SYMPOSIUM P

Dislocations and Deformation Mechanisms in Thin Films and Small Structures

April 17 – 19, 2001

Chairs

Shefford P. Baker
Dept of MS&E
Cornell Univ
129 Bard Hall
Ithaca, NY 14853-1501
607-255-6679

L. B. Freund
Div of Engr, Mech of Solids & Structures
Brown Univ
Barus-Holley Bldg, Box D
Providence, RI 02912-9104
401-863-1476

Robert Hull
Dept of MS&E
Univ of Virginia
116 Engineers Way
Charlottesville, VA 22904-4745
804-982-5658

Oliver Kraft
Max Planck Inst for Metallforschung
Stuttgart, 70174 GERMANY
49-711-209-5123

Klaus W. Schwarz
Dept of Physical Sciences
IBM T.J. Watson Research Center
Yorktown Heights, NY 10598
914-945-2879

Symposium Support
Agere Systems
†FEI Company
IBM T.J. Watson Research Center
†JEOL USA, Inc.
†MTS Systems Corp., Nano Instruments Innovation Center
†2001 Spring Exhibitor

Proceedings to be published in both book form and online
(see *ONLINE PUBLICATIONS* at www.mrs.org)
as Volume 673
of the Materials Research Society
Symposium Proceedings Series.

* Invited paper

SESSION P1: DISLOCATION AND DEFORMATION MECHANISMS IN THIN METAL FILMS AND MULTILAYERS I

Chairs: Klaus W. Schwarz and Ralph Spolenak
Tuesday Morning, April 17, 2001
Salon 10/11 (Marriott)

8:30 AM *P1.1

MODELING PLASTICITY IN POLYCRYSTALLINE GOLD THIN FILMS ON SILICON SUBSTRATES. W.D. Nix, O.S. Leung, Department of MS&E, Stanford University, Stanford, CA; D. Weiss, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

Modeling plasticity in gold thin films on silicon substrates is discussed with particular reference to the evolution of stresses during thermal cycling. Following the work of Kobrinsky and Thompson, we show that plasticity of unpassivated Au films is dominated by constrained diffusional flow of matter from the free surface of the film to the grain boundaries. This diffusional effect depends strongly on film thickness, as expected. The presence of a 10 nm thick passivation layer of W inhibits these diffusional relaxation processes and causes the stress evolution in the film to be dominated by dislocation flow. It is shown that a simple kinematic strain hardening law for plasticity, suggested by Suresh, together with a temperature-dependent elastic modulus, provides a good phenomenological account of the stress-temperature curves for passivated films. Using this modeling approach it is possible to draw a distinction between the yield strength of the film and the strain-dependent flow stress. Models of multiple misfit dislocation formation, developed separately by Willis and Freund, are used to rationalize the linear kinematic hardening laws used in this modeling. Efforts to model the stress-temperature behavior of unpassivated films by combining the effects of diffusional relaxation and dislocation mediated strain hardening will be reported.

9:00 AM P1.2

CONSTRAINED DIFFUSIONAL CREEP IN THIN Cu FILMS. D. Weiss, E. Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

Thermal stress evolution in polycrystalline thin metal films on substrates has been investigated for a long time. One focus of research has been the understanding of stress relaxation mechanisms responsible for a particular stress-temperature curve. In our work we studied the stress evolution in pure Cu and self-passivating Cu-Al alloy films on Si substrates using the wafer curvature method. We modeled stress-temperature curves of the pure Cu films using a creep law for grain boundary diffusional creep in a thin film under substrate constraint from the literature. Good matching of the theoretical curves even with subtle features of the experimental data was achieved. According to the constrained diffusional creep model, combined surface and grain boundary diffusion selectively relaxes film stress at the grain boundaries, producing an inhomogeneous stress state in the film. In our simple approach this stress state was approximated by only two stress components, an average grain boundary stress and an intragranular stress, the latter being not affected by the creep mechanism. Characteristic features of the pure Cu film curves, attributed to constrained diffusional creep, were absent in the alloy film curves, which were modeled with thermally activated dislocation glide as the only deformation mechanism. The suppression of diffusional creep in these films was explained by the retardation of surface diffusion, necessary for the creep mechanism. It is concluded that the modeling approach presented in this talk is valid to explain the thermomechanical behavior of a broader class of thin metal films on substrates, both with and without a passivation layer.

9:15 AM P1.3

AN EXPERIMENTAL AND COMPUTATIONAL STUDY OF THE ELASTIC-PLASTIC TRANSITION IN THIN METAL FILMS. Erica T. Lilleodden and William D. Nix, Stanford University, Dept of MS&E, Stanford, CA; Jonathan Zimmerman, Sandia National Laboratory, Livermore, CA; Stephen M. Foiles, Sandia National Laboratory, Alburquerque, NM.

Nanoindentation studies of thin metal films have provided insight into the mechanisms of plasticity in small volumes, showing a strong dependence on the film thickness and grain size. It has been previously shown that an increased dislocation density can be manifested as an increase in the hardness or flow behavior of a material, as described by the Taylor relation. However, when the indentation is confined to very small displacements, the observation can be quite the opposite; an elevated dislocation density can provide an easy mechanism for plasticity at relatively small loads, as contrasted with observations of near-theoretical shear stresses required to initiate dislocation activity in low-dislocation density materials. Experimental observations of the evolution of hardness with displacement show initially soft behavior in small-grained films and initially hard behavior in large-grained films. Furthermore, the small-grained films show immediate hardening, while the large grained films show the 'softening' indentation size effect (ISE) associated with strain gradient plasticity. Rationale for such behavior has been based on the availability of dislocation sources at the grain boundary for initiating plasticity. Embedded atom method (EAM) simulations of the initial stages of indentation substantiate this theory, where the proximity of the indenter to pre-existing dislocations or grain boundaries is varied.

9:30 AM P1.4

THE ANELASTIC BEHAVIOR OF Cu THIN FILMS STUDIED AS A FUNCTION OF TEMPERATURE AND PLASTIC STRAIN. R. Spolenak[a], C.A. Volkert[b], S. Ziegler[a,c], W.L. Brown[a]; [a]Bell Laboratories, Lucent Technologies, Murray Hill, NJ; [b]Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY; [c]I. Physik. Inst. A., Lehrstuhl für Physik neuer Materialien, Aachen, GERMANY.

Most of the literature that reports on the mechanical properties of thin metallic films is based on substrate curvature measurements. This technique has one major draw back: the stress applied cannot be decoupled from temperature. In order to overcome this drawback, we have developed a new version of a bulge tester that combines the capacitive measurement of the bulge deflection of a membrane with a resonance frequency measurement of the residual stress in the membrane. The combination of these two modes of operation has enabled us to study the anelastic behavior of electroplated and sputtered 1 μm thin Cu films. A membrane is plastically deformed to a pre-determined strain by controlled gas-pressure bulging of the membrane. After the bulging stress is removed, the residual tensile stress, which has been decreased by the plastic deformation, is then determined by measuring the resonance frequency as a function of time. Immediately after plastic straining, the residual (tensile) stress of membranes was observed to increase. At room temperature a maximum stress was typically reached in the order of an hour. At still longer times the stress decreased again as a result of creep. The transient increase in stress following plastic straining was larger as the amount of plastic strain produced by bulging was increased. With higher temperatures the transient became both faster and larger. At 200°C the phenomenon became so strong that the stress state the membrane had before plastic straining was reached before the maximum in the transient. These results will be compared with the strain rate sensitivity of deformation and a dislocation-based mechanism will be suggested. Differences in the behavior of sputtered and electroplated Cu films will be presented and discussed in terms of differences in their microstructure and texture.

9:45 AM P1.5

IN SITU EXPERIMENTS OF CURVATURE AND X-RAY DIFFRACTION DURING ANNEALING OF Al(Si,Cu) FILM AND Ti/Al(Si,Cu) BILAYER. Ola Bostrom, Philippe Boivin, STMicroelectronics, Cedex, FRANCE; Patrice Gergaud, Olivier Thomas, Laboratoire TECSEN, UMR CNRS, Universite d'Aix-Marseille III, Marseille Cedex; Bernard Chenevier, LMGP, CNRS, ENSPG, St Martin d'Hëres, FRANCE; Marc Legros, LPM, Ecole des Mines, Nancy, FRANCE.

The measurement of the induced substrate curvature has long been known to be a very useful way to follow the average stress evolution in thin films. In the absence of any information on the microstructure evolution, such stress vs temperature curves are difficult to interpret and the under laying mechanisms remain hidden (plasticity, grain growth, precipitation, solid state reactions, etc.). We combine x-ray diffraction and curvature measurements performed in-situ as a function of temperature. This is applied to Al(Si,Cu) thin films where we show that the initial stress drop around 120°C -140°C during the first heating cycle often associated with grain growth and/or recrystallization is instead an effect of RT microstructural hardening. We have also studied Ti/Al bilayers where the solid state reaction and its influence on the stress are followed simultaneously. The creation of $TiAl_3$ is expected to induce large stress because of the volume change of 6% between the intermetallic phase and the initial species. Surprisingly, during the first stage of the reaction, whereas the intensity of a $TiAl_3$ peak continues to increase, there is no evolution of the sample average stress.

10:30 AM *P1.6

MONOTONIC AND CYCLIC MICRO-TENSILE TESTING OF THIN METAL FILMS. Richard P. Vinci, Lehigh University, Dept of MS&E, Bethlehem, PA.

Freestanding metal thin films are of interest for use in micromechanical and microelectronic devices such as pressure sensors and switches. Micro-tensile testing is one of the most direct methods for measuring such properties, but the technique is challenging to perform accurately due to the small dimensions of the samples. It has been observed that elastic moduli of freestanding metal films tested by micro-tensile techniques are often lower than those of equivalent bulk materials by as much as 10-20%. A likely source of this modulus defect is relaxation due to micro-plastic or anelastic processes. Relaxation during testing leads to decreasing stresses for a given applied strain, or increasing strains for a given applied load. The measured modulus is therefore dependent upon the degree of relaxation that takes place during the time of the measurement. This issue, and others that arise during micro-tensile testing, will be discussed in the context of monotonic and cyclic tests performed with thin metal films.

11:00 AM P1.7

MICROPLASTICITY AND HIGH DUCTILITY DURING MICROTENSILE TESTING OF FREE-STANDING ALUMINUM FILMS.

R.R. Keller, D.T. Read, Y.-W. Cheng and J.D. McColskey, National Institute of Standards and Technology, Materials Reliability Div, Boulder, CO.

We have conducted microtensile tests on free-standing aluminum films of thickness 1 μ m, width 10 μ m, and length 180 μm, and investigated their microstructures by transmission electron microscopy without further thinning. Ductilities in excess of 20% were observed, in contrast to many earlier thin film tensile test studies which showed elongations to failure more typically near 1%. Whereas monotonic loading of coarse-grained bulk aluminum to strains of 20% usually results in the formation of lower energy dislocation arrangements such as dense tangles or cells in the vast majority of grains, we did not observe such structures. Rather, isolated dislocation segments, loose tangles and even dislocation-free grains were seen in post-mortem observations on as-strained specimens. Despite this seemingly surprising result, we show that the constrained dimensions of the thin film system affect slip activity in a predictable manner. Namely, the films do contain a sufficient density of geometrically-necessary dislocations during straining to allow for more than 20% elongation to occur via room temperature deformation mechanisms. Two factors may contribute to the observed high ductility, through suppression of necking: the observed high strain rate sensitivity and the constrained specimen geometry, particularly in the gauge width. Possible dislocation mechanisms associated with these factors will be discussed. Finally we present elastic modulus measurements from these specimens, which appear to also be dependent upon the constrained geometry.

11:15 AM P1.8

DEFORMATION MECHANISMS IN Cu THIN FILMS AS STUDIED BY MICRO-TENSILE TESTING.

Oliver Kraft, Andreas Bredereck, Ruth Schwaiger, Martina Hommel, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

Uniaxial tensile testing is one of the most important techniques for investigating the mechanical properties of bulk metals. However, tensile testing has been rather rarely applied to thin films because of their extreme fragility. To make sample handling easier, we have developed a technique to investigate the stress-strain behavior of thin metal films on a substrate. Cu films (thickness 0.4 to 3 μm) were deposited onto compliant polymer substrates. Then, film and substrate were strained simultaneously in tension while the film stress was determined by x-ray measurements. This technique allows one (i) to measure the stress in differently oriented grains, e.g. with (111)- and (100)-orientation independently, (ii) to monitor the x-ray peak width as a measure for the dislocation density during the tensile test, and (iii) to characterize the time-dependence of stress relaxation as a function of initial strain. Also, on unloading the sample, the film material is deformed in compression because it was, in contrast to the substrate, plastically deformed in the forward direction. As such, cyclic deformation could also be explored. Our results suggest that the stress-strain behavior of thin films can be described by three subsequent stages, (i) elastic, (ii) plastic with a strong increase in stress and dislocation density, and (iii) plastic with a moderate increase in stress and dislocation density. On unloading, a strong Bauschinger effect was observed. In contrast to the initial forward loading, yielding of the film was observed at a lower stress, and it is associated with a decrease in dislocation density. Yield stress and the extent of strain hardening were found to increase with decreasing film thickness and grain size; and the (111)-oriented grains are about two times stronger than the (100)-oriented grains. Based on these observations, implications for a theoretical description of the deformation in thin films are critically discussed.

11:30 AM P1.9

STUDY OF THE YIELDING AND STRAIN HARDENING BEHAVIOR OF Cu THIN FILMS ON Si SUBSTRATES USING MICROBEAM BENDING.

Jeffrey Florando, and William D. Nix, Dept. of Materials Science and Engineering, Stanford University, Stanford, CA.

There is a continuing need for the development of new techniques for studying the mechanical properties of thin films on substrates. Recently a new microbeam bending technique utilizing triangular beams was introduced. For this geometry, the film on top of the beam deforms uniformly when the beams are deflected, unlike the standard rectangular geometry in which the bending is concentrated at the support. The yielding behavior of the film can be modeled using a Ramburg-Osgood constitutive law, which is then used to predict the stress-strain relation for the film while attached to its substrate. This model has also been used to show that although in beam bending there is a gradient of stress and strain through the thickness of the film, this effect does not obscure the measurement of the yield stress in our analysis. Utilizing this technique, the yielding and strain hardening behavior of both bare and passivated Cu thin films has been investigated. After the bare films were deposited and tested, they were annealed at 500°C for 30 minutes, and re-tested to examine the effect of grain size. The beam bending results were compared to wafer curvature measurements as well as previous plasticity studies done on sputtered Cu films on substrates. The similarities and differences in the results will also be discussed.

11:45 AM P1.10

MECHANICAL BEHAVIOR OF THIN COPPER FILMS STUDIED BY A FOUR-POINT-BENDING TECHNIQUE.

Volker Weihnacht, Winfried Brückner, Institute of Solid State and Materials Research Dresden, GERMANY.

For thin metallic films on Si substrates, mostly the thermal-cycling technique has been used to exert stress into the films. This testing method is hampered by the fact that stress and temperature cannot be varied independently. Furthermore, little information about the mechanical behavior under compressive stress can be obtained at lower temperatures. A way out of this limitation is external loading, e. g., by four-point bending of coated substrates. A dedicated apparatus was constructed for such bending experiments in combination with thermal cycling in high vacuum. It enabled us to impose both positive and negative strains up to 0.8 % on Cu films on beam-shaped oxidized Si substrates at different temperatures. Before each bending experiment, a state free of thermal stress was adjusted by a convenient thermal pretreatment. After relief of the bending, the internal stress could be measured by the wafer-curvature method. Using this technique, Cu films of 0.2, 0.5, and 1.0 μm thickness were investigated at various temperatures. Some important findings at lower temperatures (<250°C) are: (i) The amount of plastic strain introduced by external bending increases with film thickness. (ii) The absolute values of the introduced plastic strains are very low throughout (<0.1 %). (iii) The yield behavior is asymmetric in tension and compression. At higher temperatures, there is no clear thickness dependence and no asymmetry in tension and compression. These results are discussed in view of recently proposed dislocation mechanisms for thin-film plasticity. Especially the accumulation and interaction of dislocations near to the film-substrate interface are considered. Close relations between our findings in the four-point-bending experiments and some features of thermal-cycling behavior described in the literature seem to exist.

SESSION P2: DISCRETE DISLOCATIONS: OBSERVATIONS AND SIMULATIONS

Chair: Frances M. Ross
Tuesday Afternoon, April 17, 2001
Salon 10/11 (Marriott)

1:30 PM *P2.1

IN-SITU TRANSMISSION ELECTRON MICROSCOPY STUDIES OF DISLOCATIONS IN METALLIC THIN FILMS.

Eric A. Stach, Ulrich Dahmen, National Center for Electron Microscopy, Lawrence Berkeley National Laboratory, Berkeley, CA; Andrew M. Minor, J.W. Morris, Jr., Department of Materials Science and Mineral Engineering, University of California, Berkeley, CA; William D. Nix, Department of MS&E, Stanford University, Palo Alto, CA.

It is well established that the mechanical behavior of thin metallic films constrained by rigid substrates is significantly different than that of the same material in either bulk or unconstrained forms. Because it is necessary to test thin film mechanical behavior while substrate constraint is maintained, a number of novel techniques have been developed over the past decade to achieve this goal. These include wafer curvature, microbeam bending, bulge testing and nanoindentation. In each of these techniques, either a direct or indirect determination of the strain response of the sample to a thermal or mechanical load is made, and the resulting changes in microstructure are inferred. In this presentation, I will discuss how we have used in-situ transmission electron microscopy to observe in real time the dislocation mechanisms that allow for strain accommodation in these systems. In particular, I will focus on the mechanisms of dislocation nucleation, propagation and interaction during in-situ wafer curvature of bicrystalline aluminum films as well as on observations of the nanoindentation of aluminum films on silicon made with a specially designed in-situ nanoindentor. In the case of wafer curvature, we have found that the high yield strengths present in these materials result from the difficulty of dislocation nucleation, and not solely from the combined effects of substrate constraint, Hall-Petch strengthening and forest hardening. Our real time observations of nanoindentation have allowed determination of the dislocation geometry associated with the introduction of geometrically necessary dislocations, as well as direct observations of dislocation interactions with the film - substrate interface. Throughout the discussion, we will focus on the experimental methods necessary to obtain quantitative information about dislocation and deformation mechanisms.

2:00 PM P2.2

DISLOCATION DYNAMICS SIMULATIONS OF DISLOCATION INTERACTIONS IN THIN FCC METAL FILMS. Prita Pant, Shefford P. Baker, Department of Materials Science and Engineering, Cornell University, Ithaca, NY; K.W. Schwarz, IBM Research, Yorktown Heights, NY.

Mesoscopic simulations of interacting dislocations in thin metal films were performed using the PARANOID dislocation dynamics code. In these simulations, dislocations are discretized into points, and the force acting to move each point is calculated taking into account the inherent stresses, the self-stresses of the dislocations and stresses due to other dislocations. Interactions between threading dislocations and misfit dislocations involving various combinations of glide planes and Burgers vectors were simulated. The configurations resulting from several loading and unloading cycles were studied. The results may provide a basis for understanding some interesting phenomena related to thin film plasticity. It is hoped that such simulations can be used to get a better insight into high yield stress, asymmetry in yield under compressive and tensile loading, and 'negative yielding' (plastic yielding resulting in an increase in film stress) - all phenomena observed in thin metal films.

2:15 PM P2.3

DISCRETE DISLOCATION SIMULATION OF THIN FILM PLASTICITY. Burghard von Blanckenhagen, Peter Gumbsch, Eduard Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

The flow stresses of polycrystalline thin metal films are much higher than the flow stresses for the corresponding bulk material and scale approximately with the inverse film thickness. The dislocation motion is restricted by the confined geometry, but the detailed mechanisms which cause the high stresses are not well understood. Current models deal with the deposition of dislocations at interfaces and boundaries (Nix–Freund model, Thompson model, Hall–Petch model). These models explain qualitatively the increase in the flow stress with decreasing film thickness (or grain size), but the predicted flow stresses are much smaller than the experimentally measures ones. Discrete dislocation dynamics simulations compute the movement of dislocations under an applied stress, taking into account the interaction between different dislocations, the dislocation self–interaction, boundary conditions and dislocation multiplication or recombination. They offer themselves to investigate dislocation glide in a thin film because configurations can be treated which are not tractable analytically and the number of dislocations is still relatively small due to the confined geometry. We simulated the plastic deformation of a columnar grain in a polycrystalline thin film including at the same time the essential elements of the aforementioned models and additionally dislocation sources and work hardening. A new model is proposed, based on the assumptions that the number of dislocation sources per grain is small, thereby requiring multiple activation of a source. The model predicts a flow stress proportional to the reciprocal of the smallest grain dimension (film thickness or grain size) and approximately four times larger than estimated by the Nix–Freund model. For larger grains and larger strains a Hall–Petch behaviour is found. The influence of the interfaces is investigated by comparing calculations for a film sandwiched between a substrate and a capping layer with those for a free standing film.

2:30 PM P2.4

IN-SITU OBSERVATION AND SIMULATION OF DISLOCATION MOTION IN THIN FOILS. D.H. Lassila, M.M. LeBlanc, G. Kay, M. Rhee and M. Wall, Lawrence Livermore National Laboratory, Livermore, CA; H. Zbib, Washington State University, Pullman, WA.

In-situ transmission electron microscope (TEM) experiments have been performed to directly observe the mobility as a function of stress in thin foils on the order of $100\mu m$ thick. An in-situ straining stage was designed and constructed that allowed a constant tensile load to be applied to a sample. Videotape of dislocation motion in high-purity molybdenum under a know applied load is used to construct plots of dislocation velocity as a function of applied load. Modeling of the experiments is being performed to investigate the effect of stress state on mobility. First, a 3-D finite element computer code simulation of the test sample using orthotropic elasticity is performed to determine the bi-axial state of stress in the where dislocation activity was observed. This stress state is then used as a boundary condition in a 3D dislocation dynamics simulation that includes free surface effects. Comparison of experimental results with the simulations allow for validation of dislocation mobility rules that have been derived from atomistic simulations or inferred from experimental techniques such a compression and tensile loading of bulk samples. We are also using these techniques to investigate dislocation pile-ups and cross slip phenomena.

2:45 PM P2.5

Abstract Withdrawn.

3:30 PM *P2.6

INFLUENCE OF FILM/SUBSTRATE INTERFACE STRUCTURE ON PLASTICITY IN THIN METAL FILMS. Gerhard Dehm, Max-Planck-Institut fuer Metallforschung, Stuttgart, GERMANY.

The stresses that develop in thin metal films deposited on rigid substrates can significantly exceed the flow stresses in the corresponding bulk metal. This phenomenon has been attributed to geometrical constraints on the film which alter the energetics of dislocation motion. Dislocation-based models for the flow stress assume that dislocations moving in the metal film on glide planes inclined to the film/substrate interface must create dislocation lines at this interface. In the present study, plan-view and cross-sectional transmission electron microscopy (TEM) specimens of thin metal films on substrates were thermally cycled in-situ in a TEM in order to analyze dislocation-based plasticity and dislocation-interface interactions. The experiments revealed that dislocation motion is influenced by the structure of the film/substrate interface. For Cu and Al films grown on silicon wafers coated with amorphous diffusion barriers, the TEM studies indicated that dislocations are pulled towards the interface, where their contrast finally disappears. However, for Al films deposited on Al_2O_3-substrates, threading dislocations advance through the layer and drag behind interfacial dislocation segments. In this case, the interface is between two crystalline lattices. Furthermore, the in-situ TEM experiments were correlated with the mechanical properties of the Cu and Al films, which were measured by the substrate-curvature method using a laser scanning technique during thermal cycling. The in-situ TEM studies revealed continuous dislocation glide at elevated temperatures, while jerky dislocation motion occurred at low homologous temperatures. Since dislocation density remained nearly constant at values of $\sim 3.10^9 cm^{-2}$ to $\sim 6.10^9 cm^{-2}$, independent of the film stress, it is assumed that the decrease in available thermal energy with decreasing temperature impedes dislocation motion and thereby contributes to the high flow stresses of the thin metal films.

4:00 PM P2.7

EFFECTS OF FILM THICKNESS ON PLASTICITY IN COPPER THIN FILMS. T. John Balk, Gerhard Dehm and Eduard Arzt, Max-Planck-Institut für Metallforschung, Stuttgart, GERMANY.

Thin metal films on substrates are able to carry significantly higher stresses than their bulk metal counterparts. Moreover, with decreasing film thickness, progressively higher stresses develop in the films during thermal cycling. In the current study, the effects of film thickness on dislocation motion and on the evolution of flow stress in thin Cu films have been investigated using transmission electron microscopy (TEM). Unpassivated pure Cu films were deposited onto Si wafers coated with SiO_x and a SiN_x diffusion barrier, resulting in primarily a $< 111 >$ texture. The films, with thicknesses ranging from 270 nm to 800 nm, were produced under both HV and UHV conditions using magnetron sputtering and in each case were immediately annealed in vacuum for 10 minutes at 600°C. The increase in flow stress that is observed upon decreasing the film thickness was studied in conjunction with in-situ thermal cycling of plan-view and cross-sectional specimens in the TEM. The motion of dislocations, especially their interaction with the film/substrate interface, and the evolving dislocation microstructure were studied as a function of the film thickness and grain size. These geometrical constraints were found to be significantly larger than the spacing between pinning points that are encountered by the gliding dislocations. The motion of dislocations was observed to be smooth and continuous at high temperatures, but became jerky below 200°C.

4:15 PM P2.8

SIMULATION OF GRAIN BOUNDARY STRENTGHENING MECHANISM WITH DISCRETE DISLOCATION DYNAMICS. S.B. Biner and J.R. Morris, Ames Laboratory, Iowa State University, Ames, IA.

The mechanism of grain boundary strengthening and the origin of the Hall-Petch relationship are investigated using a numerical method that combines the finite element method with interacting, discrete dislocations. The dislocations are modeled as line defects in a linear elastic medium. At each instant, superposition is used to represent the solution in terms of the infinite-medium solution for the discrete dislocations and a complementary solution that enforces the boundary conditions. The long-range dislocation interactions are calculated efficiently using the multipole method. Dislocation annihilation, generation and pinning at the grain boundaries are simulated through a set of rules in our constitutive model. We will examine the hardening behavior as a function of the dislocation source density and with nucleation behavior at the sources. We will also examine the role of the stress concentrations at dislocation pile-ups and of grain orientation. The details of the implementation of the numerical method for parallel computing in a cluster environment will also be described. This work was performed for the DOE by Iowa State University under contract W-7505-Eng. and also supported by Director of Energy Research, Office of Basic Sciences.

4:30 PM P2.9

OBSERVATION OF DOMAIN MOTION IN SINGLE CRYSTAL BARIUM TITANATE UNDER COMBINED ELECTRO-MECHANICAL LOADING CONDITIONS. Eric Burcsu, G. Ravichandran, K. Bhattacharya, California Institute of Technology, Division of Engineering and Applied Science, Pasadena, CA.

The nonlinear electro-mechanical behavior of ferroelectric materials is governed by the motion of domains. Since many common ferroelectric materials, such as barium titanate and PZT, are also ferroelastic, the domain motion is highly affected by stress as well as electric field. Experiments are performed on (001) and (100) oriented single crystals of barium titanate under combined electro-mechanical loading conditions. The crystal is subjected to a constant compressive stress (dead load) and an oscillating electric field of along the [001] direction. Global deformation and polarization are measured as a function of electric field at different values of compressive stress and input frequency. The use of semi-transparent electrodes and transmitted illumination allows in situ, real-time microscopic observations of domain motion using a long working-distance, polarizing microscope. The combined electro-mechanical loading results in a cycle of stress and electric field induced 90° domain switching. This is an electrostrictive behavior, with measured strains of greater than 0.8%. A degradation of the strain response is observed that is a function of applied stress and input frequency. This degradation can be correlated with crack formation during ferroelectric switching. Interactions of domain motion with defects, such as cracks, are observed under the combined loading conditions.

4:45 PM P2.10

REVERSIBLE STRAINS AND POLARIZATION OF POLY-CRYSTALLINE FERROELECTRIC THIN FILMS. JiangYu Li and Kaushik Bhattacharya, Division of Engineering and Applied Science, California Institute of Technology, Pasadena, CA.

Electrostriction in ferroelectric single crystal through domain switch has been identified as an effective way to provide large actuation strain. The electrostrictive strain in polycrystalline ferroelectric, however, is very limited, because of mutual constrains of individual grains. This constrain can be considerably relaxed in the ferroelectric thin film, where only in-plane strain component need to be compatible. This suggests that ferroelectric thin film can be a promising candidate for the large strain actuator. In this paper, we study the reversible strains and polarization of polycrystalline ferroelectric thin film under the energy minimization, with the objective to identify the optimal microstructures. We first demonstrated that, the Taylor bound, a very good estimate of reversible strain in bulk polycrystalline ferroelectric, can be easily defeated in the thin film ferroelectric. We then use the translation method to derive the outer estimates on the reversible strain and polarization. The maximal actuation strain will be estimated, and the optimal texture of the thin film will be identified.

SESSION P3: POSTER SESSION
DISLOCATIONS AND DEFORMATION MECHANISMS IN THIN FILMS AND SMALL STRUCTURES

Chairs: Shefford P. Baker and Oliver Kraft
Tuesday Evening, April 17, 2001
8:00 PM
Salon 1-7 (Marriott)

P3.1

IN SITU ELASTIC DEFORMATION STUDY OF METALLIC THIN FILMS AND MULTILAYERS BY X-RAY DIFFRACTION. P.O. Renault, P. Villain, P. Goudeau, K.F. Badawi, Laboratory of Metallurgie Physique, Poitiers, FRANCE; E. Elkaim, J.P. Lauriat, LURE, Orsay, FRANCE.

Thin films deposited by PVD techniques on non epitaxial substrates often exhibit very small grain size which may induce particular mechanical behavior. These thin films are characterized by high residual stress state which can be generated by a large number of grain boundaries and high defect densities. Due to this particular microstructure, the elastic constants of the thin films such as Young's modulus and Poisson's ratio may differ from the bulk material ones. In the present study, in situ tensile testing are performed in an x-ray diffractometer to investigate both mechanical behavior and microstructure evolution of thin films: -the results on gold thin film (320 nm thickness) deposited on brass dogbone substrate are presented. Assuming a complete elastic strain transfer from the substrate to the film, Poisson's ratios related to different (hkl) planes have been obtained. Moreover the change in the breadth of the x-ray peaks is analyzed to determine if a plastic flow occurs in the thin film. -first results on W/Cu multilayers deposited on polyimide dogbone substrates will be given in this presentation. Poisson's ratio related to different modulation wavelength (with equal layer thickness W/Cu multilayers (6, 12 and 24 nm) and total thickness of around 250 nm) will be presented.

P3.2

THE EFFECT OF SOLID SOLUTION ALLOYING ON MECHANICAL BEHAVIOR OF Pt-Ru THIN FILMS. Seungmin Hyun, Richard P. Vinci, Lehigh University, MS&E Dept, Bethlehem, PA.

Strengthening mechanisms have been widely studied in pure metal films, but the effectiveness of solid solution strengthening has not been systematically explored. In this study, Pt-Ru solid solution thin films were investigated for use as electrodes integrated with ferroelectrics, and the mechanical properties of thin films with various experimental conditions were studied. The films were prepared by DC magnetron sputtering, and the composition of Ru was varied from 0 to 20 wt% by codeposition. The stress evolution of the Pt-Ru solid solution thin films was investigated by the wafer curvature technique, and the influence of solid solution alloying was observed as a function of Ru content. The effects of thickness of the film, ramping rates and atmospheres on stress relaxations of thin films were also explored. At room temperature, flow stress increased by approximately a factor of two, but high temperature behavior was relatively insensitive to Ru content. The experimental results of stress evolution and flow stresses of films were compared to diffusional models and flow stress calculations that are based on dislocation motion in thin films.

P3.3

ON THE GROWTH AND NANOINDENTATION BEHAVIOUR OF NITRIDE MULTILAYERS. Jon Molina-Aldareguia, Stephen Lloyd, Zoe Barber and Bill Clegg, Dept of Materials Science and Metallurgy, University of Cambridge, UNITED KINGDOM.

Previous work has indicated that multilayer films can be harder than monolithic ones and it has been suggested that this arises due to the effects of the multilayer structure on dislocation motion. To investigate these ideas, TiN/NbN multilayers with bilayer thicknesses ranging from 4 nm to 30 nm have been grown on MgO (001) single crystals using reactive magnetron sputtering. Optimum multilayer structures are produced under conditions where the intrinsic residual stresses in the film are small and slightly compressive. The sharpness of the interface and the intermixing between the layers, which would be expected to strongly influence dislocation motion, has been determined using x-ray diffraction (XRD) and energy-filtered transmission electron microscopy (EFTEM). These experiments showed that the interfaces remained reasonably sharp (interface thickness $\sim$ 1 nm) and no detectable intermixing occurred in multilayers with bilayer thicknesses greater than $\sim$ 10 nm. With thinner bilayers, some intermixing occurred but the layered structure remained. Despite this, the nanoindentation hardness of the multilayer films was between 20 and 25 GPa, which is similar to that for TiN and NbN alone. To understand why this occurs, the deformation mechanisms taking place under the indent in these materials is being studied using TEM.

P3.4

EFFECT OF THE INTERFACE ON HARDNESS OF Ti-TiN NANOLAMINATED STRUCTURES. Pascal Aubert, Mouloud Ben Daia, Catherine Sant, Sid Labdi, Philippe Houdy, Université d'Evry Val d'Essonne, Laboratoire Multicouches Nanométriques, Bat. des Sciences rue du Père Jarlan, Evry cedex, FRANCE.

Nanostructured materials have been attracting more interest regarding their mechanical properties. In this case, multilayers of metal and ceramic (Titanium and Titanium Nitride) has significant technological potential for hard coatings. The aim of this study is to correlate the mechanical (hardness and Young modulus) parameters with the structural properties. For multilayers, in particular, the hardness is strongly dependent on period and interface quality. For this study we have obtainded two different series of samples, with sharp and graded interfaces, and with a period thickness equal

20,10,8,5 and 2.5 nm The Ti-TiN Multilayers was deposited by RF reactive sputtering on silicon substrates with a metallic target. The deposition was done at room temperature for Ti-TiN multilayers The total thickness, period thickness and the nature of interface for all the films, were obtained by Grazing X-Ray Reflectometry. Tribological test are carried out by pin on disc tribometer and the hardness and Young modulus were measured by nanoindention with a Berkovich diamond indentor. Nanoindentation experiment was performed on all samples. The hardness was found to be dependent on period of thickness, type of interface and deposition rate. The experiment results are discussed in terms of dislocations based models, previously introduced in the literature.

P3.5

TEMPERATURE AND STRAIN-RATE DEPENDENCE OF DEFORMATION-INDUCED POINT DEFECT CLUSTER FORMATION IN METAL THIN FOILS. Kazufumi Yasunaga, Yoshitaka Matsukawa, Masao Komatsu, Michio Kiritani, Academic Frontier Research Center for Ultra-high-speed Plastic Deformation, Hiroshima Institute of Technology, Hiroshima, JAPAN.

We reported the formation of anomalously large numbers of point defect clusters were produced in fractured tips of ductile metal thin foil elongated. There was no indication of the generation and operation of dislocations in thin area, and this suggest the possibility of deformation without dislocation (Phil. Mag. Lett., 79 (1999) 797 - 804).

In order to understand the formation process of point defect clusters, systematic experiments were performed to observe the variation of the formation of point defect clusters with thermally activated motion of point defects during deformation. The first is the deformation speed dependence and the second is the variation with temperature. Materials examined were pure aluminum, gold and nickel.

Vacancy clusters in the form of the stacking-fault tetrahedron were produced in thin area in all the three materials examined. Saturation of the formation of vacancy clusters was observed at high deformation speed. Towards the other direction, the number density of vacancy clusters decreased with decreasing deformation speed. The deformation speed below which the formation of vacancy clusters decreases was found to corresponds to the condition at which vacancies can escape to specimen foil surfaces during the deformation. This indicates that the major part of defects is produced as point defects, and the production might not be strongly dependent on deformation speed.

However, the observed very slow variation of the number density of point defect clusters with deformation speed cannot be understood solely from the migration of point defects, and small point defect clusters formed directly by deformation should be accepted. The combination of the success of these small point defect clusters to grow to visible clusters during short deformation time and the annihilation during long deformation time can account for the observed result.

P3.6

MOLECULAR DYNAMICS SIMULATION OF ASPERITY SHEAR IN ALUMINUM. Jun Zhong, Arizona State University, Department of Chemical and Materials Engineering, Tempe, AZ; Hualiang Yu, Arizona State University, Science and Engineering of Materials Program, Tempe, AZ; James B. Adams, Arizona State University, Department of Chemical and Materials Engineering, Tempe, AZ.

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

P3.7

THE INVESTIGATIONS OF MECHANISMS OF DISLOCATION NUCLEATIONS AND DISLOCATION COMPLEXES IN THIN FILMS. Michail D. Starostenkov, Gennadiy M. Poletayev, Andrey A. Ovcharov, General Physics Dept, Altai State Technical Univ.

Structure-energetical transformations, taking place in thin films under external influences: deformation, ion implantation, thermoactivating processes are investigated by the method of molecular dynamics. It was discovered, that the stage of static waves of atom displacements proceeds to the stage of formation and movement of dislocations under deformations in ideal thin films. The formation of dislocations and dislocation loops takes place under ion implantation near the pairs of inculcated atoms in metal thin films. The dislocations of discrepancy at the boundaries of phases division form under thermoactivation in bimetal thin films. The formation of dislocations and their migration accompany the process of high temperature synthesis in the system Ni-Al in bimetal thin films. The mechanisms of formation of grain boundary dislocations in thin films of intermetallids are also investigated.

P3.8

SURFACE EFFECTS IN PLASTICITY AND FRACTURE OF THIN FILMS. Valery P. Kisel, Inst. of Solid State Physics, Chernogolovka, Moscow District, RUSSIA.

This work deals with the surface polishing effect, IE, on the mobility and multiplication of individual dislocations in model single crystals: pure/impure NaCl under successively applied stresses, s (s=0.9S to 95S, where S is the resolved yield stress) and stress rates (0.0001 to 1 000 000 MPa/sec) on dislocation mobility was investigated in nominally pure alkali halide crystals in the temperature range T =0.002 to 0.96) Tmelt, Tmelt is the melting point). Some findings have been revealed: 1. Damped motion of dislocations is due to the visible cross-slip of dislocations. 2. Having covered a finite pathlength determined by crystal and test parameters, the dislocations undergo retardation and multiplication. The stress, stress rate and temperature dependences of mean pathlengths before multiplication are similar to the appropriate macroscopic deformation-stress work-hardening curves. This result confirms the key role of dislocation cross slip in crystal work-hardening at various scale lengths of observation. 3.After polishing of the near-surface layer with the thickness h (5 microns $< h <$ 35 microns) the dislocations stopped by previous loadings begin to move/multiplicate till the next full stop. 4. The obtained results and the numerous literature data on main classes of solids are completely explained by the removal of strong obstacles-dislocation jog/microprecipitates for dislocation motion. Surface films and various treatments, environments prevent conservative motion of stress-aided jogs/kinks along the dislocations to and from the sample surface thus pumping the impurity phase/jogs out or into the crystals and changing the electrical, chemical, etc. surface-environment interactions, bulk hardening or softening. 5. Scaling of the minimal stresses required to start dislocation motion, multiplication, macroscopic flow, micro- and macrofracture proves the above mechanisms.

P3.9

DISLOCATION LOCKING BY INTRINSIC POINT DEFECTS IN SILICON. I.V. Peidous, Department of Physics, University of the West Indies, Kingston, JAMAICA; K.V. Loiko, Dallas Semiconductor, Dallas, TX; W.R. Frensley, Department of Electrical Engineering, University of Texas at Dallas, Richardson, TX.

Dislocation locking phenomenon in silicon was clearly demonstrated in the 70’s to be dependent on oxygen concentration. An important role of Si-interstitials in defect evolution was revealed later. Nevertheless, most observations of dislocation locking in silicon are traditionally attributed to oxygen. Self-interstitial atoms are centers of dilatation in silicon crystal. Therefore, they must interact elastically with dislocations causing more or less significant dislocation pinning. In the present work, an estimate of the dislocation locking efficiency by self-interstitials is made based on analytical calculations and computer modeling. In addition to pinning, dislocation screening by point defects is identified as a major contributor to dislocation locking.

P3.10

Abstract Withdrawn.

P3.11

OPTICAL STUDY OF SIGE FILMS GROWN ON LOW-TEMPERATURE Si BUFFER. Y.H. Luo, J. Wan, J.L. Liu, K.L. Wang, Device Research Laboratory, University of California at Los Angeles, Los Angeles, CA.

High quality relaxed SiGe films attracted a lot of interests due to the applications for strain Si/SiGe high electron mobility transistor (HEMT), metal-oxide-semiconductor field effect transistor (MOSFET), heterojunction bipolar transistor (HBT) and other devices. In this work, the SiGe films with low temperature Si buffer layers were grown by solid-source molecular beam epitaxy. Photoluminescence and Raman spectroscopy were used to study the properties of the dislocations and the disorder effect caused by dislocations. With proper growth temperature and thickness of the low temperature Si buffer, SiGe films with low dislocation density were obtained. The tensile strain in the low-temperature Si buffer, which was believed to decrease the mismatch between the SiGe layer and the Si layer and then improve the quality of the SiGe layer, was observed. High quality relaxed SiGe layers with different Ge contents have been obtained with this method. The thermal stability of the SiGe films was also studied by measuring samples annealed at high temperatures.

P3.12

THICKNESS-FRINGE CONTRAST ANALYSIS OF DEFECTS IN

GaN. J.K. Farrer, C.B. Carter, University of Minnesota, Dept. of Chemical Engineering and Material Science.

The analysis of thickness-fringe contrast in TEM images has been shown to be a reliable method for the complete determination of the character, as well as the magnitude, of the Burgers vector of a dislocation in cubic materials. This technique uses weak-beam imaging in the TEM for Burgers vector analysis of dislocations in a wedge-shaped TEM sample. The wedge shape of the material results in the appearance of thickness fringes. The complete determination of the magnitude and direction of the dislocation is accomplished by counting the number of extra half-line equal-thickness fringes, Δn, observed around a dislocation outcrop. The number of fringes is then applied to the relation $\Delta n = \mathbf{g} \bullet \mathbf{b}$ where $\mathbf{g}$ represents the diffracting vector. By selecting two or three diffracting conditions, $\mathbf{g}$, and determining Δn for each condition, $\mathbf{b}$ can be unambiguously determined. One advantage of this technique is that analysis of the Burgers vector is not dependent upon the contrast of the dislocation or on the need to recognize the condition $\mathbf{g} \bullet \mathbf{b}=0$. TEM studies have revealed that there are three principal types of dislocations in GaN epilayers grown by conventional means. The majority of the dislocations are reported to be threading dislocations with Burgers vectors equal to $\frac{1}{3} < 11\bar{2}0 >$. The other types of dislocations observed are dislocation half loops with Burgers vectors of [0001] or $\frac{1}{3} < 11\bar{2}0 >$. In the case of epitactic lateral overgrowth of GaN, the threading dislocations are observed to "bend" in such a manner that their line direction becomes parallel to the substrate/epilayer interface. Burgers vector analysis of the dislocations in GaN grown on (111) Si substrates by selective overgrowth have been studied using the thickness-fringe contrast technique. It is shown that this technique, formerly applied to cubic materials, is equally reliable in hexagonal materials.

P3.13
DISLOCATION MOTION IN SiGe THIN FILMS STUDIED USING LOW-ENERGY ELECTRON MICROSCOPY. A.R. Woll, P. Moran, E.M. Rehder, T.F. Keuch and M.G. Lagally, University of Wisconsin-Madison, Madison, WI.

The growing technological importance of strain and strain relaxation in semiconductor thin films demonstrates the increasing need for fundamental understanding of strain relaxation mechanisms, such as dislocation creation and motion. Yet, very few tools, other than transmission electron microscopy, are suitable for studying the motion and dynamics of single dislocations. Low-Energy Electron Microscopy (LEEM) is a powerful tool for examining the dynamic evolution of crystalline surface morphology with atomic-height resolution. Changes in surface height, for example that caused by vertical glide, are readily observed. In addition, for the case of Si(001), the anisotropic (2×1) surface reconstruction provides a built-in monitor of uniaxial stress, such as that caused by a dislocation. Here we discuss the use of LEEM to elucidate dislocation motion in $Si_{1-x}Ge_x$/Si(001) thin films. In particular, we compare SiGe thin films grown on bulk Si, SOI, and boro silicate glass SOI substrates. We have found that the presence of the buried oxide in SOI accelerates plastic relaxation. LEEM results will be discussed in the context of theoretical calculations suggesting that the main function of the oxide is to reduce dislocation pinning.

P3.14
MICROSTRUCTURE OF InAs/GaAs QUANTUM DOTS STUDIED BY TRANSMISSION ELECTRON MICROSCOPY. Yongqian Wang and Zhonglin Wang, School of Materials and Engineering, Jeng-Jung Shen and April S. Brown, School of Electrical and Computer Engineering, Georgia Institute of Technology, Atlanta, GA.

In this presentation, we will report our major TEM observations on InAs/GaAs quantum dots grown by molecular beam epitaxy. In a novel approach to modifying dot properties, the quantum dots are annealed at a range of temperatures under a dissimilar anion flux, in this case P2. Prior to anneal, it is shown that the InAs islands are mainly lens-shaped and most of them are vertically self-aligned. Two-beam imaging indicates that strong strain fields exist around the dots and overlap for different layers. Although the self-aligned dots are mostly coherent to GaAs (100), relaxation by misfit dislocations is occasionally found. Much more complicated relaxation is observed in larger islands. Due to the strong interaction between the dots in adjacent layers, the nature of the relaxation is difficult to identify. Stacking faults in a V-shape are frequently formed in the lateral sides of these regions. P2-annealing at 300°C causes no obvious change in island morphology. However, remarkable changes take place upon annealing at 350°C. The vertically-aligned islands disappear, smooth interfaces were obtained. These morphological transition is believed to result from the replacement of arsenic by phosphorus in the InAs dots. This anion exchange is driven by minimization of strain energy and chemical Gibbs free energy. Although the vertically aligned islands are annealed out due to anion exchange, they left behind no trace of their previous existence. The opposite effect is observed in the relaxed regions. The position of these regions was unchanged, and the crytallinity in these regions was low. Based on the contrast change in the two beam imaging conditions, considerable intermixing is considered to have occurred during growth. Thus, temperature dependent exchange drives dramatic changes in the structural properties of quantum dots. This offers another possibility for tuning the dot morphology.

SESSION P4: DISLOCATIONS IN SMALL STRUCTURES

Chair: L. B. Freund
Wednesday Morning, April 18, 2001
Salon 10/11 (Marriott)

8:30 AM *P4.1
THREE-DIMENSIONAL SIMULATION OF MISFIT DISLOCATIONS IN QUANTUM DOTS. L.H. Friedman, D.M. Weygand and E. van der Giessen, Netherlands Institute for Metals Research, Delft University of Technology, Delft, NETHERLANDS.

Stranski-Krastanow growth is often used to produce large ordered arrays of quantum dots. In this type of growth, atoms deposited on a substrate spontaneously aggregate into growth islands due to surface energy and elastic effects. For these islands to be usable as quantum dots, it is important that they be dislocation free. However, misfit dislocations can form if large enough stresses exist due to the lattice mismatch between the island and substrate. The question of when misfit dislocations form in growth islands is addressed here in a manner similar to that in a 2D approximation by Johnson and Freund (J. Appl. Phys., 81(9), 1997, p. 6081). The 3D approach presented here uses a generic methodology for 3D discrete dislocations in a finite volume. This method combines analytic results and finite elements to obtain elastic fields that satisfy appropriate boundary conditions. First, the individual dislocations are discretized and the correspoding stress, strain and displacement fields in an infinite solid are obtained analytically. Then, the finite element method is used to correct for the boundary conditions of the growth island and substrate geometry and to add the lattice misfit strain. This procedure accounts for the dislocation-surface interactions, so-called image forces, in the 3D configuration. Results of the simulation are presented with a discussion of the differences and similarities between the 2D model of Johnson and Freund and the 3D simulation. Finally, how well this method can be applied to answering other dislocation-related questions in small confined volumes is discussed.

9:00 AM *P4.2
MODELING OF DISLOCATIONS IN AN EPITAXIAL ISLAND STRUCTURE. Xiao Hu Liu, Frances M. Ross, and Klaus W. Schwarz, IBM Watson Research Center, Yorktown Heights, NY.

The behavior of dislocations has historically been studied with an emphasis on the plastic properties of bulk materials. More recently, dislocations have become a topic of interest in the manufacture of semiconductor films and devices, where their appearance can cause serious performance problems. In such a situation, the task is to understand the behavior of just a few dislocations in a highly confined, geometrically complicated environment. As an example of such a problem, we examine the growth of dislocations in certain island structures which form spontaneously during epitaxial growth. Such dislocated islands provide an ideal environment in which to study the behavior of dislocations in small structures. In this talk we present our modeling of dislocations in $CoSi_2$ islands grown by reactive epitaxy on (111) Si substrate. The stress fields due to the lattice mismatch are calculated using standard FEM techniques, and are converted into a structured, multi-level and multi-grid stress table that is imported into the PARANOID code to study the dislocation dynamics. Single and multiple dislocations in the island have been simulated, and the predicted patterns are strikingly similar to those observed experimentally. By looking at the growth behavior of very small loops we also find that dislocation-loop nucleation becomes easier as the islands become larger, and that thick islands are dislocated at smaller size than thin ones. These results are also in good agreement with experimental observations. We conclude that current modeling techniques are sufficient to treat this type of problem at a useful level of accuracy.

9:30 AM P4.3
MISFIT DISLOCATION INTRODUCTION DURING THE EPITAXIAL GROWTH OF InAs ISLANDS ON GaP. Vidyut Gopal, Applied Materials Inc., Santa Clara, CA; Alexander L. Vasiliev, The Institute of Materials Science, University of Connecticut, Storrs, CT; Eric P. Kvam, School of Materials Engineering, W. Lafayette, IN.

The initial growth of InAs on 11% lattice mismatched GaP substrates by molecular beam epitaxy was investigated. High resolution transmission electron microscopy (HREM) images showed that the InAs grew in the form of three-dimensional islands of dissimilar sizes. Mismatch induced strain relief was effected by the direct introduction of (mostly) edge dislocations at the corners of the islands. An examination of HREM images of several islands revealed that the island aspect ratio decreased with the introduction of misfit dislocations. Strain relaxation in the smaller, relatively dislocation-free islands occurred by elastic deformation of InAs lattice planes, which was more effective far from the constrained island-substrate interface. As a result, these islands grew taller and narrower, with a gradient in the elastic strain energy. However, a higher aspect ratio resulted in a higher surface area - to - volume ratio, and increased the surface energy of the InAs islands. Consequently, there was a driving force for the reduction of the aspect ratio if an alternate avenue for strain relaxation existed. The alternate route was plastic deformation by the introduction of misfit dislocations. As the island grew, the strain at the island corners increased, and beyond a critical value, misfit dislocations were added. These dislocations relieved strain at the heterointerface, and promoted the islands to grow laterally, i.e., the aspect ratio decreased. Islands coalesced, and a continuous layer resulted by a nominal thickness of 3 nm. Thus, the morphology of InAs islands grown on GaP was determined by the balance between elastic and plastic deformation.

9:45 AM P4.4

INTERPLAY OF DISLOCATION NETWORK AND ISLAND FORMATION IN SiGe FILMS ON Si(001). C. Teichert, C. Hofer, Dept of Physics, University of Leoben, Leoben, AUSTRIA; K. Lyutovich, M. Bauer, E. Kasper, Institut für Halbleitertechnik, University of Stuttgart, Stuttgart, GERMANY.

In epitaxial growth of SiGe films on vicinal Si(001), the growth front undergoes a series of strain-relief mechanisms. These include elastic strain-relief by step bunching [1] and subsequent formation of {105} faceted crystallites [2]. Finally, the film relaxes by the introduction of misfit dislocations, that can form a network appearing as a cross-hatch pattern at the surface. SiGe films were grown in a two-temperature process [3], starting with low temperature (LT) deposition at 100°C - 250°C followed by growth at 550°C. Using atomic-force microscopy we show that the misfit dislocation network introduced after the LT stage guides the arrangement of {105} faceted, pyramid-like crystallites. The dislocation density - and therefore the uniformity of the crystallite array - can be controlled by the LT value. In the case of a very narrow dislocation network a checkerboard array of 105 faceted pits and pyramids evolves with a "lattice constant" of about 200 nm [4]. [1] Y.H. Phang, et al., Phys. Rev. B50, 14435 (1994). [2] Y.-W. Mo, et al., Phys. Rev. Lett. 65, 1020 (1990). [3] E. Kasper et al., Thin Solid Films 336, 319 (1998). [4] C. Teichert, et al., Thin Solid Films, in press.

10:30 AM P4.5

MODELING PLASTIC DEFORMATION IN CONFINED MEDIA. C. Lemarchand, B. Devincre and L. Kubin, Laboratoire d'Etude des Microstructures, CNRS-ONERA, Chatillon Cedex, FRANCE.

A new non-local model combining the discrete and continuum approaches of plastic deformation has been developed the past two years. This model, the "Discrete-Continuum Model" (DCM), aims at investigating the plasticity of complex materials containing structural heterogeneities and/or under complex loadings. It is based on the coupling of a Finite Element code and a Dislocation Dynamics code. The first calculations, aim at better understanding the microscopic processes governing plastic deformation in confined media and in the presence of high interfacial stresses (misfit stresses, elastic incompatibility stresses and image stresses). New results on the modeling of internal stress relaxation in thin films will be presented.

10:45 AM P4.6

DISLOCATION DYNAMICS NEAR THE LOCOS STRUCTURE IN SILICON. D. Chidambarrao, IBM Semiconductor R&D Center, Hopewell Junction, NY; X.H. Liu and K.W. Schwarz, IBM Thomas J. Watson Research Center, Yorktown Heights, NY.

Because dislocations can cause major charge-leakage problems in silicon devices, it is important to understand how they nucleate and propagate in the manufacturing environment. The recent development of dislocation-simulation programs has led to quantitative predictions of dislocation behavior in simple structures such as heteroepitaxial layers and film edges or corners. The work presented here moves to the next level of complexity and utility by combining process modeling with dislocation simulation to investigate dislocation behavior during the growth of a Local Oxidation Structure (LOCOS). The aim is to quantitatively predict not only the "bird's beak" geometry which arises from nonplanar boundary motion during oxide growth, but also the final configuration of the dislocations which are generated during this process. We show that both the observed oxidation structure and the observed dislocation patterns can be predicted to an accuracy of 10-20% using such an integrated approach. Of particular interest is our observation that while the diverging stresses near the bird's beak nucleate the dislocations, the final dislocation positions depend largely on long-range fields, such as thermal mismatch stresses, which are usually ignored.

11:00 AM P4.7

ATOMISTIC SIMULATIONS OF SIZE SCALE EFFECTS ON THE PLASTICITY OF METALS. M.I. Baskes, Structure Property Relations Group, Los Alamos National Laboratory, Los Alamos, NM; M.F. Horstemeyer, Center for Materials and Engineering Sciences, Sandia National Laboratories, Livermore, CA; S.J. Plimpton, Center for Computation, Computers & Math, Sandia National Laboratories, Albuquerque, NM.

We examine the effects of sample size on single crystal FCC metals. We perform simple shear and torsion molecular dynamics simulations using the Embedded Atom Method (EAM) on single crystal nickel ranging from 100 atoms to 100 million atoms to study yield and work hardening. Comparison of the results of the simple shear and torsion calculations are made and related to strain gradient plasticity concepts. The results of the atomistic simulations are compared to data from nano-indentation experiments, micro-indentation experiments, and small scale torsion experiments. This comparison shows that yield varies as $L^{-0.4}$ over five orders of magnitude in which a length scale parameter, L, is defined by the ratio of volume to surface area. The atomistic simulations reveal that dislocations nucleating at free surfaces are critical to causing microyield and macroyield in pristine material. The atomistic simulations and experimental data show that differences in applied strain rate, temperature, stacking faults (several FCC materials have been examined), deformation mode, and crystal orientation on yield and plasticity are small compared to the size scale effect.

11:15 AM P4.8

THE INFLUENCE OF A THIN FILM GEOMETRY ON PLASTIC DEFORMATION MECHANISMS IN NANOCRYSTALLINE Ni. P.M. Derlet and H. Van Swygenhoven Paul Scherrer Institute, Villigen-PSI, SWITZERLAND.

Previous work on nanocrystalline Ni under uni-axial tensile conditions, has demonstrated that the presence of a surface can lead to increased grain boundary sliding and, with increasing grain size, the creation and emission of partial dislocations. In the present work, parallel Molecular Dynamics within the Parrinello-Rahman framework is used to further investigate the influence of two parallel surfaces on the plastic deformation mechanisms in thin film nanocrystalline Ni. The resulting grain boundary microstructure under deformation will be elucidated, allowing a quantitative estimation of the separate contribution to strain by grain boundary sliding and partial dislocations emitted due to the surface. The motivation for this simulation is to understand which phenomena observed in in-situ tensile experiments performed in the electron microscope can be expected to be intrinsic properties of the deformation process and which phenomena are due to an effective thin film geometry.

11:30 AM P4.9

X-RAY SCATTERING FROM MISFIT DISLOCATIONS AT BURIED INTERFACES. Kaile Li, Paul F. Miceli, University of Missouri-Columbia, Department of Physics and Astronomy, Columbia, MO; Karen L. Kavanagh, Simon Fraser University, Department of Physics, Burnaby, BC, CANADA.

Motivated by x-ray scattering experiments on heteroepitaxially grown thin films, we present model calculations of the diffuse x-ray scattering arising from misfit dislocations. The model is based on elastic calculations of displacements arising from various types of defects whose positions are spatially uncorrelated. These numerical results give support to a phenomenological model [Phys. Rev. B **51**, 5506 (1995)] that predicts the scaling of the diffuse scattering intensity with the perpendicular momentum transfer, Q. At low Q, the diffuse width scales inversely with the defect size, which is given by the film thickness due to the effect of the elastic image field, whereas at high Q, the diffuse width is mosaic-like, scaling with Q. New experimental results for $In_xGa_{1-x}As/GaAs$ will be presented and compared to the model. The calculations are in good agreement with these experiments, as well as other measurements in the literature, for very high or very low dislocation densities.

Support is gratefully acknowledged from the National Science Foundation (NSF) under contract DMR-9623827 and the Midwest Superconductivity Consortium (MISCON) under DOE grant DE-FG02-90ER45427.

11:45 AM P4.10

SMALLER IS SOFTER: A DISCRETE DISLOCATION ANALYSIS OF AN INVERSE SIZE EFFECT IN A CAST ALUMINUM ALLOY. A.A. Benzerga[a], S.S. Hong[a], E. Van der Giessen[b], A. Needleman[a] and K.S. Kim[a]. [a]Brown University, Division of Engineering, Providence, RI; [b]Delft University of Technology, Koiter Institute Delft, THE NETHERLANDS.

Tensile tests were carried out on specimens made of an A356 cast aluminum alloy with 7% Si as the main alloying element. The specimens were cast at two cooling rates. For both processing conditions the microstructure within each grain consists of pro-eutectic aluminum dendrites separated by a boundary eutectic region of segregated silicon particles of $\approx 2-3$ μm diameter. The fast cooling rate gives rise to a secondary dendrite arm spacing of approximately 23 μm, while the secondary dendrite arm spacing obtained with the slow cooling rate is about 90 μm. The stress-strain curves exhibit an unusual size effect on the flow properties; the fine microstructure has a softer plastic response than the coarse microstructure. This inverse size effect is investigated using a simple discrete dislocation plasticity model. The dislocations are represented as line defects in an elastic solid. Dislocation nucleation, annihilation and drag during dislocation motion are incorporated through a set of constitutive rules. Obstacles to dislocation motion are randomly distributed in the dendrite and the eutectic regions. Obstacle density and strength distributions are independently assigned to each region. The thickness of the eutectic region is found to be a key parameter in determining the inverse size effect on hardening. In addition, the size effect is found to depend on the extent to which dislocation nucleation takes place in the eutectic region.

SESSION P5: DISLOCATIONS AND DEFORMATION IN EPITAXIAL LAYERS

Chairs: Robert Hull and Dureseti Chidambarrao
Wednesday Afternoon, April 18, 2001
Salon 10/11 (Marriott)

1:30 PM *P5.1
FRACTURE AND THE INTRODUCTION OF MISFIT DISLOCATION ARRAYS DURING AlGaN/GaN HETEROEPITAXIAL GROWTH. Jerrold A. Floro, David M. Follstaedt, Paula P. Provencio, Karen E. Waldrip, Sean J. Hearne, and Jung Han, Sandia National Laboratories, Albuquerque, NM.

III-nitride materials are the subject of intense study due to their novel optoelectronic properties, but our understanding of the fundamental mechanical behavior lags far behind the technology. Hexagonal AlGaN alloys grow heteroepitaxially on GaN in a state of biaxial tensile stress due to the lattice mismatch. Above some critical thickness, the AlGaN layer undergoes a combined relaxation mode in which a low density channel crack array is introduced initially, followed by the formation of a dense but well-ordered misfit dislocation network within the epitaxial interface. The competition between fracture and dislocation introduction appears to be determined by limitations on defect nucleation. Cracks apparently are able to nucleate first, at asperities in the AlGaN. Once cracks do form, they then foster limited nucleation of misfit dislocations that rapidly glide large distances from the cracks. Subsequently, multiplication processes take over that produce pure interfacial misfit segments which complete the relaxation process. I will discuss the many challenges and opportunities in this research area with regard to understanding the specific slip systems, cross-slip, the role of climb, dissociation into partials, dislocation reactions, the detailed mechanisms for multiplication, and ultra-rapid relaxation processes. 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.

2:00 PM P5.2
DEVELOPMENT OF CROSS-HATCH MORPHOLOGY DURING GROWTH OF LATTICE MISMATCHED LAYERS.
A. Maxwell Andrews, James S. Speck, Materials Department, University of California, Santa Barbara, CA; A.E. Romanov, Ioffe Institute, St. Petersburg, RUSSIA; M. Bobeth, W. Pompe, Technical University of Dresden, GERMANY.

An approach for understanding the cross-hatch morphology in lattice mismatched heteroepitaxial film growth is developed. It is argued that both strain relaxation associated with misfit dislocation formation and lateral surface step flow are required for the appearance of mesoscopic scale surface undulations during layer growth. That is, cross-hatch is a consequence of local smoothing, via step elimination, which results in mesoscale roughness. The results of Monte Carlo simulations for dislocation assisted strain relaxation and consequent film growth predict the development of cross-hatch patterns with a characteristic surface undulation magnitude $\sim$50 Å in an approx. 70% relaxed $In_{0.25}Ga_{0.75}As$ layers. This is supported by atomic force microscopy (AFM) observations of cross-hatch morphology in the same composition samples grown well beyond the critical thickness for misfit dislocation formation.

2:15 PM P5.3
MECHANISM FOR THE REDUCTION OF THREADING DISLOCATION DENSITIES in $Si_{0.82}Ge_{0.18}$ FILMS DEPOSITED ON SILICON ON INSULATOR SUBSTRATES. E.M. Rehder[a], T.S. Kuan[b], and T.F. Kuech[a]; [a]Materials Science Program, University of Wisconsin-Madison; [b]University at Albany, State University of New York.

We are studying the relaxation behavior of $Si_{0.82}Ge_{0.18}$ films directly deposited on silicon on insulator (SOI) substrates. Deposition is carried out in an ultrahigh vacuum chemical vapor deposition system at 550°C. The growth system is equipped with in situ monitors for reflectivity corrected pyrometry and sample curvature. The reflectivity correction greatly improves temperature measurement and control, while the sample curvature provides film strain information in real-time. Following growth, films are characterized by atomic force microscopy, X-ray diffraction, and transmission electron microscopy. The $Si_{0.82}Ge_{0.18}$ films have a greatly reduced threading dislocation density compared to films deposited on Si substrates, while the threading dislocation density is increased in the SOI substrate. We propose a mechanism to explain the change in dislocation structure on the SOI substrate. The buried amorphous oxide layer of the SOI substrate performs two important functions in producing these results. It provides an image force attracting dislocations in the film toward the oxide and upon reaching the amorphous oxide the misfit dislocation segments annihilate. These factors modify the mechanism on bulk Si by which perpendicular intersecting dislocations cross each other. During an intersection, the existing dislocation moves into the substrate. Repeated dislocation intersections lead to a dislocation pile-up in the substrate, preventing passage of additional dislocations. On the SOI substrate, the annihilation of the misfit dislocations allows intersections to continue without pinning. The result is nucleation of fewer dislocations leading to fewer threading dislocations.

2:30 PM P5.4
RELAXATION OF $Si_{1-x}Ge_x$ BUFFER LAYERS ON Si(100) THROUGH HELIUM IMPLANTATION. M. Luysberg, D. Kirch, H. Trinkaus Institut für Festkörperforschung, Forschungszentrum Jülich, GERMANY; B. Holländer, St. Lenk, S. Mantl, Institut für Schicht- und Ionentechnik, Forschungszentrum Jülich, GERMANY; H.-J. Herzog, T. Hackbarth, Daimler-Chrysler AG, Research and Technology, Ulm, GERMANY; P.F.P. Fichtner Dept. de Metalurgia, Univ. Fed. Do Rio Grande do Sul, Porto Alegre, BRAZIL.

Strain relaxed $Si_{1-x}Ge_x$ layers on Si(100) are of great importance as virtual substrates for the fabrication of $Si_{1-x}Ge_x$/Si heterostructure devices. Due to the large lattice mismatch between $Si_{1-x}Ge_x$ and Si, relaxation requires the formation of misfit dislocations, often accompanied by detrimental threading dislocations. The goal of many different approaches of $Si_{1-x}Ge_x$ buffer layer fabrication is to achieve relaxed layers free of threading dislocations (TD). One promising technique to promote strain relaxation and to reduce the TD density is the implantation of He^+ into pseudomorphic layers followed by thermal annealing. In our paper we investigate the structural properties of He implanted $Si_{1-x}Ge_x$ layers on Si by transmission electron microscopy, X-ray diffraction and Rutherford-Backscattering. In particular, the dislocation types- and densities and the arrangement of He bubbles created by the implantation are studied. Proper choice of the implantation energy results in a narrow defect band just underneath the substrate/epilayer interface. During annealing at 850°C to 1000°C, He-filled bubbles are created, which act as dislocation sources for misfit dislocation generation. Complete annihilation of the threading dislocations is theoretically predicted, if a certain bubble density with respect to the buffer layer thickness is maintained. The variation of the implantation dose and the annealing conditions changes the density and size of He bubbles resulting in characteristic differences of the dislocation structure. Nearly complete strain relaxation of $Si_{1-x}Ge_x$ layers with Ge fractions up to 30 at.% is obtained at temperatures as low as 850°C and the samples appear free of threading dislocations within the SiGe layer to the limit of transmission cross-sectional electron microscopy. Very first n-type modulation doped FET structures grown on these relaxed SiGe buffer layers showed promising electrical properties.

2:45 PM P5.5
MISFIT ACCOMMODATION IN III-V SEMICONDUCTOR EPITAXIAL LAYERS. André Rocher, CEMES-CNRS, Toulouse, FRANCE.

In epitaxy, the relaxation of lattice mismatch is obtained classically by introducing at the interface a misfit dislocation network in order to accommodate the atomic structure between the substrate and the epitaxial layer without stress. TEM observations using moiré fringes on plan view samples have been performed in order to evaluate the strain field of both relaxed and strained structures. The moiré fringes obtained with the reflections normal and inclined to the interface permit us to evaluate the uniformity and the amplitude of the strain

fields of thin epitaxial layers with TEM resolution. The GaSb/(001)GaAs systems, grown under optimal conditions, have been studied by this technique. The measured moiré fringe spacings correspond well with the theoretical values, thus indicating a complete accommodation of the 8% lattice mismatch. This relaxation is obtained by a square grid of Lomer dislocations, perfectly organised. In ternary compounds, such as GaInAs/GaAs, the dislocation mechanism is not the first to occur in the misfit relaxation. Investigated specimens are 10 nm thick films of GaInAs with 25% of In grown on (111)B GaAs. The absence of both misfit dislocations and moiré fringes for reflections normal and inclined to the interface shows that the real strained state of the epitaxial layer is different from that predicted by the elastic theory. The misfit stress due to 1% lattice mismatch is high enough to induce mechanisms different from the ideal 2D growth: an indium segregation creates a transient composition with a random distribution of the III elements which would avoid directly the misfit stress. This transition zone, between the substrate and the uniform epilayer, should be equivalent to a set of non localised dislocation cores for misfit accommodation.
Acknowledgements: Thanks to Chantal Fontaine and Stéphanie Blanc (LAAS-CNRS, Toulouse) for providing GaInAs/GaAs specimens and fruitful discussions.

3:30 PM P5.6

INFLUENCE OF MISFIT FORMATION ON THE SURFACE MORPHOLOGY OF SiGe EPITAXIAL LAYERS GROWN ON UNIQUELY ORIENTED Si SUBSTRATES.

M.E. Ware, R.J. Nemanich, Physics Dept, North Carolina State Univ, Raleigh, NC; R. Hull, J.L. Gray Dept of MS&E, Univ of Virginia, Charlottesville, VA.

The growth of epitaxial SiGe alloys on uniquely oriented Si substrates is inherently strained due to the larger lattice spacing of the alloy. This strain and its subsequent relaxation manifests itself by affecting the structure of the layer. For low Ge concentrations, $\sim$30% or less, an epitaxial layer will begin to relax through the development of surface corrugations. As the layer is grown thick misfit dislocations form relaxing the film completely. We have investigated the effects of misfit formation on the surface morphology of Si(0.7)Ge(0.3) layers grown on uniquely oriented Si substrates with surface normals rotated from [001] towards [111] by up to 22 degrees. For all substrates the misfit formation has a similar effect. The existing surface structures become ordered along the lines of the misfits. We have imaged the surfaces with atomic force microscopy. For thin layers, tens of nanometers, images show corrugation with features on the order of 100nm wide having no long-range order. For thick layers, 100 to 200nm, images show the corrugation aligning over large ranges, greater than tens of micrometers. The corrugations align along the [-110] direction on all surfaces, and along two other directions that intersect each other at smaller angles as the surface normal of the substrate approaches [001]. The two latter directions are degenerate on the (001) surface becoming the [110] direction. The lines of alignment correspond closely with the lines of intersection of the (111) slip planes with each of the surfaces. Transmission electron microscopy has confirmed that misfit dislocations have formed in the layer aligned to the long-range surface corrugations.

3:45 PM P5.7

A QUANTITATIVE COMPARISON OF DISLOCATION MEDIATED STRAIN RELAXATION IN Si/SiGe MULTILAYER FILMS PRODUCED BY MBE, APCVD, LPCVD, AND UHVCVD.

Daniel B. Aubertine, Marc A. Mander, Ann Marshall, Paul C. McIntyre, Stanford Univ, Dept of MS&E, Stanford, CA; Glen Wilk, Lucent Bell Laboratories, Murray Hill, NJ; Pat Mooney and Jack Chu, IBM T.J. Watson Research Center, Yorktown Heights, NY.

We present the results of experiments measuring the contribution of misfit dislocation density to strain relaxation in epitaxial SiGe/Si multilayers grown on Si substrates by various thin film growth techniques. The techniques investigated include molecular beam epitaxy, ultra high vacuum chemical vapor deposition, low-pressure chemical vapor deposition, and atmospheric-pressure chemical vapor deposition. Multilayers prepared with these techniques exhibit a range of as-grown dislocation densities and varying degrees of interface abruptness. Film composition is measured with backscattering spectroscopy, while the epitaxial quality and substitutionality are checked with minimum yield channeling results. Measurements of absolute strain in the multilayers are made with x-ray diffraction by determining the position of the (004) multilayer peaks relative to the (004) Si substrate peaks. During these x-ray experiments, interface abruptness is observed by measuring the intensity of higher order multilayer satellite peaks. As grown and post-annealed misfit dislocation densities are investigated with plan-view transmission electron microscopy. The nature of the misfit dislocations and the degree of dislocation interaction are further assessed using two-beam diffraction contrast and high-resolution transmission electron microscopy of plan-view and cross-section samples. After characterization in the as-grown state, a set of annealing experiments is performed on each film. In this manner a systematic comparison of dislocation density and absolute misfit strain is made between films produced by the investigated growth techniques. Ongoing efforts to artificially introduce dislocation arrays in a controlled fashion via nanoindentation techniques will also be presented.

4:00 PM P5.8

RHEED INTENSITY OSCILLATIONS AND STRAIN RELAXATION DURING InN HETEROEPITAXY ON GaN.

Yee Fai Ng, Xiaoliang Wang, Maohai Xie, David Shuk Yin Tong, The Univ of Hong Kong, Dept of Physics, Hong Kong, PR CHINA.

Reflection high-energy electron diffraction (RHEED) specular beam intensity oscillations are observed during InN heteroepitaxy on GaN, by molecular-beam epitaxy (MBE) over a range of In fluxes at low substrate temperatures. Strong and sustained oscillations lasting for more than ten periods suggest a predominant two-dimensional (2D) growth mode. By measuring the time evolution of the spacing between the neighboring integral diffraction streaks, strain relaxation during growth is monitored. It is found that a fully-strained InN layer persists for about a half bilayer (BL), while completely relaxed films are achieved after more than three BLs' deposition. The RHEED oscillation data and the strain relaxation profiles imply that the system undergoes a process where dislocations are introduced prior to islanding during relaxation. The extent of relaxation is found to depend strongly on the substrate temperature. The full-width-at-half-maximum (FWHM) intensity of the specular beam is also measured as a function of InN thickness, and the beam shows a gradual broadening until it reaches a steady size. This suggests a change in surface morphology which roughens as InN growth proceeds.

4:15 PM P5.9

TEMPERATURE-DEPENDENT MODEL OF STRAIN RELAXATION IN EPITAXIAL FILMS IN THE PRESENCE AND ABSENCE OF THREADING DISLOCATIONS.

Mahadevan Khantha, Univ of Pennsylvania, Dept of MS&E, Philadelphia, PA.

Recent experiments on Si-Ge heteroepitaxy have demonstrated that temperature and residual stress are two dominant parameters that control the nucleation of misfit dislocations in both planar and non-planar epitaxial films. The nucleation is also affected by the presence of threading dislocations in the substrate. A cooperative process of misfit dislocation nucleation is proposed which predicts a temperature-dependent critical thickness for a planar film of fixed composition. This is a thermally-driven stress-assisted mechanism in which the nucleation of many atomic-size (subcritical) dislocation loops becomes energetically favorable as temperature increases due to screened dislocation interactions. The screening arises from the plastic strain associated with the atomic-size dislocation loops which helps to lower the "effective modulus" of the film and the screening is amplified as temperature increases. The motion of threading dislocations influences the onset of cooperative nucleation by enhancing the screening effect. The critical thickness is lower in the presence of threading dislocations but remains strongly temperature-dependent. The predictions of the model are compared with observations.

4:30 PM P5.10

THEORETICAL APPROACH TO STRAIN RELAXATION ENGINEERING IN LAYER-BY-LAYER HETEROEPITAXIAL GROWTH OF SEMICONDUCTOR THIN FILMS.

Dimitrios Maroudas and Luis A. Zepeda-Ruiz, Department of Chemical Engineering, University of California, Santa Barbara, CA.

Heteroepitaxial growth of semiconductor thin films on semiconductor substrates is used widely in the fabrication of optoelectronic devices, the function of which depends crucially on the defect levels in the epitaxial films. Optimizing the structural quality of the epitaxial layer requires development of substrate engineering and film engineering strategies for relaxation of strain due to lattice mismatch. Theoretically, this can be accomplished by optimization of a quantitative measure of strain relaxation with respect to the appropriate operating parameters for both the substrate and the epitaxial film. In this presentation, the use of compliant substrates and the grading of the epitaxial film composition are the engineering strategies under consideration in the case of layer-by-layer growth. A systematic theoretical framework is presented that can be implemented toward optimal strain relaxation engineering in this case. The corresponding operating parameters include compliant substrate thickness, growth surface orientation, and film compositional grading. Continuum elasticity and dislocation theory is combined with atomistic simulations to provide quantitative descriptions for the energetics of the film/substrate system and the dynamics of film strain relaxation during growth. Specifically, system-level analysis is based on functional forms for the energetics provided by the continuum theory and parametrized rigorously through molecular-statics and Monte Carlo simulations of structural and compositional relaxation.

In addition, the dynamics of strain relaxation is expressed through a phenomenological mean-field theoretical framework, where the dislocation field is described through a linear misfit dislocation density. Results are presented for strain relaxation through misfit dislocation formation in InAs/GaAs(111)A and InAs/GaAs(110). The results are compared with recent experimental measurements for model validation, and the modeling approach is used for further strain relaxation analysis in the InAs/GaAs heteroepitaxial system.

4:45 PM P5.11
A KINETIC MODEL FOR THE STRAIN RELAXATION IN HETEROEPITAXIAL THIN FILM SYSTEMS. Y.W. Zhang, S.J. Chua, Institute of Materials Research and Engineering, NUS, SINGAPORE; T.C. Wang, LNM, Institute of Mechanics, CAS, PR CHINA.

During heteroepitaxial growth, when the thickness of a thin film exceeds a critical value, usually the introduction of dislocations becomes energetically favourable. For many applications, the dislocations lying on the interface between the film and substrate are not deleterious. However, the so-called threading dislocations, which extend across the thickness of the film, can cause strong adverse effects. Yet to achieve a low threading dislocation density is still a daunting task. Energetic models were proposed to explain the strain relaxation process. However, these energetic models only described the relaxation process correctly at high temperatures. At lower and intermediate temperature ranges, dislocation kinetics must be taken into account. Most of previous kinetic models have assumed that the excess stress for the nucleation of threading dislocations is the same as that for the propagation of threading dislocations. And yet they do not consider the effect of the film surface roughness, which has been shown to be very sensitive to the threading dislocation density. In the present work, a kinetic model, which is based on the physical mechanisms of the mismatch strain relaxation, is proposed. In considering a threading dislocation nucleation process, we argue that the excess stress for threading dislocation nucleation is different from that for threading dislocation propagation. Therefore the excess stress for threading dislocation nucleation is derived. Considering the random distribution of threading dislocations, a formula for threading dislocation annihilation is proposed. Considering the hardening effect of misfit dislocations on the nucleation and motion of threading dislocations, a resistance term is introduced. Considering the sensitivity of dislocation nucleation to surface roughness, a parameter is introduced to consider this effect. We have tested this model on the $Si_{1-x}Ge_x/Si(100)$ systems. It is shown that the model has reproduced a wide range of experimental data. Moreover, the model predicts that smoothing surfaces plus high temperature growth can significantly reduce the threading dislocation density. The implications of the other predictions of this model on the threading dislocation reduction are also discussed.

SESSION P6: DISLOCATION FUNDAMENTALS: OBSERVATIONS, CALCULATIONS AND SIMULATIONS

Chair: Eric A. Stach
Thursday Morning, April 19, 2001
Salon 10/11 (Marriott)

8:30 AM *P6.1
SURFACE NUCLEATION OF TWIN BANDS IN THIN FILMS. P. Pirouz, Case Western Reserve University, Department of MS&E, Cleveland, OH.

Twinning is one of the mechanisms by which stress in a heteroepitaxial film relaxes. There are a few proposed mechanisms for the formation of deformation twin bands in bulk materials that involve the operation of multiplicative sources of partial dislocations. These mechanisms, e.g., the classical pole mechanism and the partial Frank-Read source, generally require the presence of pre-existing dislocations in the material, something that may not be present in the initial stages of film growth. Thus the possibility of repetitive surface nucleation of partial dislocations with the same Burgers vector on neighboring glide planes arises. However, the energetics of dislocation nucleation from a surface has often been considered in terms of overcoming the force exerted on an emerging dislocation by its image force. Consequently, and since this force varies as the reciprocal of the distance from the surface, the image force approaches infinity near the surface and the activation enthalpy for the nucleation turns out to be so large that dislocation nucleation cannot be considered to be a thermally activated process. Recent work has shown that dislocation nucleation from a surface step can dramatically decrease the activation enthalpy and bring it down to values where thermal activation may play a significant part in the process. In this paper, nucleation of total and partial dislocations from a surface step will be considered and the possibility of nucleation of similar partials on neighboring glides planes to form a twin microband discussed.

9:00 AM P6.2
MICROTWINNING AND RELAXATION IN EPITAXIAL FePd LAYERS GROWN BY MOLECULAR BEAM EPITAXY. D. Halley, Y. Samson, A. Marty, C. Beigne, P. Bayle-Guillemaud, J.E. Mazille, CEA, Grenoble, FRANCE; B. Gilles, CNRS/LTPCM, Grenoble, FRANCE.

We investigated the properties of equiatomic FePd epitaxial films grown by MBE on Pd (001). This fcc alloy shows an ordered CuAu (I) type structure (L10 phase) that attracts a considerable interest : under proper growth conditions, uniaxial ordering within this L10 phase leads to a perpendicular magnetic anisotropy. Here, we focus on the original epitaxial strain relaxation process in those films. The relaxation of the 1% misfit between Pd and the ordered FePd leads to the formation of microtwins. They are due to the pile-up of $a/6 < 112 >$ partial dislocations along adjacent $\{111\}$ planes, as observed by High Resolution Electron Microscopy. The emergence of such microtwins on the surface of the alloy causes discontinuities whose height is proportional to the number of dislocations constitutive of the microtwin. These discontinuities, aligned along $< 110 >$ directions were observed by Scanning Tunneling Microscopy, allowing a quantitative analysis Above a critical thickness, the density of microtwins evolves only slightly during growth, as new dislocations pile up along pre-existing microtwins rather than nucleating new microtwins. The statistical analysis of STM images provided the density of partial dislocations and proved that they took on most of the relaxation of the epitaxial misfit. Moreover, RHEED and X-Ray diffraction indicate an approximately linear dependence of the relaxation on the FePd thickness. STM demonstrates this is due to a linear dependence on the thickness of the number of dislocations in each microtwin. A theoretical explanation is obtained through an elastic modelling of the repulsion between dislocations cores. The microtwins have strong consequences on the magnetic domain walls as observed by Magnetic Force Microscopy: walls are clearly pinned on those defects, leading to straight magnetic walls along $< 110 >$ directions.

9:15 AM P6.3
DISLOCATIONS INTERSECTING A (111) SURFACE IN Ag: STM MICROSCOPY AND MOLECULAR DYNAMICS SIMULATIONS. Jesper Christiansen[a,b], Karina Morgenstern[c,d], Jakob Schiótz[a], Karsten W. Jacobsen[a]. [a]CAMP, Dept of Physics, Tech Univ of Denmark, Lyngby, DENMARK; [b]Materials Research Dept, Risó National Laboratory, Roskilde, DENMARK; [c]Inst of Experimental Physics, FU Berlin, Berlin, GERMANY; [d]CAMP, Dept of Physics and Astronomy, Univ of Århus, Århus, DENMARK.

We examine the intersection of dislocations with a (111) surface in silver. In an experimental setup, Scanning Tunneling Microscopy (STM) is used to measure step profiles of the dislocations. To investigate the situation theoretically we use Molecular Dynamics (MD) with a modified Effective Medium Theory potential, fitted to accurately reproduce the intrinsic stacking fault energy of silver, to find the quasi-stable configurations of a $\frac{1}{2}[110]$ screw dislocation intersecting a (111) surface with free boundary conditions. We examine two initial configurations in the simulation cell, a perfect screw dislocation and a configuration of two partial Shockley dislocations a few nm apart. After energy-minimization we determine which resulting structure ends up with the lower energy. We find that MD correctly reproduces the overall features of the experimental step profiles, including the splitting width and the width of the partials at the surface.

9:30 AM P6.4
THE NUCLEATION OF DISLOCATIONS AND SUBSEQUENT INTERACTIONS WITH FACETED INTERFACES IN Au THIN FILMS. Gene Lucadamo and Douglas L. Medlin, Thin Film and Interface Science Dept., Sandia National Laboratories, Livermore, CA.

Understanding how dislocations nucleate and interact with interfaces in thin films has both technological and scientific importance. To gain insight into these phenomena, we have studied the nucleation of dislocations at $\{112\}$ grain boundary facet junctions in sigma = 3, Au thin-film bicrystals using in situ transmission electron microscopy (TEM) and defect analysis techniques. In addition, the motion of the dislocations and the facet junctions was investigated as the dislocations migrated along $\{111\}$ twin boundaries that were parallel to the substrate surface. The TEM results showed that $1/6 < 112 >$-type dislocations, generally with screw character, propagated from $\{112\}$ facet corners and that the movement was frequently coupled with the motion of the boundary facets. These experimental results were used to develop and refine models providing a more detailed description of the interaction of dislocations with boundaries and facet junctions. This work is supported by the U.S. Department of Energy under contract No. DE-AC04-94AL85000 in part by the Office of Basic Energy Science, Division of Materials Science.

9:45 AM P6.5

INTERFACIAL FCC TO 9R RECONSTRUCTION VIA DISLOCATION EMISSION AT A 90 DEGREE [110] TILT BOUNDARY IN GOLD. D.L. Medlin, Thin Film and Interface Science Dept, Sandia National Laboratories, Livermore, CA; S.M. Foiles, Materials and Process Modeling Dept., Sandia National Laboratories, Albuquerque, NM.

We are investigating the structure of interfacial dislocations at grain boundaries in thin films. In this presentation, we discuss the relationship between the structural relaxations at (111)/(112) facets in 90° [110] tilt boundaries in epitaxial gold thin films and the dislocations present at this interface. Atomistic calculations and high resolution transmission electron microscopy observations show that the (111)/(112) interface possesses a dissociated structure consisting of a periodic array of stacking faults with one fault to every three planes. This stacking sequence is equivalent to the 9R phase, and is similar to the dissociated configuration that has been observed previously at $\Sigma=3$ (112) boundaries in low stacking fault energy metals. Key geometrical aspects of the relaxations at these two types of interface, including the distribution of stacking faults and the bending of planes near the boundaries, can be understood in terms of the dislocation structure of the interface. Both interfaces are composed of a periodic array of pure-edge (90°) and mixed (30°) character Shockley partial type dislocations. In the $\Sigma=3$ interface, the ratio of 90° to 30° dislocations is 1:2, whereas in the (111)/(112) interface the ratio is reversed, 2:1. In both cases, separation of the 90° and 30° dislocations produces the 9R sequence by introducing a stacking fault every third plane. Extensions of this dislocation description to more general boundaries will be discussed. This work is supported by the U.S. Department of Energy under contract No. DE-AC04-94AL85000 in part by the Office of Basic Energy Science, Division of Materials Science.

10:30 AM *P6.6

DISLOCATION CORE SPREADING AT METAL/AMORPHOUS INTERFACES. Huajian Gao, Lin Zhang, Dept of Mechanical Engineering, Stanford University, Stanford, CA; Shefford Baker, Dept of Materials Science and Engineering, Cornell University, Ithaca, NY.

It has been observed using transmission electron microscopy that the contrast of interface dislocations in Al-Cu thin films on SiO_2 substrates disappears in the electron beam. We model this phenomenon assuming this dislocation contrast dissolution is due to spreading of dislocation core along the metal/amorphous interface. We assume that slip along the interface occurs wherever shear stress exceeds the bonding strength, with slip rate governed by a linear viscous law. This problem is formulated as a coupled elasticity and diffusion problem. An implicit finite difference method with Gauss-Chebyshev quadrature scheme is developed to solve the resulting integro-differential equations. The film strength is investigated according to Nix-Freund model by taking into account the effect of dislocation core spreading. It is found that for weak interfaces, the film strength is strongly decreased by dislocation core spreading.

11:00 AM P6.7

A DETAILED STUDY OF THE TEXTURE EVOLUTION DURING MARTENSITIC PHASE TRANSFORMATION IN COBALT THIN FILMS. Heiko Th. Hesemann, Oliver Kraft, Eduard Arzt, Max-Planck-Institut für Metallforschung and Universität Stuttgart, Institut für Metallkunde, Stuttgart, GERMANY; Peter Müllner, ETH Zürich, Institut für Angewandte Physik, Zürich, SWITZERLAND; David Nowak, Shefford Baker, Cornell University, Department of MS&E, Ithaca, NY; Ken Finkelstein, Detlef Smilgies, CHESS, Ithaca, NY.

In this work, we have studied the influence of film stress and film thickness on the temperature induced fcc-hcp martensitic phase transformation in Co thin films. Martensite in shape memory alloy films is under consideration for use in microactuators. Co films with thickness between 0.2 μm and 3 μm were deposited onto silicon wafers and were investigated using the substrate curvature method and X-ray diffraction. It was found that the transformation is associated with a drop of the film stress in the thicker films, whereas no stress drop was detected in the 0.2 μm thick films. Furthermore, it was observed that the martensite start temperature decreases with increasing film stress. X-ray experiments conducted at the Cornell High Energy Synchrotron Source revealed that only a small fraction of Co transforms in the 0.2 μm thick films. For all film thicknesses, it was observed that the transformation behavior is closely linked to the texture. The transformation occurs only in texture components in which the transformation reduces the film stress. These results are discussed in the light of possible mechanisms for martensite nucleation and growth.

11:15 AM P6.8

A NONEQUILIBRIUM FIELD THEORY OF DISLOCATIONS. Amit Acharya, Carnegie Mellon University, Dept. of Civil & Env. Eng., Pittsburgh, PA.

A finite deformation, nonequilibrium field theory of dislocations is presented for the analysis of crystal plasticity in small scale structures. The theory provides a framework for the rigorous determination of the stress field(s) and temporal evolution of a single, a few or a distribution of dislocation(s) in a crystal, along with the associated large deformations produced due to dislocation motion. The constitutive inputs of the theory are specifications only for crystal elasticity and slip system dislocation velocities and nucleation. The model can account for the nonlinear elastic aspects of short-range interactions since it is not restricted by linearity in the (continuum) crystal elasticity which, following modern trends, may incorporate information on interatomic bindings and lattice periodicity arising from symmetry considerations. A few examples are solved to illustrate the recovery of conventional results and physically expected ones within the theory. Based on the theory of exterior differential equations, a non-singular solution for stress/strain fields of a screw dislocation in an infinite, isotropic, linear elastic solid is derived. A solution for an infinite, neo-Hookean nonlinear elastic continuum is also derived. Both solutions match with existing results outside the core region. Bounded solutions are predicted within the core in both cases. Assuming a constant dislocation velocity for simplifying the analysis, an evolutionary solution resulting in a slip-step on the boundary of a stress-free crystal produced due to the passage and exit of an edge dislocation is also described. The solution for the expansion of a polygonal dislocation loop under a constant velocity field is also derived within the theory.

11:30 AM P6.9

DISLOCATION NETWORKS STRAIN FIELDS INDUCED BY Si WAFER BONDING. J. Eymery, F. Fournel[a], K. Rousseau, D. Buttard, F. Leroy, F. Rieutord, J.L. Rouvière. CEA/Grenoble, Département de Recherche Fondamentale and [a]LETI/Département de Microtechnologies, Grenoble Cedex, FRANCE.

Buried dislocation superlattices are obtained by bonding ultra thin single crystal Si (001) films on (001) Si wafers [1]. With specific bonding conditions, the twist of the two Si wafers induces a regular square grid of dissociated dislocations and the tilt a 1-D array of straight mixed dislocations. As shown by electron microscopy, the Burgers vector is a/2 [110] for both types of dislocations. Grazing incidence X-ray diffraction is used to obtain complementary results concerning the subsurface dislocation strains fields. The regularity of the two networks gives a periodic displacement of the atomic positions and superlattice peaks in X-ray diffraction. The position and width of these peaks near the (220) in-plane reflection will be first discussed by taking into account instrumental resolution, and then the strain fields will be quantitatively analyzed by measuring the attenuation of the interfacial dislocation rods. These measurements are compared to a continuum theory model [2] taking into account the free surface and the thickness of the thin upper crystal for the screw and edge components of the dislocations. It is shown by these calculations and by X-ray experiments that the elastic strain field propagates up to the surface. This property is already used to obtain periodic nucleation centers for the self-assembled growth of nanostructures. [1] F. Fournel, H. Moriceau, N. Magnea, J. Eymery, J.L. Rouvière, and B. Aspar, Mat. Sci. & Eng B 73 (1-3) (2000) 42-46. [2] R. Bonnet and J.L. Verger-Gaugry, Phil. Mag. A, 66 (1992) 849.

11:45 AM P6.10

MECHANISMS OF THREADING DISLOCATION REDUCTION IN GROWING FILMS. Alexei Romanov, Ioffe Institute, St. Petersburg, RUSSIA; James S. Speck, Materials Dept., University of California, Santa Barbara, CA.

An approach has been developed to describe the threading dislocation (TD) density reduction in growing lattice-mismatched epitaxial films. Two fundamental issues in TD reduction include (i) relative dislocation motion and (ii) reactions between dislocations. One type of TD motion is specific to non-relaxed (i.e. strained films) when a mobile TD produces a misfit dislocation (MD) segment along film/substrate interface diminishing in such a way the global misfit strain and stress in the film. During the stress induced motion of TDs they may be blocked by MDs from conjugate families. These immobile TDs can then be unblocked as a result of the consequent film growth. The other type of effective TD motion takes place in growing relaxed films when the point, at which an inclined TD meets the film surface, laterally displaces when the film growth proceeds. In addition to blocking and/or unblocking of TDs the reactions among TDs themselves are possible. These are annihilation, fusion and scattering reactions. In the framework of the approach the following main results have been obtained. (a) The characteristic scaling law ($\sim 1/h$) for TD density variation with buffer layer thickness h has been proved. (b) The effect of slow TD reduction in GaN buffers has been explained by low TD reaction probabilities due to TD line directions practically normal to the film surface. (c) Exponential diminishing of TD density with the size of selective area growth regions has been demonstrated. (d) The restricted ability of a single strained layer in TD reduction has been established. (e) The role of relaxation enhancing interlayers in TD reduction has been shown to be related to the diminishing of MD local stresses and corresponding TD unblocking. The results of theoretical models are supported by experimental studies on TD reduction and strain relaxation in growing GaAs, $In_xGa_{1-x}As$ and GaN layers

SESSION P7: DISLOCATIONS AND DEFORMATION MECHANISMS IN THIN METAL FILMS AND MULTILAYERS II

Chairs: Richard P. Vinci and Daniel Josell
Thursday Afternoon, April 19, 2001
Salon 10/11 (Marriott)

1:30 PM P7.1
MISFIT DISLOCATIONS IN EPITAXIAL Ni/Cu BILAYER AND Cu/Ni/Cu TRILAYER THIN FILMS. Tadashi Yamamoto, Amit Misra, Richard G. Hoagland, Mike Nastasi, Harriet Kung, and John P. Hirth, Materials Science and Technology Division, Los Alamos National Laboratory, Los Alamos, NM.

Spacing and configuration of the misfit dislocations at the interfaces between two layers are of prime importance for the high-strength multilayers. In this preliminary report, misfit dislocations at the interfaces of bilayer (Ni/Cu) and trilayer (Cu/Ni/Cu) thin films are examined by plane-view TEM observation. Copper and nickel layers are prepared by sequential electron-beam evaporation and grown cube on cube onto the (001) surface of NaCl. In the bilayers, the spacing of misfit dislocations is measured as a function of nickel layer thickness. g-dot-b analysis of the cross-grid of misfit dislocations revealed edge character with $< 110 >$ Burgers vectors in the (001) interface plane. The critical thickness, at which misfit dislocations start to appear with the loss of coherency, was found to be about 2 nm. The spacing of the misfit dislocations decreases with increasing nickel layer thickness and reaches a plateau at the thickness of 300 nm. The minimum spacing is observed to be about 20 nm. In the trilayers, misfit dislocations formed at both interfaces. The spacing of the misfit dislocation is in agreement with that of the bilayers with similar nickel layer thickness. The misfit dislocation arrays at the two interfaces have the same line directions and Burgers vectors but are displaced with respect to each other in the interface plane. This suggests that the strain field of the dislocations has a strong influence on the position of the misfit dislocations at the subsequent interface. Mechanical properties of multilayers will also be discussed in correlation with the structure of misfit dislocations.

1:45 PM P7.2
MECHANICAL PROPERTIES OF ALUMINUM-SCANDIUM MULTILAYERS. Mark A. Phillips, Vidya Ramaswamy, William D. Nix, Bruce M. Clemens, Stanford University, Dept of MS&E, Stanford, CA.

Scandium additions to bulk aluminum alloys result in formation of Al_3Sc precipitates by homogenous nucleation. These precipitates are coherent with the matrix and produce a superior age-hardening effect. However, the maximum solid solubility of scandium in aluminum is about 0.25 at.%, so bulk systems contain only small interfacial regions, and it becomes difficult to experimentally separate interfacial effects from bulk effects. It is proposed that by construction of artificially layered materials (e.g. multilayer thin films), it will be easier to determine interfacial properties. Thin film deposition processes allow deposition of layers of any range of compositions, of any thickness, enabling production of a significant volume of Al-Al_3Sc interfaces for study by thin film testing techniques. In these experiments 1 μm thick polycrystalline Al and Al-Sc multilayer samples are grown via magnetron sputtering to examine the effects of interfaces on the strength of Al films. The films are tested by two methods: nanoindentation and bulge testing. Nanoindentation is widely used for the study of mechanical properties of thin films and provides a convenient method for comparison of film properties. In the bulge test, uniform pressure is applied to one side of a free standing thin film window, causing it to deflect. The stress and strain in the film can be determined from measurements of pressure and the window deflection. The bulge test is a flexible technique, permitting characterization of a film's elastic, plastic, and time-dependent deformation.

2:00 PM P7.3
STRUCTURE AND MECHANICAL BEHAVIOR RELATIONSHIP IN NANO-SCALED MULTILAYERED MATERIALS. N. Mara, A. Sergueeva, A.K. Mukherjee, Department of Chemical Engineering and Materials Science, University of California, Davis, CA.

Multilayered films form a very important group of materials in whose novel fundamental properties there is great interest [1]. More specifically, nanometer-scale polycrystalline multilayered films (with layer thickness less than 100 nm) have been the subject of many recent experimental and theoretical studies [2-5]. These fine-scale composite materials typically exhibit high yield strength, which at room temperature can approach half of the theoretical strength. Most attempts to characterize the mechanical behavior of such thin films have been carried out using nanoindentation and scanning force microscopy or their combination and there are no data regarding their mechanical behavior at elevated temperatures. In the present investigation, the microstructure and mechanical properties of polycrystalline Cu-Nb nanolayered composites prepared by sputtering method were evaluated. Samples were tested in uniaxial tension at temperature ranges from 25 to 550 degrees Celsius and at different strain rates using a custom-built computer controlled constant strain rate tensile test machine, which allows testing of very small samples with a 1-mm gage length and thicknesses as small as 0.01 mm and providing new opportunities in mechanical characterization of multilayered materials. The high strength of these new materials is attributed to their layered, nanoscale structure and a variety of related strengthening mechanisms. Their layered variations in composition, stiffness, residual elastic strain, crystal structure and interfacial defects are expected to impede dislocation glide between layers, while intralayer grain boundaries and their nanoscale layer thickness restricts dislocation motion within layers. Mechanical testing data suggests that some of these strengthening mechanisms are active in multilayers. This investigation is supported by a grant from the NSF Division of Materials Research. References. 1. L.L. Chang and B.C. Giessen (eds.), Synthetic Modulated Structures, Acad. Press, New York, 1986. 2. C. Suryananayana and F.H. Froes, Metall. Trans. A, 23A (1992) 1071. 3. S. Veprek, S. Reiprich, Thin Solid Films 268 (1995) 64. 4. S.A. Barnett and M. Shinn, in Annu. Rev. Mater. Sci., 24 (1994) 481. 5. J. Grilhe, in Mechanical Properties and deformation behavior of materials having ultra-fine microstructures, Kluwer Academic Publishers, Boston, MA, 1993, 255.

2:15 PM P7.4
CREEP BEHAVIOR OF MULTILAYER THIN FILMS. Daniel Josell, Metallurgy Division, NIST, Gaithersburg, MD; Alexis Lewis, Adriann Mann and Timothy Weihs, Johns Hopkins University, Baltimore, MD.

We describe the results of biaxial creep experiments with silver-nickel multilayers. The experiments utilize multilayer thin films on thin sapphire substrates whose curvature was studied in-situ during heating and annealing. Two phenomena, in particular, are emphasized in this presentation. The first feature is the occurence of two yield events that occur during heating of the specimens. These yield events are analyzed, using an analysis similar to that of Shen and Suresh to determine the properties of the individual metal layers in the composite structure. The second feature of these experiments is the occurence of an equilibrium, nonzero biaxial curvature even at temperatures above those at which the Ag and Ni layers have begun to creep. The nonzero biaxial curvature is generated by the interface free energy (tension) associated with the many Ag/Ni interfaces in the multilayer samples. These interfaces strive to reduce their area, hence free energy, by contracting the films. However, the films are not freestanding; the Ag/Ni interfaces cannot contract the film without bending the substrate. The curvature of the elastic substrate therefore changes until the force that it imposes on the film matches that required to balance the interface tensions. Using the equilibrium curvatures obtained after annealing at high temperatures, with appropriate microstructural information and the analysis of Josell, we have made the first measurements of the Ag/Ni interface free energy using a biaxial creep geometry. In addition, we have shown that one must account for effects of interfaces when performing stress relaxation experiments on multilayer films on substrates. Namely, when relating stress to strain rate, one must assume that the film relaxes to an effective nonzero stress state. Y.L. Shen and S. Suresh, Acta Mater 44, 1337 (1996). D. Josell, Acta Metall. Mater. 42, 1031 (1994).

2:30 PM P7.5
ANALYSIS OF DISLOCATION-MEDIATED VOID GROWTH MECHANISMS IN STRAINED METALLIC THIN FILMS. Dimitrios Maroudas and M. Rauf Gungor, Department of Chemical Engineering, University of California, Santa Barbara, CA.

Void growth and evolution underlies a number of failure mechanisms in strained ductile metallic thin films. Continuum-level quantitative descriptions of void dynamics require a fundamental understanding of the elementary dynamical mechanisms, as well as constitutive relations for plastic deformation phenomena in the vicinity of the evolving void surface. Toward this end, molecular-dynamics (MD) simulations provide an excellent source of detailed information. In this presentation, the response to hydrostatic and biaxial tensile straining is analyzed of cylindrical voids that extend throughout the supercell thickness along the [111] crystallographic direction; the analysis is based on isothermal, isostrain MD simulations using supercells that contain millions of atoms and following an embedded-atom-method parametrization for Cu. Monitoring the evolution of the void volume and surface morphology is used to examine void growth and faceting. In addition, the dislocation field, stress field, and atomic mobility in the vicinity of the growing void are analyzed in detail. For both modes of straining and over a range of temperature and strain, the MD simulations reveal that void growth is mediated by screw dislocation pair emission from the void surface and subsequent dislocation propagation; as a result, the void is surrounded by a plastic zone outside of which the strain is practically equal to the applied strain. Over the same range of operating parameters, the kinetics of void growth is examined systematically and analytical forms are derived for the evolution of the void fraction. Furthermore, the void growth rate also is expressed in terms of the plastic displacement rates normal to the void surface. These analytical expressions provide effective constitutive links between MD simulations and continuum mechanical failure models in ductile metallic films.

2:45 PM P7.6

DISLOCATIONS IN THIN METAL FILMS OBSERVED WITH X-RAY DIFFRACTION. Léon J. Seijbel, Rob Delhez, Laboratory for Materials Science, Delft University of Technology, Delft, NETHERLANDS.

We present the simultaneous determination of stress and of dislocation densities in thin metal layers by X-ray diffraction methods. This implies the measurement of macro-stress and strain (from diffraction-line positions as a function of specimen tilt) and the measurement of micro-stress and strain (from the broadening of the same diffraction-line profiles). By measuring the diffraction-line profiles under various angles with the layers, it is possible to obtain the dislocation densities on all present glide planes by a modified size-strain separation method. In our method the elastic anisotropy of the materials is taken into account. The method has been applied to three layer substrate systems: 500 nm molybdenum sputter deposited on an oxidized silicon (100) wafer (Mo/Si), 500 nm nickel sputter deposited on an oxidized silicon (100) wafer (Ni/Si), and 2 μm nickel electrochemically deposited on steel (Ni/Fe). The Mo/Si system has a sharp fiber and in plane texture. This reduces the number of possible slip systems, i.e. angles between the diffraction vector and the Burgers vector to four and thus enables a detailed analysis. In the two nickel systems the texture is not that sharp and therefore the orientations of the Burgers vectors with respect to the surface normal do not fall in a small angular range and the dislocations must be divided into “orientation groups”. In the Ni/Fe system the stress in the layer is only determined by the intrinsic or growth stress, because the difference in thermal expansion between Ni and Fe is small. The stress in the Ni/Si system is determined also by the thermal stresses caused by the thermal treatments. The different behaviours of the stresses and the dislocations in the three systems can be related to the characteristics of the individual systems.

3:30 PM P7.7

MICROSTRUCTURE AND STRESS IN SUBMICRON PASSIVATED Al(Cu) LINES STUDIED BY X-RAY MICRODIFFRACTION. Bryan C. Valek[a], N. Tamura[b], R. Spolenak[c], A.A. MacDowell[b], R.C. Celestre[b], H.A. Padmore[b], J.C. Bravman[a], W.L. Brown[c], B.W. Batterman[b,d], and J.R. Patel[b,d]; [a]Dept. Materials Science & Engineering, Stanford University, Stanford, CA; [b]ALS/LBNL, Berkeley, CA; [c]Bell Laboratories, Lucent Technologies, Murray Hill, NJ; [d]SSRL/SLAC, Stanford University, Stanford, CA.

Microstructure and mechanical properties at the microscopic scale (grain orientation, grain boundaries, grain size distribution, local strain/stress gradients, defects, etc.) are prominent parameters defining the electromigration resistance of interconnect lines in modern integrated circuits. Recently, techniques have been developed for using submicron focused white and monochromatic x-ray beams to obtain local orientation and strain information within individual grains of thin film materials. In this work, we use the X-ray microdiffraction beamline (7.3.3) at the ALS to map the orientation and local stress variations in passivated Al(Cu) test structures (width: 0.7, 4.1 μm) as well as in Al(Cu) blanket films. The temperature effects on microstructure and stress were studied in those same lines by in-situ orientation and stress mapping during a temperature cycle between 25°C and 265°C. Results show large variations in the different stress components which significantly depart from their average values obtained by more conventional X-ray techniques using laboratory sources. Implications of these results on the mechanical properties of these lines as well as on their behavior during service will be presented.

3:45 PM P7.8

PLASTICITY OF ELECTROMIGRATION-INDUCED HILLOCKING AND HOW IT AFFECTS THE CRITICAL LENGTH. Joris Proost[a,c], K. Maex[b] and L. Delaey[a], [a]Dept. of Materials Science, Leuven University, BELGIUM; [b]IMEC, Leuven, BELGIUM; [c]Div. of Engineering and Applied Sciences, Harvard University, Cambridge, MA.

When passing electrical currents through metallic conductor lines in integrated circuits, the resulting drift velocity is generally taken to decrease linearly with the inverse line length, following the work of Blech. A central parameter in Blech’s theory is the threshold length, defined as the critical interconnect length at which the electromigration flux completely vanishes due to a counteracting mechanodiffusion flux. We provide experimental evidence from drift experiments on pure Al for a deviation of the length-dependence of the drift rate from the widely used Blech equation. This discrepancy is especially manifested in the near-threshold regime. New analytical expressions are presented for the length-dependence of the drift velocity by considering diffusional creep as the plastic flow mode involved in EM-induced hillocking. It will be shown how this relaxes one of the major assumptions of the classical Blech theory, in which the stress at the anode is kept constant at a threshold stress. The new model is validated to the experimental data by quantifying the diffusional flows involved in both EM-induced drift and hillocking. Also the impact on the determination of EM-threshold is discussed.

4:00 PM P7.9

DEFORMATION MECHANISMS IN ANODIC OXIDE THIN FILMS DEPOSITED ON BCC REFRACTORY METALS. R. Gibala, Department of MS&E, The University of Michigan, Ann Arbor, MI.

Anodically deposited thin oxide films on body-centered cubic metals such as Nb, Ta, Mo, and W can exhibit unusually large plasticities during deformation of the film-substrate composite. The oxide-coated bcc metal substrates can also often exhibit substantial softening (plasticity enhancement) relative to the baseline uncoated material. Such results are presented for Group V and Group VI metals with amorphous oxide films deposited anodically on them. Oxides in the range of thicknesses of 50-500 nm deform compatibly with metal substrates or, in some cases, with deformable polymer substrates on which they have been deposited. The oxides deform plastically by deformation shear banding which is morphologically similar to that observed in amorphous polymers and metallic alloys. The effect of several deformation and deposition variables are described. Attempts to model the deformation shear banding process in thse ductile oxide thin film is also discussed.

4:15 PM P7.10

DEFORMATION MICROSTRUCTURE OF COLD SPRAYED COATINGS STUDIED BY ELECTRON MICROSCOPY. Christine Borchers, Thorsten Stoltenhoff, Frank Gärtner, Heinrich Kreye, Univ of the Federal Armed Forces, Dept of Mechanical Engineering, Hamburg, GERMANY; Hamid Assadi, Tarbiat Modarres Univ, Dept of MS&E, Tehran, IRAN; Klaus Krug, Georg August Univ, I. Physical Institute, Göttingen, GERMANY.

Cold spraying is a new coating technique, in which the formation of dense, tightly bonded coatings occurs only due to the kinetic energy of high velocity particles of the spray powder. These particles are still in the solid state as they impinge on the substrate. The bonding of the particles is a result of extensive plastic deformation and related phenomena at the interfaces. The deformation microstructure is found to vary largely with the mechanical properties of spray material and the impact velocities. Finite element modelling shows that plastic shear instability at the interfaces is a crucial criterion for tight bonding of the particles. TEM investigations can relate these shear instabilities to the localized deformation microstructures of the particle/particle interfaces. The analysis indicates thermal influence at the contact zones, which can be attributed to localized heat release upon particle impact. The heat release also gives evidence for a mechanical softening of the particle/particle interfaces during the high strain rate deformation. In addition, the defect density is studied by resistivity measurements. Whereas at room temperature the measured resistivity of cold sprayed copper coatings is no more than 10% higher than the resistivity of pure bulk copper, which indicates good macroscopic properties, the residual resistivity gives further information on the microscopic defect structures.

4:30 PM P7.11

PLASTIC DEFORMATION OF THIN METAL FOILS WITHOUT DISLOCATIONS AND FORMATION OF POINT DEFECTS AND POINT DEFECT CLUSTERS. Michio Kiritani, Academic Frontier Research Center for Ultra-High-speed Plastic Deformation, Hiroshima Institute of Technology, Hiroshima, JAPAN; Yoshiharu Shimomura, Faculty of Engineering, Hiroshima University, Higashi-hiroshima, JAPAN.

Discovery of the formation of anomalous high number density of

vacancy clusters in very thin areas of elongated thin foils lead the present authors to propose the plastic deformation of crystalline metals without dislocations (Phil. Mag. Letters, 79 (1999) 797).
Experiments are performed to confirm the proposed mechanism of deformation and also to elucidate the point defect reactions. Materials investigated are fcc metals (Al, Au, Cu, Ni and their alloys), bcc metals (Fe, V) and ordered alloys (Cu_3Au, Ni_3Al).
All the point defect clusters in fcc metals are vacancy clusters in the form of stacking fault tetrahedron even in aluminum. Dynamic microstructural evolution was observed during in-situ deformation in an electron microscope. When the deformation proceeds slowly on a wide area, vacancy clusters which had been introduced by prior deformation gradually disappeared. When a heavy deformation proceeds in a localized narrow area, new vacancy clusters are generated. During these heavy deformation up to more than 100% in strain, the absence of the generation and motion of dislocations which can account for the amount of deformation was confirmed.
Variation of the formation of vacancy clusters in fcc metals was investigated by widely changing the elongation speed and deformation temperature. The deformation speed below which the vacancy cluster formation decreases was found to correspond to the condition for vacancies to escape to specimen surface during deformation, namely indicating vacancies are formed as vacancies not as observable vacancy clusters.
Other metals behaved similar to fcc metals in terms of deformation morphology, though the point defect cluster formation was quite different.
Molecular Dynamics simulation the mechanism of the plastic deformation without dislocation which results in the point defect generation is now under progress.
Result of high-speed deformation (strain rate up to 10^7/s) which aims at the deformation of bulk samples without dislocation will also be reported

4:45 PM P7.12
COMPUTER SIMULATION STUDY OF ATOMISTIC PROCESSES OF PLASTIC DEFORMATION OF FCC THIN FILMS AND GENERATION OF VACANCIES. Y. Shimomura, Faculty of Engineering, Hiroshima University, Higashi-Hiroshima, JAPAN; M. Kiritani, Academic Frontier Research Center for Ultra-High-Speed Plastic Deformation, Hiroshima Institute of Technology, Hiroshima, JAPAN.

Kiritani et. al. (Phil. Mat. Lett., 79 (1999) 797) reported that a large number of vacancy clusters are formed in elongated thin fcc metal films such as aluminum copper and gold. Electronmicroscopically observable thin films were formed before the final stage of fracture. Dislocations were not observed in these thin films, which suggested that plastic deformation is not due to dislocation glide, and the generatin of vacancies is responsible or at least the formation of vacancies is the result of the deformation without dislocation. A Molecular Dynamics computer simulation is carried out using EAM potentials to study the atomistic mechanism of plastic deformation of elongated thin films. In the case of very thin films such as 10 atomic layer thick, dislocations can be generated at surfaces, starting from fluctuation tilting of atom rows at surfaces under stress. Such dislocations cannot nucleate in a little thicker foil of 100 nm thick due to the difficulty of tilting of long atom row at the surface. An alternative mechanism of plastic deformation of thin foil observed during simulation is as follows. When a thin film of fcc crystal is deformed elastically to about 8%, atom rows become distorted from a regular fcc configuration and small islands of extra atom rows start to nucleate between (111) planes. The formation of these extra atom rows are carried out by the movement of atoms as $< 110 >$ crow-ions from nearby atom positions. Multiplication of these (111) planes carry out the deformation releasing the accumulated elastic stress. As the result, vacancies and their small clusters are left at the position from which atoms were sent out. Vacancy reaction processes which leads to the formation of observable vacancy clusters are also examined.