L6.21
MAGNETORESISTIVE, MECHANICALLY HARD SUPERLATTICES OF THE CrN, NbN, TiN MULTILAYERS DEPOSITED ON MONOCRYSTALLINE Si WAFERS.
Andrzej Wolkenberg, Technical Univ, Dept of Metallurgy and Materials Engineering, Czestochowa, POLAND and Institute of Electron Technology, Warsaw, POLAND; Hanna Wrzesinska, Institute of Electron Technology, Warsaw, POLAND; Andrzej Bochenek, Adam Tokarz, Zygmunt Nitkiewicz, Technical Univ, Dept of Metallurgy and Materials Engineering, Czestochowa, POLAND; Tomasz Przeslawski, Institute of Electron Technology, Warsaw, POLAND; Piotr Dluzewski, Institute of Physics P.Ac.Sci., Warsaw, POLAND.

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 5g probe. There are investigated the nitride layers (CrN, TiN, NbN), superlattices composed of these nitrides and thin metal films deposited either on the monocrystalline Si or metallic substrates as steel or copper. We propose here a model for superlattice hardening analogous to supermodulus effect explained on Fermi surface - Brillouin zone interaction theory similar to RKKY procedure for the galvanomagnetic properties. We present also the measurements of magnetoresistivity (MR) effect corresponding to the superlattices, which consist of the multilayers TiN/NbN deposited on Si substrates. There are investigated the layers of different thickness of TiN and NbN obtained by the reactive sputtering method with different superlattice period values. The composite hardness of the structures (superlattice on Si) reaches 80GPa and MR value in the best probes was ∼80% (beginning from some Gs 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 030 16

L6.22
STRESS DEVELOPMENT IN DRYING POLYMER COATINGS AFTER SOLIDIFICATION. H. Lei, L.F. Francis, W.W. Gerberich, and L.E. Scriven, Eastman Kodak, Rochester, NY.

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 inhibited 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 yielding and post-yielding viscous deformation was developed to predict stress evolution in drying of polymer coatings after solidification. The coupled equations of diffusion and stress development are solved 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 Esser, Naval Research Laboratory, Washington, DC; Aris Christou, 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, SiO_2, and diamond. It is corrosion resistant. Niobium has the third lowest resistance of the refractory metals. Finally, the melting point of TM = 2467°C suggests that niobium will have high temperature stability and low interdiffusion coefficients. The kinetics of Nb/Au thin films was examined using sheet resistance measurements, X-Ray diffraction and Auger Electron Spectroscopy depth profiling techniques. The data is analyzed using the Matano method and a technique proposed by Zhang and Wong to take into account the formation of an intermediate intermetallic phase. The relevant diffusion coefficients for two kinetics regimes are presented. Finally, niobium/gold was used as a Schottky contact on GaAs and subjected to an anneal study. It was found that the series resistance of Schottky diodes decreased with a 1 hour anneal at 250°C to 12.5W, and remained stable for temperatures of 250°C for 672 hours. It was found that the series resistance degraded significantly for anneal conditions that were likely to cause intermetallic formation, increasing to 100W. The result of this work is a set of engineering guidelines for the application of niobium/gold metallizations to gallium arsenide circuits and devices.

L6.24
EFFECTS OF VARIOUS PASSIVATION DIELECTRICS AND VIA RESERVOIR LENGTHS ON STRESS AND ELECTROMIGRATION RELIABILITY OF METAL INTERCONNECTS. Young Bae Park, Young Ah Cho, Hyuk Hyun Ryu, Won Gyu Lee, System IC R&D 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 tri-axial tensile stress state that is favorable for void formation which is a major concern for the reliability of multi-level 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 dielectrics and metal line widths. And 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 were PETEOS (Plasma Enhanced Tetra Ethyl Ortho Silicate), FOX (Flowable 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, 10um) and via reservoir lengths (0.04, 0.08, 0.12, 0.30um) were used for stress measurement and EM test of metal interconnect, respectively. We tried to experimentally reveal the relationship among EM lifetime, stress of metal line, and also stress of passivation layer. 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 passivation dielectric and 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, Qianghua Xie, Ginger Edwards, Joe Kulik, 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 silica 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 pores. Transmission electron microscopy (TEM) and spectroscopic ellipsometry are used to determine material density, pore size, and orientation. Shifts in the electron energy loss spectra may be correlated with the porosity measured by spectroscopic ellipsometry to yield information on the nano-porosity of these films 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 compromises in the silica network due to organic (methyl, ethyl) inclusions.

L6.26
Abstract Withdrawn.

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

Soft magnetic materials such as Fe-Cr(Al, Mo) yield to magneto-mechanical 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, magnetostriction constant, relative permeability) 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 nanoindenter 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 beams. Mechanical properties such as elastic modulus and hardness, as well as creep rate, have been assessed by nano-indentation. 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 magnetostriction constant and residual stresses. Creep rate 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 domains structure 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. Zhigang Xu, Qiuming Wei, Sergey 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 thin film is important for the fuel cell applications. The YSZ thin films have been synthesized with atmospheric combustion chemical vapor deposition (ACCVD) technique with liquid fuel. The as-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 nano-indentation. The effect of processing parameters, such as substrate temperature, ratio of fuel to oxygen and concentration 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
Chairs: Huajian Gao and Shefford P. Baker
Wednesday Morning, November 28, 2001
Room 304 (Hynes)

8:30 AM *L7.1
RATCHETING-INDUCED CRACKS IN THIN FILM STRUCTURES. Z. Suo, M. Huang, Mechanical and Aerospace Engineering Department and Materials Institute, Princeton 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 test to qualify integrated materials structures 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 temperatures. This paper presents a systematic study 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 ratchets, namely, accumulates in the same direction as the temperature cycles. The ratcheting deformation may build up stress in the brittle materials, 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 avert this failure mode. Concepts presented here are generic to related phenomena in thin film structures. PDF file of the preprint can be downloaded from www.princeton.edu/ ˜suo, Publication 118.

9:00 AM L7.2
THIN FILM FRACTURE DURING NANOINDENTATION OF HARD FILM - SOFT SUBSTRATE SYSTEMS. M. Pang, K.D. Weaver, and D.F. Bahr, 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. The key point of the model is to superpose the solutions of both large deflection of the oxide film and plastic deformation of the substrate. In current study, the model is further refined by testing a variety of materials with different film formation conditions. The tested materials include titanium/titanium oxide, aluminum/aluminum oxide, copper/copper oxide, and tungsten films on aluminum and copper. Film formation conditions are thermal oxidation, anodic polarization, and vacuum deposition. Film fracture occurs at applied surface tensile stresses ranging from 400 MPa to 5 GPa and above. The results show that the model can fit well over a wide range of materials. Additionally, the applicability of this 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. Litteken 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 barrier 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 elastic layers, and by producing patterned polymer lines of varying aspect ratio. The significant extent 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 similar length scales. Trends in adhesion 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 MECHANISMS IN A CLASS OF THERMAL BARRIER SYSTEMS. Anette M. Karlsson, 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 associated with many of the failure types observed in thermal barrier systems. In this study, we investigate NiCoCrAlY bond coats with

EB-PVD ZrO2. The failure mechanisms in this class of thermal barrier systems are intrinsically linked to the morphological features of the TGO/BC interface that develops during thermal cycling. When 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 size is yielding and reverses yielding of the bond coat, as the temperature cycles. A numerical scheme, utilizing the finite element method, is developed to investigate parameters governing the geometric instabilities. Of particular interests in this study is how the initial shape of the imperfection influences the non-elastic response of the bond coat and the TGO. Results are presented to illustrate how the development of stresses and plastic strain during thermal cycling results in a shape change in the layered structure.

9:45 AM L7.5

THE EFFECTS OF COPPER ON THE INTERFACIAL FRACTURE OF GOLD FILMS. N.R. Moody, N. Yang, Sandia National Laboratories, Livermore, CA; D.P. Adams, Sandia National Laboratories, Albuquerque, NM; M.J. Cordill, D.F. Bahr, Washington State University, Pullman, WA.

Adhesion is a critical factor in the performance of hybrid microcircuits where the diffusion of copper from leads during processing and service can alter the adhesive strength and therefore interfacial fracture susceptibility of gold conducting films. However, data are limited. We have therefore begun to study the interfacial fracture susceptibility of gold, gold-on-copper, and gold-2w/o-copper films. Nanoindentation showed that the elastic moduli and hardness values of the gold and gold-on-copper films were similar but less than corresponding values for the gold-copper alloy film. A tungsten overlayer was then sputter deposited onto the three film systems to apply a uniform compressive stress. When combined with nanoindentation, these stresses triggered 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-copper alloy and gold-on-copper multilayer films adhere better than single layer gold films. In this presentation, these results will 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. Jacob Israelachvili, Chad Park, Norma Alcantar, Jian-Mei Pan, Department of Chemical Engineering and Materials Department, University of California, Santa Barbara, CA.

Much is known about the fundamental mechanisms of material failure, such as fracture and crack propagation, but much less about the reverse effect of how bulk materials form or consolidate 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 interaction forces between thin films as well as to image their interface. We have studied how bulk gold and platinum films form at the nano-scale when two initially rough surfaces (composed of 5-10 nm asperities) 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 apparently general effect, which suggest that it may also occur in other systems and situations.

11:00 AM L7.7

ADHESION OF COPPER TO SELF-ASSEMBLED MONOLAYERS. Guangchun Cui[a], Michael W. Lane[b], and G. Ramanath[a]. [a]Rensselaer Polytechnic Institute, Dept of Materials Science & Engineering, Troy, NY. [b]IBM T.J. Watson Research Center, Yorktown Heights, NY.

As the critical feature size 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 & 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 evolving micro and nano device systems. We have recently demonstrated the attractive barrier properties of SAMs with an aromatic terminal functional group and a silane group.[1] In order for these SAMs to be used in applications, their role in improving interfacial adhesion to SiO_2 or SiO_2-based low-k dielectrics must be investigated. In this work we quantitatively measure interfacial adhesion in $Cu/SAM/SiO_2$ 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 (XPS). The adhesion results are correlated with the chemical structure of the SAMs, and discussed in the context of their diffusion barrier properties. [1] A. Krishnamoorthy, K. Chanda, S.P. Murarka, J.G. Ryan, and G. Ramanath, "Self-assembled near-zero-thickness molecular layers as diffusion barriers for Cu metallization", *Applied Physics Letters* 78, 2467 (2001).

11:15 AM L7.8

NANOINDENTATION INDUCED FRACTURE IN HARD MULTILAYERS THIN FILMS. D. Bethmont, Y. Wang, A. Karimi, Department of Physics, Swiss Federal Institute of Technology (EPFL), Lausanne, SWITZERLAND.

Mechanical properties and fracture behavior of multicomponent titanium aluminum carbonitride thin films were investigated using nanoindentation measurement methods. The films were prepared as a single layer, multilayers, and nanocomposite materials 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 through thickness or interfacial cracks 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 ploughing mechanism leading to ductile character of scratch grooves. When the applied load reaches a critical value periodic microcracks appear regularly on both sides of the contact site due to tensile stress behind the indenter. Further displacement of indenter into the film develops very high 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 particulates randomly distributed within the films because of arc deposition technique. These structural inhomogeneities yield microcracking much earlier than the overal film, modify median scratch track, and develop a complex stress field ahead of the indenter which causes the propagation of lateral irregular cracks and thereby spallation of the film fragments 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 carbonitrides and attempts to highlight the relationships 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. Barbosa, III, R.P. Vinci, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA; R.S. Ridley, Sr., C.H. Strate, R.S. Dwyer, T. Grebs, Fairchild Semiconductor, Mountaintop, PA.

The desire for improved process yield has motivated the power semiconductor industry to better understand the effects of process variations on reliability. Wafers are often thinned to reduce the through-thickness resistance of their end product devices. Processes involved in 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 stress state was varied through sputtering temperature and pressure, and was measured 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 BLISTER DELAMINATION TESTS. Z.L. Zhang, SINTEF Materials Technology, Trondheim, NORWAY; J. Vlassak, Division of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

The blister test has certain advantages for measuring thin film adhesion toughness. Equations for energy release rate in the blister test are available in the literature. The energy release rate was formulated based on either the global energy consideration or a nonlinear plate 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 pressuring the membrane was neglected. Using finite element 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 axisymmetric and plane strain 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 equations from the literature for the blister test can be applied. This observation is particularly important for practical applications where in many cases the extent of delamination is considerably less. Furthermore, a large hole opening angle and a compliant substrate will 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 PM ***L8.1**

MECHANICAL STABILITY AND CHARACTERIZATION OF DIELECTRIC THIN FILMS. Robert F. Cook, Jeremy Thurn, Dylan J. Morris, Yvete A. Toivola and Michael P. Hughey, 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: 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 permeabilitiy characteristics. The mechanical stability of dielectric thin films is then of crucial importance for advanced device designespecially 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 and four-point bend for adhesion. Particular attention will be given to new methods of probe calibration for nano-scale hardness, modulus and toughness estimation and to implementation of a double-substrate method for thermal expansion coefficient determination. The behavior of a wide variety of oxide, nitride, oxynitride, 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 are just as interesting and changeable as those of the more-traditionally studied metallic films.

2:00 PM L8.2

NANOSCALE MECHANICAL PROPERTIES OF NANO-STRUCTURES. S.A. Syed Asif, Hysitron Inc., Minneapolis, MN; K.J. Wahl and R.J. Colton, Naval Research Laboratory, Washington, DC.

With the development of advanced fabrication techniques, it is becoming possible to produce structures and Micro Electro-Mechanical Systems (MEMS) devices with lengths approaching the nanometer scale (e.g. Nano Electro-Mechanical Systems, NEMS). The performance of such devices is explicitly 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 indentations accurately. Furthermore, as the length scale of the indentation approaches the scale of the nanostructure, the mechanical response can differ 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, coupling scanned probe imaging and depth-sensing indentation capabilities, was used to examine patterned quartz nanostructures (ridges) as well as 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 PM L8.3

A NOVEL EXPERIMENTAL TECHNIQUE FOR MECHANICALLY TESTING THIN FILMS AND MEMS MATERIALS. Horacio Espinosa, Bart Prorok, Rong Wang, Max Fischer, 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. The experiment consists of loading a fixed-fixed membrane with a line load that is applied to the middle of the span with a nano-indenter column. A Mirau microscope-interferometer is conveniently aligned with the nano-indenter to directly measure strains. This is accomplished through a specially 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 then 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 SiO2 layers.

2:30 PM L8.4

FRACTURE BEHAVIOR OF MICRO-SIZED SPECIMENS PREPARED FROM AN AMORPHOUS ALLOY THIN FILM AT AMBIENT AND ELEVATED TEMPERATURES. Kazuki Takashima, Ryuichi Tarumi, Yakichi Higo, Tokyo Institute of Technology, P&I Lab., Yokohama, JAPAN.

MEMS devices are usually fabricated from a thin film deposited on a substrate by a suitable surface micromachining 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 micro-sized cantilever beam type specimens prepared from an electroless deposited Ni-P amorphous alloy thin film at ambient and elevated temperatures. Cantilever beam type specimens with dimensions of 10 x 10 x 50 μm^3 were prepared from 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 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-sized 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 PM L8.5

STRESS RELAXATION MECHANISMS IN TETRAHEDRAL-AMORPHOUS CARBON FOR MEMS APPLICATIONS. T.A. Friedmann, T.M Alam, J.P. Sullivan, T.E. Buchheit, W.K. Schubert, P.G. Kotula, 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 stress relieved by thermal annealing. We have studied the stress relaxation kinetics using an 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 strain-relieving transformations between sp^2 and sp^3 carbon.[1,2] Recently, we have made fully 13C enriched films by ablating from a ^{13}C (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 Raman, SEM, TEM, and cross-section EELS experiments.
The low stresses that are achievable in a-D makes this material suitable for surface-micromachined microelectromechanical systems (MEMS). We have demonstrated several MEMS structures from this material. One set of structures includes simple one-level cantilever beams and tensile pull tabs designed to evaluate the micromechanical properties of these films. Tensile test results were obtained by using a nanoindentor to pull laterally on the samples until fracture occurred. The a-D fracture strength was found to be dependent upon the gauge volume in a manner consistent with simple Weibull statistics.

[1] J.P. Sullivan, T.A. Friedmann, and A.G. Baca, J. Electr. Mat. 26, 1021 (1997).

[2] O.R. Monteiro, J.W. Ager, D.H. Lee, R. YuLo, K.C. Walter, and M. Nastasi, J. Appl. Phys.; 88, 2395 (2000).

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3:30 PM *L8.6

A NEW MICROMACHINED TEST STRUCTURE FOR IN SITU FATIGUE STUDY OF METAL THIN FILMS. M.P. de Boer^a, C.M. Matzke^b, M.S. Baker^a, S. Montague^a, T. Lemp^a and P.G. Kotula^c, Sandia National Laboratories, ^{a}MEMS & Novel Si S&T, ^{b}Microdevice Technologies, ^{c}Materials Characterization.

The use of metals is becoming increasingly important in mechanical thin film device applications such as micromirrors 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 general method for preparing optimized structures to study these 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 GPa regime, thereby inducing plastic deformation. Layout geometry allows the degree of stretching versus bending stress in the film to be controlled. Preparation 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 3-D 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 amenable to in situ plan-view (and cross-section) imaging by transmission electron microscopy while the film is actively loaded to a controlled stress level. This work will lead to an improved understanding of the reliability of metal films to be used for structural applications.
Acknowledgement: 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 L8.7

IN-SITU MECHANICAL CHARACTERIZATION OF A FREE-STANDING 100 NANOMETER THICK ALUMINUM FILM IN SEM USING MEMS SENSORS. M.A. Haque and M.T.A. Saif, Dept. of M&IE, 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 275 microns long freestanding 99.99% pure sputtered Aluminum film with grain size about 60 nanometers, tested insitu inside an SEM chamber. The specimen is cofabricated with MEMS force sensor and structures to produce following unique features, 1. Ability to measure any prestress in the specimen, 2. Reduction of any force misalignment by five orders of magnitude, 3. Elimination of any gripping mechanism, 4. Small setup size to fit SEM and TEM chambers, and 5. Ability to measure creep activation energy. The MEMS force sensor is calibrated with a nanoindenter. In the first loading cycle, the specimen behaved linear elastically up to 460 MPa. The elastic modulus value was found to be 69.6 GPa. The specimen showed no viscoelasticity upon unloading. In the second cycle, it started to show yielding at about 625 MPa. At about 680 MPa, loading was stopped and creep was observed. As the specimen creepd, the MEMS force sensor reduced the stress to 625 MPa in about 15 minutes, and no creep was observed below that stress. This gives the value of creep activation energy of 22 kcal/mole (bulk value 27 at 300 K). Upon unloading, the specimen again behaved linear elastically. The loading history and the materials response for the third cycle was identical to the second cycle. The specimen was then strained upto 3.6% (675 MPa stress) without failure. At this point, voids with mean diameter 300 nanometers were seen on the specimen. The experimental results strongly suggests that 1. The grains deform linear elastically upto 625 MPa, after which creep mechanism produces the yielding effect, 2. There is no strain hardening at this size scale, which indirectly suggests the creep mechanism is not dislocation mechanism based, 3. The material started creeping whenever the stress exceeded 625 MPa.

4:15 PM L8.8

ADSORPTION-INDUCED FAILURE MODES OF THIN-FILM RESONATORS. Robert Kazinczi, Jeff Mollinger, Andre Bossche, Delft Univ of Technology, Dept of Information Technology and Systems, Delft, THE NETHERLANDS.

The further downscaling of micromechanical 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 surface adsorbs atoms and molecules from the surrounding media. The adsorbates change the surface tension and the mass of the micromechanical structure. 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 0.001N/m. This corresponds to a compressive stress of 3MPa in the surface film. Oxy-nitride formation 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 3% due to cracking of the absorbed and/or oxide layer, than recovers logarithmically. The initial drop and the recovery rate is environment dependant. 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 L8.9

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 (TMS) as the silicon precursor at 100-200°C in the pressure range of 2–8 Torr. PECVD TMS 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 TMS 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 confirmality with respect to deposition conditions were discussed in this study. The adjustable stress and conformality of the PECVD TMS 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 L8.10

THE DESIGN OF MULTILAYERED POLISILICON FOR MOEMS APPLICATIONS. D. Sherman^a, H. Kahn^a, S.M. Phillips^b, R. Ballarini^c and A.H. Heuer^a, Case Western Reserve University, Cleveland, OH; ^{a}Department of MS&E; ^{b}Department of Electrical Engineering and Computer Science; ^{c}Department of Civil Engineering.

A rigorous analysis of a multilayered polysilicon laminated system, constructed by alternating two low-pressure 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 different crystallization 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 stresses as a function of layer thickness, as well as a procedure to design the desired curvature of a multilayered device. A linear deformation field is assumed. It is shown that precise design of the thicknesses of the individual layer is a prerequisite for controlled curvature. The procedure we have developed predicts the curvature of single and multilayered polysilicon systems with good precision.

SESSION L9: ADHESION AND FRACTURE II

Chairs: Neville R. Moody and Cheng-Hsin Chiu
Wednesday Evening, November 28, 2001
Room 304 (Hynes)

8:00 PM *L9.1
THIN-FILM PRESSURE SENSITIVE ADHESIVES: APPLICATIONS IN TRANSDERMAL DRUG DELIVERY. Reinhold H. Dauskardt, 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 is complicated by their highly viscoelastic properties. As such, there are almost no reproducible test methods or quantitative adhesion data available for this class of materials. In this presentation, a mechanics approach to quantify the adhesive properties of representative PSAs is described. Delamination of PSAs is accompanied by cavitation in the PSA and the formation of an extensive cohesive zone behind the debond tip. The presence of such rate-dependent, large-scale bridging provides 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 these adhesives through controlled modifications of cross-link density and pharmaceutical loading 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. Bilello, Center for Nanomaterials Science, Department of MS&E, 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 x-ray imaging/storage system with $\sim 20\mu$m resolution. The system is capable of recording 30 images/sec; hence, at 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 and the adhesion strength are correlated with the physiochemical nature of the coating-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 thin coatings. This work is supported, in part, by the ARO under GRANT #DAAG 55-98-1-0382, and the authors also thank USDoE for support for work done at SSRL.

8:45 PM L9.3
CRITICAL THICKNESS FOR CRACKING OF $Pb(Zr_{0.53}Ti_{0.47})O_3$ THIN FILMS DEPOSITED ON Pt/Ti/Si(100) SUBSTRATES. Ran Fu, Ming-Hao Zhao, Dexin Lu, and Tong-Yi Zhang, Hong Kong Univ of Science and Technology, HONG KONG.

This paper studied the critical thickness for cracking of $Pb(Zr_{0.53}Ti_{0.47})O_3$ (PZT) thin films deposited on Pt/Ti/Si(100) substrates. The Pt/Ti layers with thicknesses of 150nm and 30nm, respectively, were deposited on the substrates by the RF sputtering. Then, the $Pb(Zr_{0.53}Ti_{0.47})O_3$ thin films were deposited using the sol-gel 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 0.80mm 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 characteristic of the film/substrate system.

9:00 PM L9.4
STRESS DEVELOPMENT AND ADHESION BEHAVIOR IN THIN CERAMIC COATINGS MONITORED BY POSITRON ANNIHILATION DURING BENDING. R. Escobar Galindo[a], A. van Veen[a], H. Schut[a], N. Carvalho[b], C. Strondl[c], J. Th. M. de Hosson[b]. [a]Interfaculty Reactor Institute, Delft University of Technology, Delft, THE NETHERLANDS; [b]Materials Science Centre and NIMR, University of Groningen, Groningen, THE NETHERLANDS; [c]Hauzer Techno Coating, Venlo, THE NETHERLANDS.

Positron 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 positron beam. This configuration allows studying stress development and delamination phenomena of different coating/substrate systems. Varying the positron implantation energy, the coating layer or the coating/substrate interface can be studied separately. A typical experiment consists of monitoring the positron annihilation parameters 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, stresses can be released or increased, e.g., from an initial compressive state to a tensile state. Positrons have proven to be very well capable of observing the transition from compressive to tensile state in thin sputtered 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:H (2.5μm) layers on tool steel and low carbon steel substrates.

9:15 PM L9.5
WEAR AND ADHESION OPTIMIZATION OF THIN TiAlN-, CrN- AND C-FILMS BY STRUCTURE DESIGN. O. Knotek[a], E. Lugscheider[a], K. Bobzin[a], A. Leyendecker[b], G. Erkens[b], [a]Material Science Institute, University of Technology Aachen, GERMANY; [b]CemeCon GmbH, GERMANY.

The structure of TiAlN-, CrN- and the other thin films depends on coating methodes, 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 surface. 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 an 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 of application a columnar structure is to be preferred in other the distribution of the harder phases in the matrix has to be optimized in size, amount and location. The homogenity is not always the optimum. The use of different targets and a continuous change of the reactive gas mixture can influence these structure variations 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. Patrick Wellner, Oliver Kraft and Eduard Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

NiAl thin films can be used as protective coatings for structural components 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, NiAl films with different compositions (45.0 to 51.5 at-% Al) and thicknesses (0.4 to 3 μm) were deposited by magnetron co-sputtering onto silicon substrates and annealed at 600°C in ultrahigh vacuum. The films exhibited grain sizes comparable to the film thickness. Furthermore, the films were found to have a fiber textures with the strongest component being (110) for the Ni-rich and (111) for the Al-rich films. The stress evolution in the films between room temperature and 850°C was studied using the wafer-curvature technique. Above 700°C, the film stresses were completely relaxed indicating that very effective processes for plastic deformation are present. On cooling, tensile stresses of the order of 1 GPa developed. Although such high stresses developed in all films investigated, only the Al-rich NiAl films exhibited severe cracking, indicating that the fracture toughness depends on composition. Based on a simple estimate, the fracture toughness of these films was determined to be about 1 MPa $m^{-1/2}$, which is somewhat smaller compared to commonly reported bulk values for polycrystalline NiAl. Current investigations focus on the influence of film composition, thickness, and microstructure on the ductile-to-brittle transition during cooling.

9:45 PM L9.7
MECHANISMS OF FOREIGN OBJECT DAMAGE ON THERMAL BARRIER COATINGS. Rizhi Wang, Anthony G. Evans, Princeton Materials Institute, Princeton University, Princeton, NJ; Robert W. Bruce, GE Aircraft Engines, Cincinnati, OH.

Thermal Barrier Coatings (TBC) used in advanced gas turbines is susceptible to degradation upon impact by foreign objects, especially at high temperature. The responsible mechanisms have been analyzed by using Focused Ion Beam Microscope (FIB). The assessment was performed on samples composed of a Rene N5 nickel-based superalloy substrate, with a platinum-aluminide bond coat and a 7% yttria-stabilized zirconia thermal barrier made by electron beam physical vapor deposition. They were impacted with alumina particulates at 1230°C within a burner rig facility. FIB cross-sections at sites impacted by individual particulates revealed zones of plastic deformation in the TBC and bond coat, as well as inclined kink bands in the TBC. The kink boundaries initiated cracks that progressed toward the bond coat and then extended parallel to the interface within the TBC. These cracks are the source of subsequent spalling and material loss. A model characterizing this mechanism is developed and discussed.

10:00 PM L9.8
MEASURING MECHANICAL PROPERTIES OF THIN POLYMER FILMS USING A NANOSCALE JKR TEST. S.A. Syed Asif, Hysitron, Inc., Minneapolis, MN; and Kathryn J. Wahl, U.S. Naval Research Laboratory, Washington, DC.

Determining surface mechanical properties of small devices, thin films or small volumes may be impossible by traditional contact mechanics and nanoindentation methods, which lack either high spatial resolution or surface sensitivity. In this paper, we present a dynamic nanoscale Johnson-Kendall-Roberts (JKR) test to study adhesive contacts to polymer thin films. The nanoscale JKR test combines measurements of load and contact stiffness as a function of indenter penetration depth. We present localized mechanical property measurements (e.g. storage and loss moduli, adhesion energy, cohesive stress) for contacts with diameters smaller than the optical limit. Studies were performed using probes with tip radii between 1 and 10 microns against polydimethyl siloxane surfaces with varying cross-link densities. Smaller probe diameters and increased cross-link density shifted the measured response away from pure JKR into the Maugis-Dugdale transition regime. We compare storage modulus and surface energy measured from nanoscale JKR results to both calculated values and those measured with conventional nanoindentation.

SESSION L10: COMPUTATIONAL MODELING AND EXPERIMENTS I

Chairs: Jerrold A. Floro and Cengiz Sinan Ozkan
Thursday Morning, November 29, 2001
Room 304 (Hynes)

8:30 AM *L10.1
KINETICS OF BUCKLING OF A COMPRESSED FILM ON A VISCOUS SUBSTRATE. D.J. Srolovitz[a], N. Sridhar[b], Z. Suo[a] and B.N. Cox[b], [a]Princeton Materials Institute, Dept. of Mechanical and Aerospace Engr., Princeton University, Princeton, NJ; [b]Rockwell Science Center, Thousand Oaks, CA.

Viscous relaxation of glass substrates provides a means of relaxing stresses in thin films and islands. Compressively-stressed elastic films on finite-thickness viscous substrates undergo a buckling instability that relieves stresses but destroys the planarity of the film. A linear-stability analysis is performed to determine the onset, rate of growth and wavelength of this buckling instability as a function of the misfit strain, the film and viscous layer thicknesses, the elastic constants of the film and the viscosity of the glass. We find that the conditions for the onset of the buckling instability of the film on a glass layer is the same as that for a compressively-stressed free-standing film. Unlike the free standing film, the rate of the growth of the instability of the film on the viscous occurs slowly, with a characteristic time. At long times, the amplitude of the perturbation saturates and the wavelength of the perturbation increases. The linear perturbation analysis results is compared with a non-linear theory and recent experiments.

9:00 AM L10.2
PLASTIC DEFORMATION OF THIN FILMS. N. Nozaki, Y. Kogure and Masao Doyama, Teikyo University of Science and Technology, Uenohara, Yamanashi, JAPAN.

Copper thin single crystals were compressed, stretched, or bent. Each of the crystal contains several thousands of atoms. Molecular dynamics were used with the embedded atom potential which have been determined by the authors. The surfaces were free and the effect of surface was taken into account automatically, in a some sense. In all cases, dislocations were not easily created on smooth stretched surfaces. Dislocations were often created at the bent compression region. If a notch was made on a surface, Heidenreich-Shockley partial dislocations were created near the tip of the notch. Stress-strain curves were obtained. Followings are examples: A thin crystal which was bent contained 5068 atoms, 11a in [100], 6a in [010] and 32 a in [001]. The axis of the bend was [100]. Partial dislocations were created at the compressive region. Another thin crystal which was buckled contained 4550 atoms. It has 60a in [100], 5d in [1-10] and 8d in [001], where a is the lattice constant and d is the nearest neighbor distance. Partial dislocation started at the compressive region of the specimen. Atom movement during plastic deformation will be shown by a movie at the conference.

9:15 AM L10.3
MECHANICAL PROPERTIES OF THIN FILMS MATERIALS FOR THE 2D NUMERICAL ANALYSIS OF DEEP SUBMICRON CMOS TECHNOLOGIES. Vincent Senez, IEMN-ISEN, UMR CNRS-8520, Villeneuve d'Ascq, FRANCE; Giovanni Carlotti, INFM and Dipartimento di Fisica dell'Universita', Perugia, ITALY; Ingrid de Wolf, IMEC, Leuven, BELGIUM; Roberto Balboni, CNR-Istituto LAMEL, Bologna, ITALY; Alessandro Benedetti, Department of Electronic and Electrical Engineering, University of Sheffield, Sheffield, UNITED KINGDOM; Donata Piccolo and Gianfranco Mastracchio, STMicroelectronics, 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 thermo-mechanical 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 Beam Bending (BB). This method has been used to characterize TEOS and HDP silicon dioxide, LPCVD silicon nitride, sputtered titanium and C49/C54 TiSi2 which are largely used in the front end processes of modern deep submicron CMOS technologies. Subsequently, we have used a process simulator (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 conventionnal finite element methods. For the validation of the method, test structures have been designed and processed which consists in shallow trench isolation (STI) with different pitches where the active area 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 structures by convergent beam electron 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.

9:30 AM L10.4
ATOMISTIC THEORY OF ELASTICITY FOR EPITAXIAL GROWTH. Cameron Connell, Russel Caflisch, UCLA, Department of Mathematics, Los Angeles, CA.

We present an atomistic formulation of elasticity, suitable for calculations of strain in epitaxially grown thin films. Our method is based on the minimization of an elastic energy. The energy is constructed as the sum of the local deformation energies over all atoms in an atomic lattice. In particular, our energy can be regarded as a generalization of the Keating 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 atomistic nature of the energy allows it to be tailored to those local geometries where continuum theory does not apply, for instance at atomistically 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 solution. Some specific features we are able to incorporate are the lattice 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 BEYOND THE HALF-SPACE PARADIGM. Robert V. Kukta, State University of New York, Dept of Mechanical Engineering, Stony Brook, NY.

The elastic interaction between atomic steps determines an important part of the thermodynamic 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 half-space 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-ranged behavior that extends out to separation distances of 100 step-heights or larger in some cases. Energies of multiple-step configurations are also discussed where differences between the revised model and the half-space model are found to be even more substantial. Novel features of the model are (1) the elastic field cannot be represented by a line multipole on a half-space, (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. Cheng-hsin 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 semi-conductor 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 and patterns 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 cells, 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 that is stable against coarsening. The stability is further demonstrated by simulation for a model system that can develop pyramid 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 material properties. The implication of 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, Qizhen Li, Yunzhi Wang, Peter M. Anderson, Ohio State University, Dept of Materials Science and Engineering, Columbus, OH; Tim Foecke, National Institute of Standards and Technology, Metallurgy 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 values 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 energy of a dislocated material in terms of a sum of self and interaction energies of discrete dislocation line segments. The models then update dislocation positions according to a path of steepest decent in energy or 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.

11:15 AM L10.8

STRESS-STRAIN RESPONSE OF FREE STANDING GOLD THIN FILMS USING A NOVEL TESTING MACHINE. Sauri Gudlavalleti, Lallit Anand, Department of Mechanical Engineering, MIT, Cambridge, MA.

Free-standing gold thin films (500-700 nm thick) have been tested in simple tension using a novel testing machine. The testing machine enables measurement of both elastic and plastic response of the films with high resolution (20 nm for displacements and 25 μN for forces). Dogbone shaped specimens (gage width 1 mm and length 4 mm) were prepared by e-beam deposition on polished silicon wafers, followed by patterning and releasing using standard silicon micromachining techniques. The films exhibit grain sizes of 200 - 400 nm and a strong <111> texture perpendicular to the plane of the film. Tests show high initial yield strength, high rate of strain hardening, and high ultimate strength but low ductility. TEM studies show that the high initial strength and high strain hardening rate is attributable to the small grain size and very rapid and high dislocation density generation upon straining. A continuum crystal plasticity calculation including the initial crystallographic texture of the gold specimen is shown to match the elastic-plastic load-deflection curve of the specimens; the single threading dislocation models for the strength of thin films appear not to be applicable.

11:30 AM L10.9

KINETICS OF MOLECULAR ADSORPTION ON SOLID SUBSTRATES BY SURFACE STRESS. Guangming Li, Larry W. Burggraf, Dept of Engineering Physics, Air Force Institute of Technology, WPAFB, OH; William Baker, Dept of Mathematics and Statistics, Air Force Institute of Technology, WPAFB, OH.

There is renewed interest in adsorbate-induced changes in surface stress of compliant substrates like MEMS cantilevers for highly sensitive chemical and biological detection. We have investigated the stress responses of a series of chemically modified silicon cantilevers exposed to various chemical vapors at ambient conditions. Different types of adsorption kinetics were studied in systems with different substrate-adsorbate and adsorbate-adsorbate interactions. Significant oscillatory changes in surface stress were observed associated with the molecular adsorption due to the non-equilibrium thermodynamic surface phenomena in some surface systems. We modeled these surface kinetic phenomena based on a set of rate equations governing molecular adsorption, nucleation, growth, and decay, and proposed that the surface stress-based techniques can be used as a sensitive analytical tool to understand surface kinetic processes of the weakly interacting molecular systems.

11:45 AM L10.10

ASSESSMENT OF A NEW RELATION FOR THE ELASTIC COMPLIANCE OF A FILM-SUBSTRATE SYSTEM. Andrei Rar, George M. Pharr, The University of Tennessee, Dept of Materials Science & Engr, Knoxville, TN, and Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN; Haitao Song, Rice University, Dept of Materials Science and Engineering, Houston, TX.

A new closed-form relation for the elastic compliance of a film-substrate system indented by a flat cylindrical punch is presented. The relation is an extension of the work of Gao et al. (Int. J. Solids Struct. 29, 2471, 1992) modified in a way that increases the range of applicability to a wider range of modulus mismatch between the film and substrate. The accuracy of the relation is assessed by comparison to finite element simulations, other approximate numerical solutions, and nanoindentation experiments performed in simple systems. The relative benefits and limitations are discussed. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

SESSION L11: COMPUTATIONAL MODELING AND EXPERIMENTS II

Chair: Maarten P. de Boer
Thursday Afternoon, November 29, 2001
Room 304 (Hynes)

1:30 PM *L11.1

ANALYSIS OF NUCLEATION OF DISLOCATION LOOPS FROM THE SURFACE IN THE PEIERLS-NABARRO FRAMEWORK. Guanshui 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 Peierls-Nabarro framework. The stress dependent activation energies that are required to activate dislocation loops from their stable to unstable saddle point configurations are determined. Comparing to 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. Bunnik and Barend J. Thijsse, Delft University of Technology, Dept of Materials Science, Center for Research on Ion-Solid Interactions and Surfaces (CRISIS), 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 assited growth (10 eV per atom) were studied, in order to separate thermal effects from ballistic effects. We find that the heterostructure of the system gives rise to a complex evolutionary behavior. The first Cu monolayer grows as a perfect polymorphic bcc plane, after which 3D island growth sets in. The lateral island size is approximately 5 nm. Maximum roughness occurs after 0.7 nm nominal thickness. Then 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 fcc or hcp coordinated. Thereafter the growth continues steadily in a mosaic fcc(111)/hcp(111) surface orientation (i.e. not fully close-packed), while the roughness increases slowly, only 0.02 nm/nm. Threading 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. $< 111 >$ crowdions accumulate in the first Cu plane, and linear defects move around to 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 III-V NITRIDE THIN FILMS. Ashok Rajamani, R. Beresford, K.H.A. Lau, E.C. Chason, B.W. Sheldon, Brown University, Division of Engineering, Providence, RI.

AlN 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 inducing compression. The initial 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 based on film growth conditions such as island size, precursor super-saturation, and facet surface properties.

2:30 PM L11.4

ANALYSIS OF INELASTIC DEFORMATION IN POLYCRYSTALLINE METAL FILMS ON SUBSTRATES. Yoonjoon Choi and Subra Shuresh, 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 examined by exploring 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 Al 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. Swadener, 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 MS&E, 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 in iridium increased with decreasing depth of penetration. However, for spherical indenters, hardness was not affected by 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. *Research at the Oak Ridge National Laboratory SHaRE 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-AC05-00OR22725 with UT-Battelle, LLC.

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

Chair: Frans Spaepen
Thursday Afternoon, November 29, 2001
Room 304 (Hynes)

3:30 PM *L12.1

EFFECTS OF GROWTH KINETICS ON RESIDUAL STRESS DEVELOPMENT IN POLYCRYSTALLINE THIN FILMS. E. Chason, B. Sheldon, L.B. Freund, A. Rajamani, 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 simple 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 growth 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 Klingner, Peter Paufler, Institute for Crystallography and Solid State Physics, Dresden University of Technology; Thomas Holz, Reiner Dietsch, 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 thickness) were carried out by means of X-ray reflectometry, wide angle diffractometry and a novel laser mapping device. As the W/C multilayers were dedicated to technical application as X-ray monochromators and subjected to optimization of stacking parameters (thickness and number of layers) for a long term (mechanical) stability also further investigations will be discussed. Comparison of waver distortion 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 from a technical point of view. This is because 100 percent of the surface area (diameter 2 inches) could be converted from the smooth (as-deposited) to a structured (relaxed) state stable at room temperature. Investigations using optical and atomic force microscopy showed that the topology of the surface 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 the same height run along arbitrary directions. The structure thus obtained had characteristic lateral wavelengths of the order of 50 micrometer. The maximum height of the ridges was found to be between 2 and 3 micrometer. 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 ORIENTED NiTiCu SHAPE MEMORY THIN FILMS GROWN BY MOLECULAR BEAM EPITAXY. R. Hassdorf, J. Feydt, R. Pascal, S. Thienhaus, T. Sterzl, B. Winzek, M. Moske, Research Center Caesar, 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 materials are employed - mainly the classical NiTi-based alloys - for applications in medical technology like tooth braces, cardiovascular stents, microgrippers, 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 *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 two-dimensional 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-(002) orientation as well as the martensite-(022)/(002) orientations aligned along the film normal within ±3°. The splitting of the martensite-(022) 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 FILM. M.J. Daniels, Center for Nanomaterials Science, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI; D. King, Technology Assessment and Transfer, Annapolis, MD; J.S. Zabinski, 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 polymorphic nanoquasicrystalline grain structure, $\approx$ 2.5 - 10.0 nm. Subsequent annealing at 500°C for 4 hours, at base pressures of below $5*10^{-5}$ Torr, and with Ar flow to 5 - 10 mT, fully develops 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 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 $\approx$ 30 nm thick. Residual strain analysis shows that this second phase induces a compressive residual stress, and a nominal residual strain of 0.10% 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 POLE ENHANCED INDUCTIVELY COUPLED PLASMA. T. Meziani, P. Colpo, G. Ceccone, P. Leray, P.N. Gibson, D. Summa, and F. Rossi, European Commission-Joint Research Centre, Institute for Health and Consumer Protection, Ispra (VA), ITALY; P. Ranson GREMI-ESPEO, Université d'Orléans/CNRS, Orléans, FRANCE.

A novel inductively coupled plasma source (the Magnetic Pole Enhanced ICP or MaPE-ICP) designed and characterized in our laboratory was used for diamondlike carbon (a-C:H) deposition. The MaPE-ICP uses a magnetic pole to concentrate the magnetic flux on the load (i.e. plasma) and shows 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 from the ion flux bombarding the substrate, in contrast to capacitively coupled discharge. Diamondlike carbon coatings were deposited with this source from CH_4 and C_2H_2 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 detection and ion energy distribution measurements have been carried out at the substrate holder surface in order to identify directly the impinging species on the growing film. Coatings characterization including FTIR spectroscopy, Raman spectroscopy, and X-ray reflectivity (XRR) were undertaken for various processing 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

Chairs: Joost J. Vlassak and Antanas Daugela
Friday Morning, November 30, 2001
Room 304 (Hynes)

8:30 AM *L13.1
EVOLUTION OF INTRINSIC STRESS DURING DEPOSITION OF POLYCRYSTALLINE AND AMORPHOUS THIN FILMS. Jerrold A. Floro and Sean J. Hearne, 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 net 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 common 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 the stress evolution for Ag, Ti, and amorphous Si films in the context of possible stress generation mechanisms. Multilayer growths of amorphous Si / amorphous Ge will also be discussed, as these shed additional light on the origins of intrinsic stress in continuous films. 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.

9:00 AM L13.2
STRESSES DUE TO CHEMICAL POTENTIAL GRADIENTS IN NON-STOICHIOMETRIC OXIDE FILMS. Ramanathan Krishnamurthy and Brian W. Sheldon, 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 stresses 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 exist 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 dopant contents 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. Sheldon, K.H.A. Lau, and Ashok Rajamani, Brown University, Division of Engineering, Providence, RI.

Previous treatments of tensile, intrinsic growth stresses in polycrystalline films are based on the idea that stress is induced 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 results that contradict this picture will be presented. This work examines several different vapor deposited films with low atomic mobilities (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 tension relative to the interface side. A mechanics 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. Jaccodine, and T.J. Delph, Depts. 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. These were found to result in significant decreases in 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 examination 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 MnAl THIN FILMS. Gregory A. Fischer, Army Research Laboratory, Adelphia, MD; M. Lea Rudee, Graduate Program in Materials Science/Center for Magnetic Recording Research, U.C.S.D., La Jolla, CA; Vitali F. Nesterenko, Sastry Indrakanti, Graduate Program in Materials Science/Department of Mechanical and Aerospace Engineering, U.C.S.D., La Jolla, CA.

The effect of hot isostatic pressure processing (HIP) on MnAl films has been compared to vacuum annealing for the purpose of obtaining substantial amounts of tau-phase MnAl in films under 200 nm. Films were films deposited by dc sputtering from both MnAlNiC and MnAl targets. As-deposited films were nearly amorphous. Post deposition annealing in vacuum produced only small amounts of the ferromagnetic tau-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 MnAlNiC target, these changes in crystallinity were accompanied by changes in the M-H loops of the samples. MnAlNiC HIP samples had improved magnetic properties compared to those of equal thickness annealed in vacuum. The 100 nm HIP sample sputtered from the MnAl target also showed an increase in moment, though the changes were not as dramatic as those seen in the samples sputtered from the MnAlNiC 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 tau-phase in 100 nm films, the appropriate pressure for forming tau-phase in 50 nm films is yet to be determined.

10:30 AM *L13.6
THERMOMECHANICAL BEHAVIOR OF THIN Ag FILMS ON SUBSTRATES. Shefford P. Baker, Krishanu 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 encapsulated between 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, extensive plastic deformation at very low stresses, 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 fcc 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.F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN; P. Zschack, University of Illinois at Urbana-Champaign, APS-UNICAT, Argonne, IL; B.A. Pint, 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. This paper describes the initial results from the use of focused monochromatic synchrotron radiation to measure stresses in growing alumina films during high-temperature air exposures of Fe and Ni-based 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-AC05-00OR22725 with UT-Battelle, LLC. The UNICAT facility at the Advanced Photon Source (APS) is supported by the Univ of Illinois at Urbana-Champaign, Materials Research Laboratory (U.S. DOE, the State of Illinois-IBHE-HECA, and the 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. Ilan 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 metallizations. 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.

Amit Misra, Harriet Kung and Richard G. Hoagland, 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 nano-scale 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 $\leq$ 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. Spolenak[a,†], N. Tamura[b], B.C. Valek[c], M.D. Morris[a], W.L. Brown[a], A.A. MacDowell[b], R.S. Celestre[b], H.A. Padmore[b], J.C. Bravman[c], B.W. Batterman[b,d] and J.R. Patel[b,d]; [a]Agere Systems (formerly of Bell Laboratories, Lucent Technologies), Murray Hill, NJ. [†]now at Lehigh University, Bethlehem, PA; [b]ALS/ LBNL, Berkeley, CA; [c]Dept. Materials Science & Engineering, Stanford University, Stanford, CA; [d]SSRL/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 white x-ray beam 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.