Final Technical Report

for

DOE Grant No. DE-FG03-89ER45387

MECHANICAL PROPERTIES OF MATERIALS WITH NANOMETER SCALE MICROSTRUCTURES

for the period

1 April, 1989 to 31 August, 2004

Submitted to:

Division of Materials Sciences
Office of Basic Energy Sciences
United States Department of Energy
Washington D.C. 20545

Attention: Dr. Yok Chen

Submitted by:

Department of Materials Science and Engineering
Stanford University, Stanford California 94305

Principal Investigator: Professor William D. Nix

October, 2004

Table of Contents

I. Introduction

We have been engaged in research on the mechanical properties of materials with nanometer-scale microstructural dimensions. Our attention has been focused on studying the mechanical properties of thin films and interfaces and very small volumes of material. Because the dimensions of thin film samples are small (typically 1 μm in thickness, or less), specialized mechanical testing techniques based on nanoindentation, microbeam bending and dynamic vibration of micromachined structures have been developed and used. Here we report briefly on some of the results we have obtained over the past three years. We also give a summary of all of the dissertations, talks and publications completed on this grant during the past 15 years.

Section II of this report includes a brief summary of our now recently published work on the mechanical properties of Al/Al_3Sc multilayers. Other reports in section II focus mainly on results that have not yet been published. Section III of the report is a list of all of the Ph.D. dissertations that have been supported by this grant. The list extends over 15 years and is numbered to reflect the total number of DOE supported Ph.D. students who have studied with W.D. Nix over the years. A complete list of oral presentations for the entire 15 year grant period is given in Section IV of the report. Finally, Section V of the report provides a complete list of papers that have been published during the life of the grant.

The revolution in microelectronics and the associated development of high density, data storage technologies have brought about a great demand for understanding and controlling the mechanical properties of thin films and their interfaces. The extraordinary development of the microprocessor is directly related to the shrinking feature size in integrated circuit technology. Although the primary functions of these materials are electrical in nature, the mechanical properties of these materials and their interfaces are of crucial importance to the successful manufacture and use of these devices. Thus the mechanical properties of microelectronic thin films have become as important to the manufacture of integrated circuits as the mechanical properties of structural materials are to the building of advanced structural systems. Micro-electro-mechanical systems (MEMS) also make extensive use of thin metal films on substrates. The mechanical properties of these materials are of crucial importance for both device performance and reliability. All of these thin film device developments have provided technological motivation for our research. Additionally, understanding the mechanical properties of materials at the nanometer scale has become an important and challenging scientific problem. The effort to understand the mechanical properties of materials at the nanometer-scale is contributing to our fundamental understanding of the controlling mechanisms of mechanical behavior.

One of the major accomplishments of the past three years has been the completion of our work on the mechanical properties of Al/Al_3Sc multilayers. We have made and studied the mechanical properties of Al/Al_3Sc multilayers consisting of inherently soft Al layers (6–100 nm in thickness) separated by thin (0.5–5nm) layers of the ordered intermetallic phase Al_3Sc, which is coherent with the Al layers. These multilayers are perhaps the strongest Al based alloys ever created. In addition, they represent a model material system for studying strengthening processes in multilayers. The Al_3Sc layers are perfectly coherent with the Al layers, making these alloys analogous to Ni/Ni_3Al superalloys of importance for high temperature applications. Our studies of the mechanical properties of these materials have been conducted using nanoindentation. We find that the hardnesses of the multilayers with the thickest Al layers are well described by a model in which dislocations are confined to the soft Al layers. But for thinner Al layers the strength falls below that predicted by the model of dislocation confinement. This weakening is especially pronounced for the thinnest Al_3Sc layers, suggesting that shearing of the Al_3Sc layers is responsible for the weakening. We have modeled these deformation processes and have concluded that the weakening effects are caused by the annihilation of oppositely signed dislocations causing the Al_3Sc layers to be sheared. The models we have developed for these processes coincide well with the experimental data.

We have also completed our work on the effects of microstructure on the early stages of nanoindentation of gold thin films. We find that the microstructure has a huge effect on the nature of the load-displacement curve at small depths of indention. When very small indentations are made near the centers of large grains, single crystal like behavior is observed. The load-displacement curve is characterized by Hertzian loading up to a critical load, followed by a displacement burst associated with the nucleation of dislocations. Very high contact stresses develop in the early stages of indentation and they are followed by much lower contact stresses after dislocations are nucleated. Such indentations are thus characterized by strain softening. These observations also allow the theoretical shear strength of the crystal to be estimated. The measured shear strengths are close to those predicted by the Frenkel relation. But the indentation behavior of very thin films of gold having very small grain sizes is quite different. For these microstructures we find smooth indentation loading curves indicating initially "soft" behavior at the smallest depths of indentation. We have argued that these effects are caused by the close proximity of grain boundaries, which act as easy sources for dislocations. Our EAM modeling of these processes confirms that grain boundaries can act as sources of dislocations and that this can have profound effects on the nature of the load-displacement curve. For such fine-grained films the contact pressure gradually increases in the early stages of indentation, suggesting the expected effects of strain hardening. These studies clarify the role of microstructures on the nature of nanoindentation in the early stages of indentation.

We have developed a microbeam bending technique for determining elastic-plastic, stress-strain relations for thin metal films on silicon substrates. The method is similar to previous microbeam bending techniques, except that triangular silicon microbeams are used in place of rectangular beams. The triangular beam has the advantage that the entire film on the top surface of the beam is subjected to a uniform state of plane strain as the beam is deflected, unlike the standard rectangular geometry where the bending is concentrated at the support. We have developed a method of analysis for determining two Ramberg-Osgood parameters describing the stress-strain relation for the film. These parameters are obtained by fitting the elastic-plastic model to the measured load-displacement data, and utilizing the known elastic properties of both film and substrate. As a part of the analysis we compute the position of the neutral plane for bending, which changes as the film deforms plastically. This knowledge, in turn, allows average stress-strain relations to be determined accurately without forcing the film to closely follow the Ramberg-Osgood law. The method we have developed can be used to determine the elastic-plastic properties of thin metal films on silicon substrates up to strains of about 1%. Utilizing this technique, both yielding and strain hardening of Cu thin films on silicon substrates have been investigated. Copper films with dual crystallographic textures and different grain sizes, as well as others with strong <111> textures have been studied. Three strongly textured <111> films were studied to examine the effect of film thickness on the deformation properties of the film. These films show very high rates of work hardening, and an increase in the yield stress and work hardening rate with decreasing film thickness, consistent with current dislocation models.

A new dynamic measurement system has been developed to investigate damping in thin metal films. This system includes a vacuum chamber, in which a free-standing bilayer cantilever sample is vibrated using an electrostatic force, and a laser interferometer to measure the displacement and velocity of the sample. With this equipment, internal friction as low as 10^{-5} in micrometer thick metal films in a temperature range of 300K to 750K can be measured. Free-standing cantilevers with different frequencies have been fabricated using well-established IC fabrication processes. The cantilevers consist of thin metal films on thicker Si substrates, which exhibit low damping. From measurements of internal friction of Al thin films at various temperatures and frequencies, it is possible to study relaxation processes associated with grain boundary diffusion. The activation energy calculated from the damping data is 0.57eV, which is consistent with previous research. This value suggests that the mechanism of internal friction in pure Al films involves diffusion-controlled grain boundary sliding. A model to describe these damping effects has been developed. By deriving an expression for the diffusional strain rate using a two-dimensional Coble creep model and modifying the conventional standard linear solid model for the case of bending, it is possible to give a good account of the observed damping.

II. Research Report

A. Microstructure and nanoindentation hardness of Al/Al_3Sc Multilayers

(Mark A. Phillips)

In the paper describing this work, we discuss the fabrication, characterization and mechanical properties of a unique multilayer system constructed of aluminum and scandium. Thin film deposition techniques were used to create high-quality polycrystalline multilayered films consisting of inherently soft Al layers (6–100 nm in thickness) separated by thin (0.5–5nm) layers of the ordered intermetallic phase Al_3Sc, which is coherent with the Al layers. We used X-ray diffraction, transmission electron microscopy and in situ wafer curvature to characterize the microstructure and intermixing of the Al/Al_3Sc multilayer films produced by sputter deposition. The characterization work is described in the paper cited below.

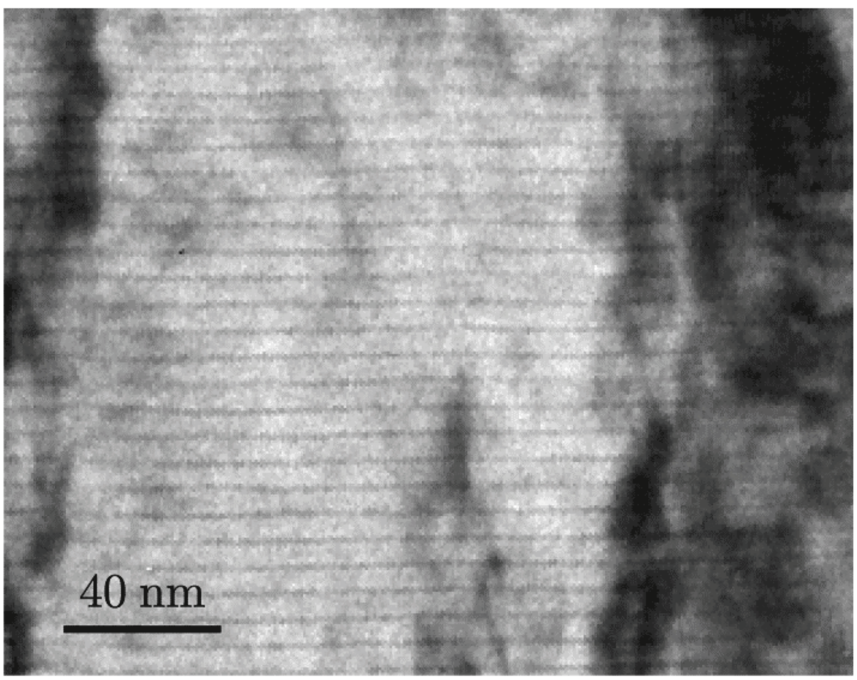

Fig. A.1 Transmission electron micrograph showing a coherent Al/Al_3Sc multilayer made by sputter deposition.

To illustrate the multilayer structures created in this research, we show in Fig. A.1 a transmission electron micrograph of the multilayer structure. High resolution electron microscopy showed that the Al_3Sc layers are fully coherent with the Al layers. The simplicity of this microstructure makes this a particularly good model system for studying strengthening processes in metal multilayers.

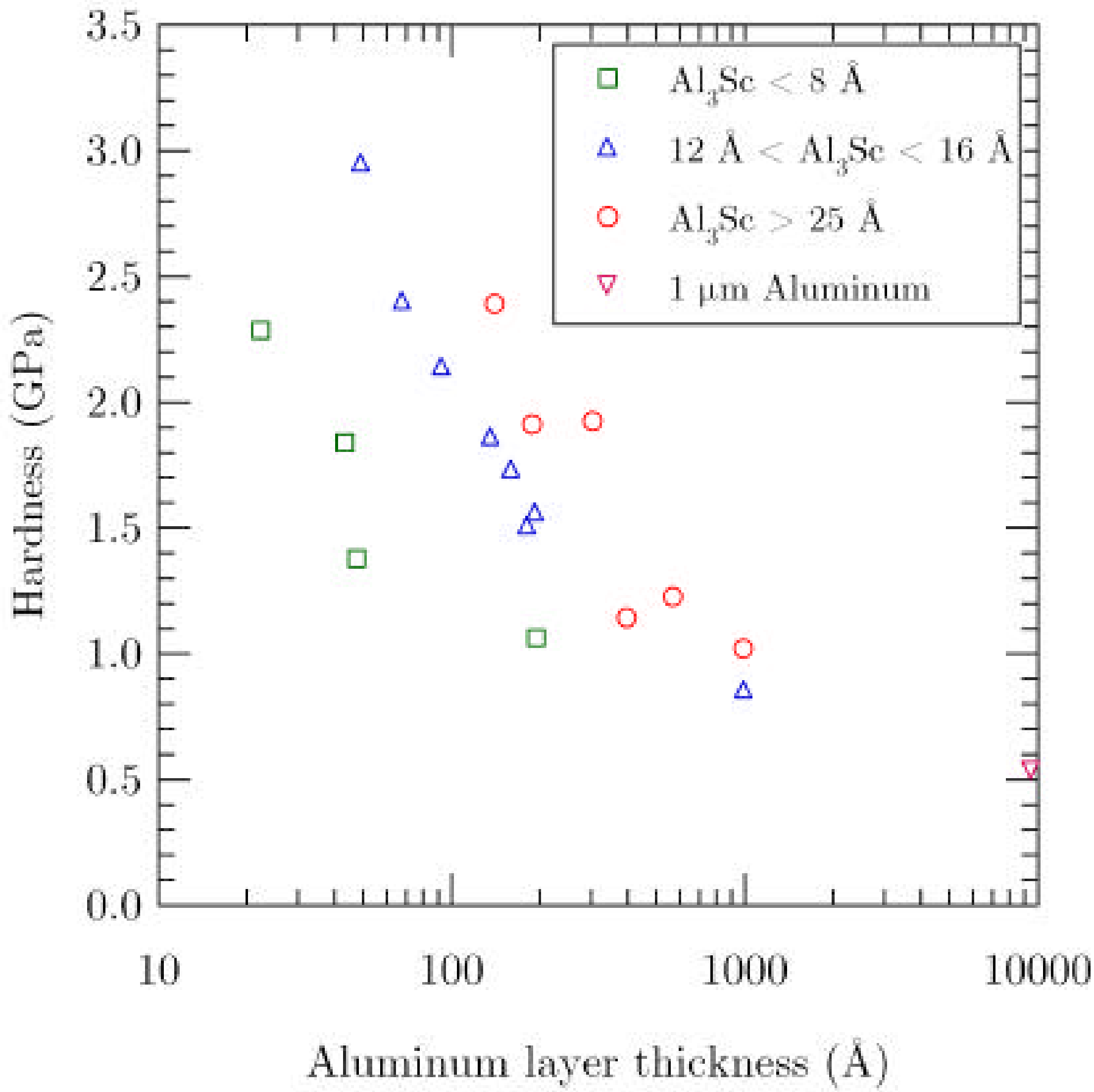

Fig. A.2 Hardness of Al/ Al_3Sc multilayers as a function of Al layer thickness. The different groups of data correspond to different Al_3Sc layer thicknesses.

The accompanying hardness results show that layering with nanoscale coherent Al_3Sc layers increases the hardness by between 200 and 500% over that of a pure aluminum film. Figure A.2 shows the measured hardness of various multilayers as a function of the Al layer thickness. The data are shown in three groups, with different Al_3Sc thicknesses. While the strength clearly increases with decreasing Al layer thickness, as expected, the multilayers with the thinnest Al_3Sc are weakest. As described in the paper cited below, we believe this dependence on the thickness of the Al_3Sc layers is related to shearing of the coherent barrier layers by the collapse of dislocation dipoles.

M.A. Phillips, B.M. Clemens and W.D. Nix, "Microstructure and nanoindentation hardness of Al/Al_3Sc multilayers," Acta Materialia, 51, 3157-3170 (2003).

B. A model for dislocation behavior during deformation of Al/Al3Sc (fcc/L12) metallic multilayers

Mark A. Phillips

In the previous paper, we described the microstructure and indentation hardness of nanoscale Al/Al_3Sc multilayers. In the work described by the second paper, cited below, the large increases in hardness with decreasing aluminum layer thickness are analyzed in detail and several simple dislocation mechanisms are proposed to describe the behavior. Strengthening can be explained by assuming that yielding occurs first by forming dislocation loops in the aluminum layers—the dislocations are constrained by layers of Al_3Sc. We show that the anti-phase boundary (APB) energy of the L12, Al_3Sc structure can be used to quantify the resistance of the interfaces in the multilayer structure to dislocation motion and that the thickness of the Al_3Sc layer also plays an important role in determining the strengthening response.

Figure B.1 shows the result of such modeling. The line shows the strength predictions for the case in which dislocations are completely confined to the Al layers and not allowed to penetrate the Al_3Sc barrier layers. We see that for the largest Al layer thickness and for the thickest Al_3Sc layers, the model coincides with the data. But for thinner Al layers and especially for very thin Al_3Sc layers the model greatly over predicts the observed strengths. As discussed in the paper cited below, we believe these "weakening" effects arise because the thinner Al_3Sc layers are cut by collapsing dislocation dipoles.

1. M.A. Phillips, B.M. Clemens and W.D. Nix, "A model for dislocation behavior during deformation of Al/Al_3Sc (fcc/L1(2))metallic multilayers," Acta Materialia, 51, 3171-3184 (2003).

$$H = 2.7\,\sigma_y$$

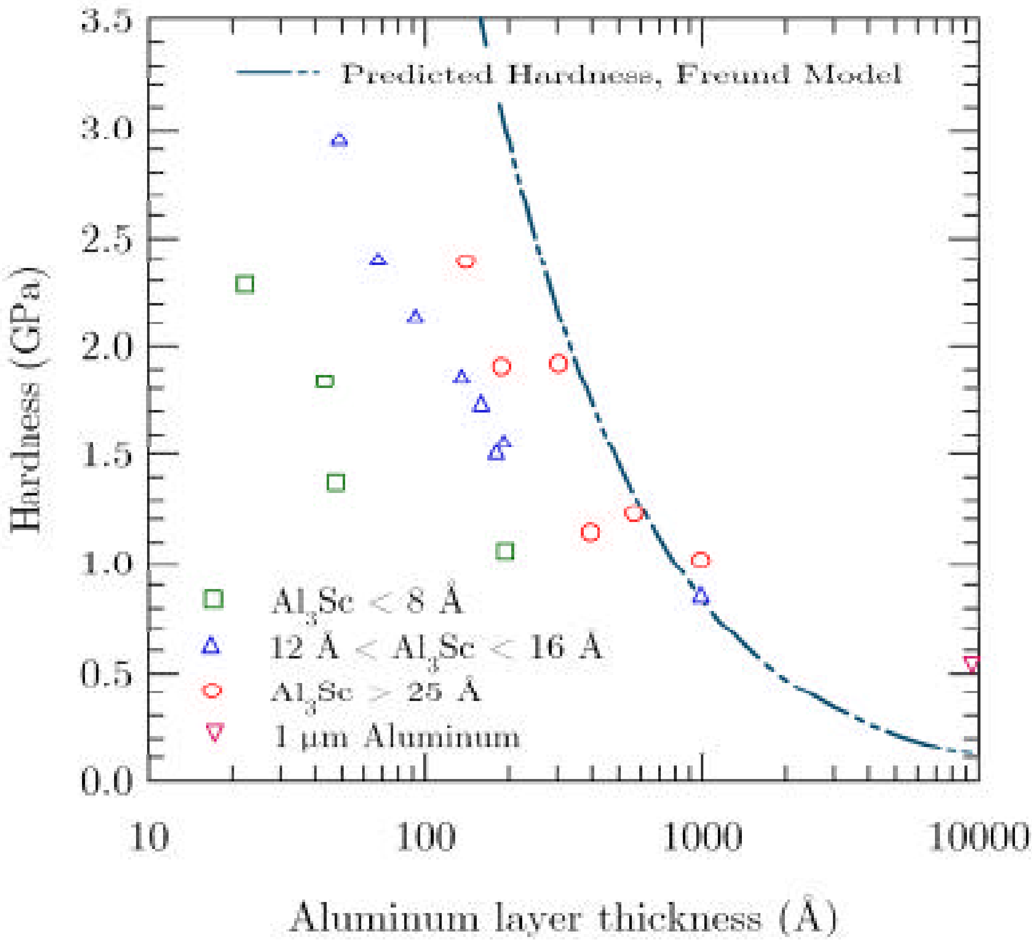

Fig. B.1 Hardness of Al/ Al_3Sc multilayers as a function of Al layer thickness. The different groups of data correspond to different Al_3Sc layer thicknesses. The line shows the strength predicted if dislocations are completely confined to the Al layers.

C. Microstructural length-scale effects in the nanoindentation behavior of thin gold films

(Erica T Lilleodden)

Nanoindentation experiments have been conducted on epitaxial and polycrystalline gold thin films in an effort to study the effects of microstructural length-scales on indentation plasticity. We show that the deformation of single-crystalline and coarse-grained films is characterized by Hertzian elastic loading, followed by discrete displacement bursts and subsequent softening with increasing depth of indentation. In contrast, the loading response of fine-grained films is continuous, and characterized by elastic-plastic behavior from the earliest stages of indentation, and gradual hardening with increasing indentation depth. We argue that these different behaviors are closely related to the mechanisms of initiating plasticity. Homogeneous dislocation nucleation at theoretical shear stresses is required for indentation into dislocation-free, single crystalline volumes while grain boundaries provide a ready source for dislocations in fine-grained films. This picture of indentation plasticity is additionally supported by observations of grain boundary proximity effects on the critical load at which plasticity commences.

Here we show some of the most important results of this study. Fig. C.1 shows an AFM image of an epitaxial film of gold that was grown onto a (001) silicon substrate. The figure shows flat terraces about 400 nm in width, separated in height from each other by about 3 nm. This is an ideal sample for study, as the terraces are extremely flat and the epitaxial crystals are of good quality. Five different load-displacement curves obtained for this film are shown in Fig. C.2. These indentation results were obtained using a Berkovich indenter with a tip radius of about 50 nm. We see that the load-displacement relations for the five tests are in almost perfect agreement up to an indentation depth of about 5 nm. These load-displacement curves indicate purely elastic, Hertzian contact between the rounded diamond indenter and the gold surface. The elastic loading curves can be accurately predicted using the Hertz theory and the elastic properties of gold and diamond. But displacement bursts occur for all of the indentations at displacements greater than about 5 nm. These displacement excursions are caused by the nucleation of dislocations in these highly perfect crystals. From the critical loads needed to initiate the displacement bursts, one can estimate the theoretical shear strength of gold. We find critical shear strengths close to the Frenkel estimate of $\tau_{th} = \mu / 2\pi$. The different critical loads for the different tests are probably related to the proximity of the indentations to the ledges shown in Fig. C.1. We assume that the strain bursts will occur at smaller loads if the indentations are close to the ledges, because of the effects of stress concentrations at the ledges. Thus the highest critical loads correspond to the best estimates of the theoretical strength.

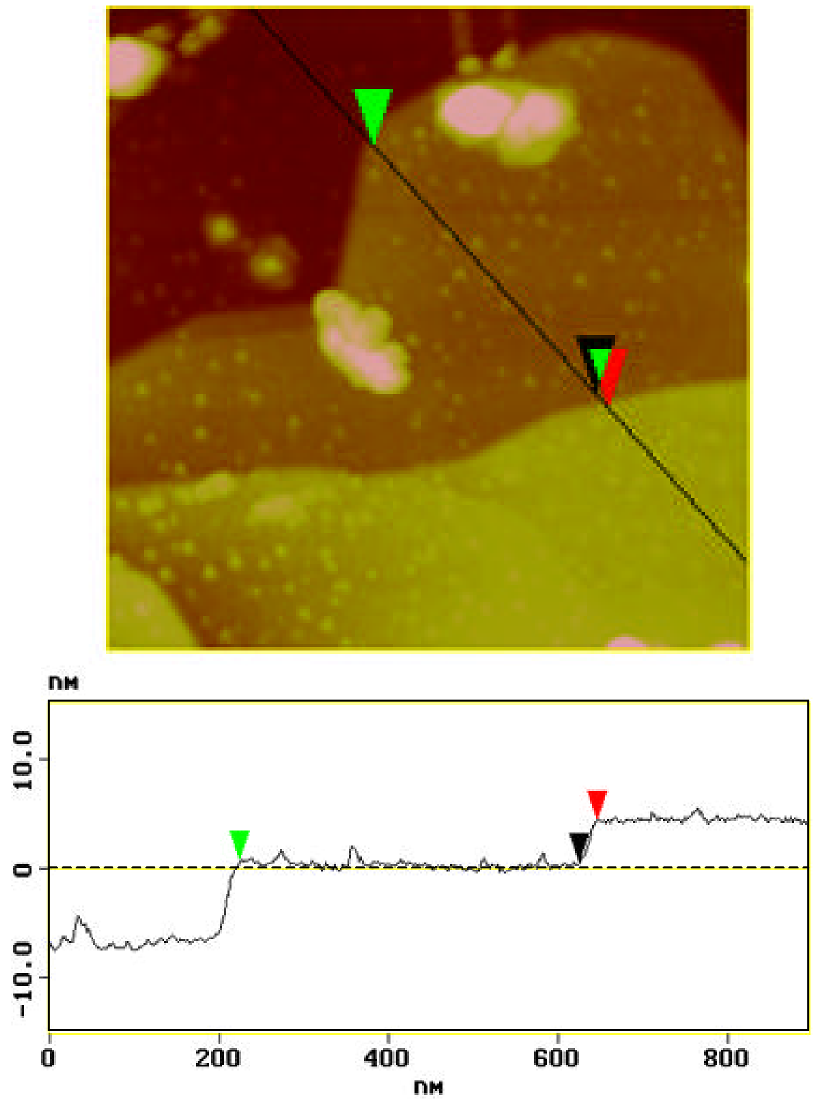

Fig. C.1 AFM image and corresponding cross-sectional profile across the terraced surface of the 160 nm (001) Au bi-crystalline film shows steps approximately 3 nm high and spaced 400 nm apart.

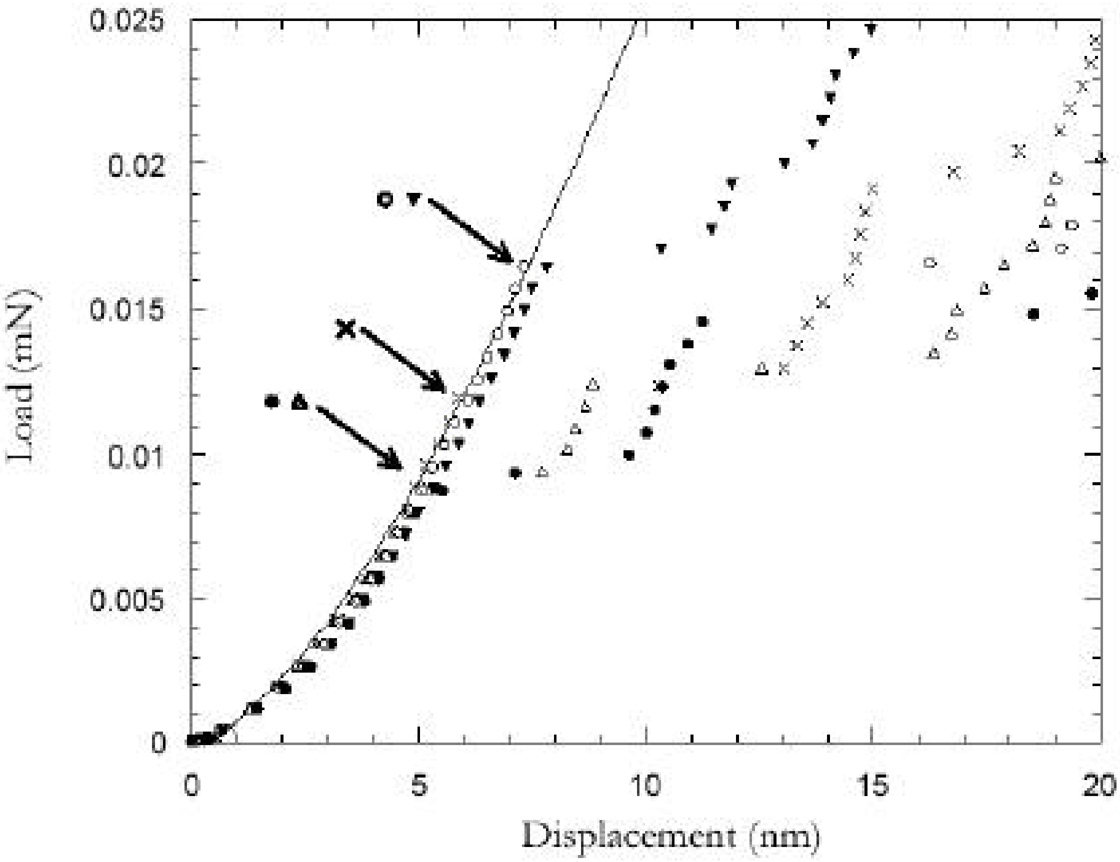

Fig. C.2 Load vs. displacement response for 5 individual indentations into the 160 nm (001) epitaxial Au film on Si, showing the pop-in behavior during the initial stages of loading. The Hertzian prediction (solid line) describes the elastic response extremely well. Indent to indent variation in the critical load at pop-in is observed.

The effect of microstructure on the initiation of plasticity is shown in Fig. C.3, where the load-displacement relations for both coarse grained and fine grained Au are compared. The indentation for the coarse grained sample of gold (film thickness of $1\mu m$) was made in the center of a large grain. We see elastic Hertzian loading followed by displacement bursts. But for the fine-grained sample (film thickness of $160nm$) the load displacement curve is smooth and continuous from the very beginning, indicating that plasticity is initiated at a very low contact stress. We believe this is caused by grain boundaries which are acting as prolific sources for dislocations. In the case of the fine-grained-film, the indentation curve is characterized by strain hardening while for the large grained sample strain softening occurs as soon as dislocations are nucleated.

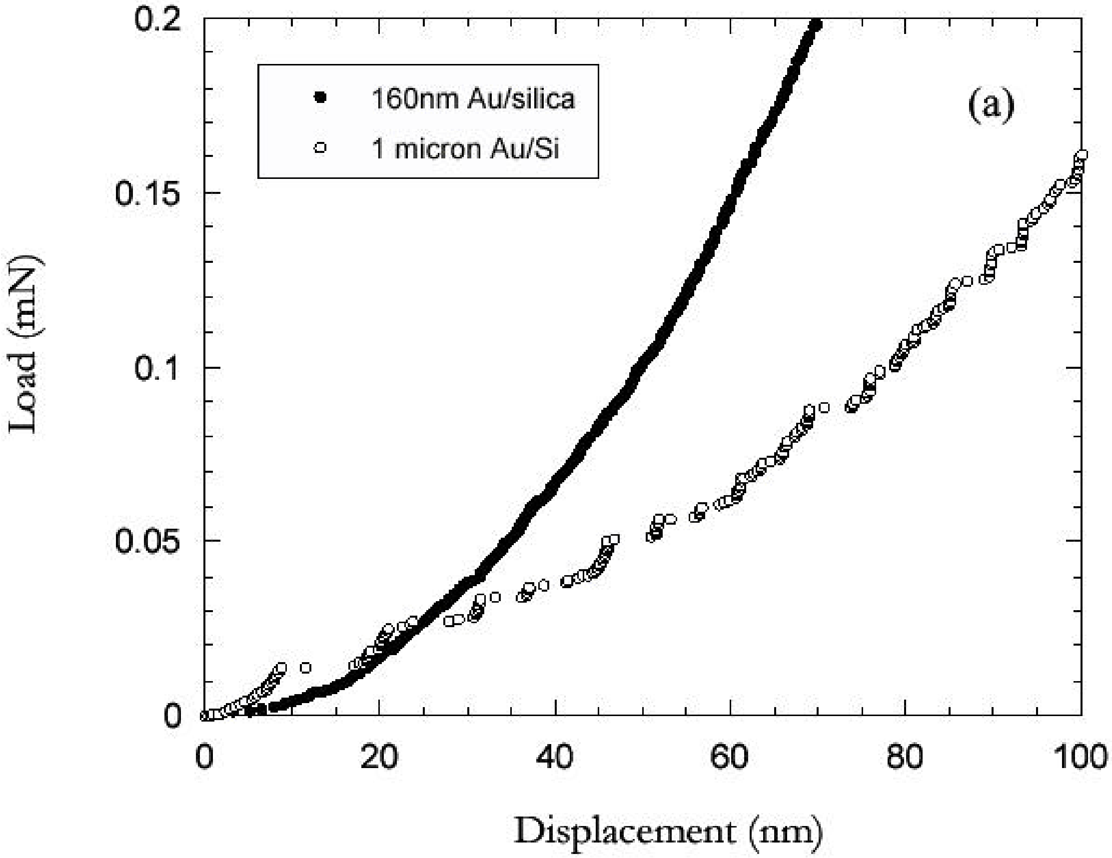

Fig. C.3 Load-displacement behavior revealing the difference between the coarse-grained 1 μm Au/Si sample (open circles) and the fine-grained 160 nm Au/silica(closed circles).

D. A microbeam bending method for studying stress-strain relations for metal thin films on silicon substrates

(Jeffrey N. Florando)

1. Introduction

The expanding field of Micro-Electrical Mechanical Systems (MEMS) has led to the development of many useful engineering devices [1-3]. The decreasing dimensions of these devices require the development of new test methods for monitoring the mechanical reliability of these systems. Many different kinds of tests are available for studying the mechanical properties of materials in small dimensions [4]; however, there is still need to develop simple test methods for measuring the isothermal stress-strain relations for thin films still attached to their substrates.

In an effort to devise a simple method for studying the elastic and plastic properties of thin films on substrates, a microbeam bending technique has been developed. The method is similar to previous work done on microbeam bending [5-6], except that triangular silicon microbeams are used. The triangular beam has the advantage that the entire film on the top surface of the beam is subjected to a uniform state of strain as the beam is deflected, unlike the standard rectangular geometry where the bending is concentrated at the support. We have fabricated rectangular and triangular silicon beams using micromachining and semiconductor processing techniques. Copper films are deposited on top of the Si beams, and the bi-layer beams are deflected using a nanoindenter. The sample geometry coupled with the high resolution of the nanoindenter allows this technique to have high strain resolution.

Typically, the onset of yielding is determined by the first deviation from linearity on the load-displacement curves; however, due to the relatively thick elastic Si substrate, the deviation in the load-displacement behavior is very gradual and small in magnitude. To extract the stress-strain behavior of the film from the load-displacement relation, a simple numerical model has been developed. The yielding behavior of the film can be modeled using a Ramberg-Osgood constitutive law for the film, which is then used to predict the stress-strain relation for the film while attached to its elastic substrate. This model has also been used to show that although there is a gradient of stress and strain through the thickness of the film during bending, this effect does not obscure the measurement of the yield stress of the film.

Utilizing this technique, the yielding and strain hardening behavior of bare Cu thin films have been investigated. A Cu film was thermally cycled from room temperature to 500^{o}C, and the film was tested after each cycle. The thermal cycles were performed to examine the effect of thermal processing on the stress-strain behavior of the film. Cu films with dual, <100>/<111>, textures, as well as others with strong <111> textures have also been studied. Strongly textured <111> films were deposited at three different film thicknesses to examine the effect of texture, as well as film thickness, on the yield properties of the film. The <111> textured films show very high rates of work hardening, an effect that has been predicted by recent modeling of thin film plasticity.

2. Theory

The advantage of the triangular beam is the constant moment per unit width acting in the beam during deflection. As shown in Fig. D.1, the bending moment at any point along the beam is the product of the load, *P* and the lever arm, *L-x*. Using similar triangles, the moment per unit width at any point in the beam can be expressed as:

$$M = \frac{M_t}{w} = \frac{P(L-x)}{w} = \frac{P(L-x)}{w_o\left(\frac{L-x}{L}\right)} = \frac{PL}{w_o} = constant. \quad (1)$$

Thus the triangular beam is subjected to a constant moment (per unit width) and the beam naturally develops a uniform curvature. As a consequence, the entire film on the top surface of the beam is subjected to a uniform state of plane strain and the film can be expected to yield simultaneously at all points along the beam, unlike the standard rectangular geometry where yielding first occurs at the support. In what follows we assume that a bi-layer triangular beam consisting of a metal film of uniform thickness on top of an elastic beam of uniform thickness is deflected at its end with a force P resulting in a displacement u. The response of the beam is assumed to be time independent so that any foreword running parameter, such as the load point deflection, can serve as time. The incremental load point deflection, δu, may then be regarded as a time derivative for a time-independent process. Corresponding increments in the load, δP, moment (per unit width), $\delta M = (L / w_o)\delta P$, and curvature, $\delta\kappa = \left(2 / L^2\right)\delta u$, similarly represent time derivatives for this time-independent bending process. Using the uniform strain state for the triangular geometry, the stress-strain properties of the film can be extracted using the analyses described below.

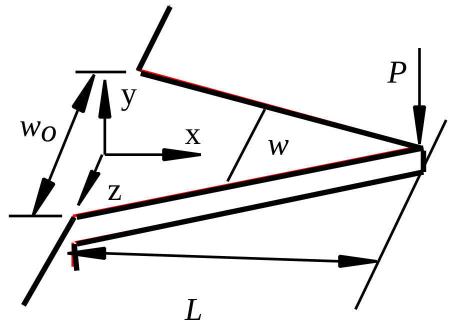

Fig. D.1 Top view schematic of a triangular microbeam.

2.1 Elastic-Plastic Analysis

We consider the deflection of a triangular microbeam of thickness t_s with a metal film of thickness t_f bonded to the top surface of the beam. The length of the beam is L and the width of the beam at its base is w_o. For a film on an elastic substrate with known elastic properties, the deformation properties of the film can be extracted from the bending response of the bi-layer beam. Silicon, which behaves in a linear elastic manner in these experiments, is used as the substrate material, so yielding is limited to the metal film. We first model the deformation behavior of the film using the empirical Ramberg-Osgood stress-strain law,

$$\bar{\varepsilon}^f = \frac{\bar{\sigma}^f}{E_f} + \frac{\sigma_o}{E_f}\left(\frac{\bar{\sigma}^f}{\sigma_o}\right)^m , \qquad (2)$$

where $\bar{\varepsilon}^f$ is the equivalent strain, $\bar{\sigma}^f$ is the equivalent stress, E_f is Young's modulus, σ_o is the uniaxial yield stress, and *1/m* is a strain hardening exponent. This model, in conjunction with elastic deformation of the substrate, can be used to extract the properties of the film (σ_o and *m*) from the experimental load-displacement data. To make this determination we first assume values for σ_o and *m* and calculate the incremental loads and displacements during bending. We choose those values of σ_o and *m* that provide the best fit to the experimental load-displacement data. For a given set of σ_o and *m,* the incremental bending loads can be related to the incremental bending displacements using

$$\delta P = \left(\frac{2w_o}{L^3}\right)\left\{\int_0^{t_s} B_s\left(y-y_o\right)^2 dy + \int_{t_s}^{t_s+t_f}\left[\left(1-K_2\nu_f\right) - \left(\frac{2\sigma_{xx}^f-\sigma_{zz}^f}{2\sigma_{zz}^f-\sigma_{xx}^f}\right)\left(K_2-\nu_f\right)\right]^{-1} E_f\left(y-y_o\right)^2 dy\right\}\delta u$$

(3)

where B_s is the plane strain modulus of the substrate and where the in-plane stresses in the film, σ^f_{xx} and σ^f_{zz}, are found for each increment of bending using

$$\delta\sigma^f_{xx} = \left[\left(1 - K_2 \nu_f\right) - \left(\frac{2\sigma^f_{xx} - \sigma^f_{zz}}{2\sigma^f_{zz} - \sigma^f_{xx}} \right) \left(K_2 - \nu_f\right) \right]^{-1} E_f \delta\varepsilon_{xx}, \tag{4}$$

and

$$\delta\sigma^f_{zz} = K_2 \delta\sigma^f_{xx}, \tag{5}$$

with the strain increment $\delta\varepsilon_{xx}$ given by

$$\delta\varepsilon_{xx}(y) = \delta\kappa\left(y - y_o\right), \tag{6}$$

where $\delta\kappa = \left(2 / L^2\right)\delta u$.

The parameter K_2 in these relations is found using

$$K_2 = \frac{\left(\nu_f \bar{\sigma}^f - K_1\left(2\sigma^f_{xx} - \sigma^f_{zz}\right)\right)}{\left(\bar{\sigma}^f + K_1\left(2\sigma^f_{zz} - \sigma^f_{xx}\right)\right)}, \tag{7}$$

where

$$K_1 = \frac{m}{2} \left(\frac{\sigma^f_{zz} - \frac{1}{2}\sigma^f_{xx}}{\bar{\sigma}^f} \right) \left(\frac{\bar{\sigma}^f}{\sigma_o} \right)^{m-1}. \tag{8}$$

Finally, the position for the neutral plane for bending, y_o, must be computed after each increment of bending by solving

$$0 = \delta M = \int_0^{t_s} B_s \left(y - y_o\right) dy + \int_{t_s}^{t_s + t_f} \left[\left(1 - K_2 \nu_f\right) - \left(\frac{2\sigma^f_{xx} - \sigma^f_{zz}}{2\sigma^f_{zz} - \sigma^f_{xx}} \right) \left(K_2 - \nu_f\right) \right]^{-1} E_f \left(y - y_o\right) dy \tag{9}$$

Utilizing eqn.(3), a theoretical load-displacement curve can be calculated for a film that follows the Ramberg-Osgood law. By changing the yield stress σ_o, and the strain hardening exponent

1/m, the theoretical load-displacement curve can be fitted to match the experimental data. A match of the model to experimental data is shown in Fig. D.2. The data used here is for a *1 μm* thick Al-1%Si film on a *4.2 μm* thick Si beam. The model matches the data well using a yield stress of 280 MPa and an m of 8.

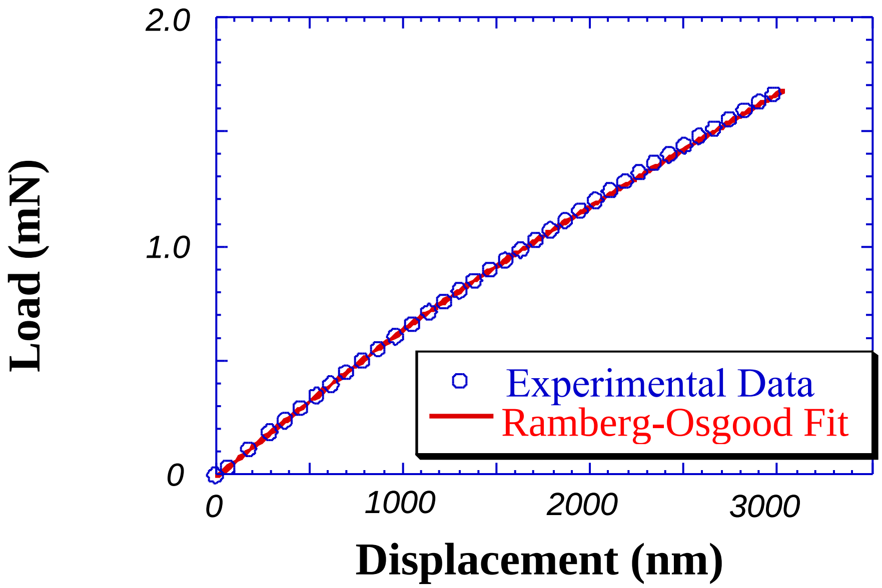

Fig. D.2 Matching the Ramberg-Osgood model to the experimental load-displacement data for a 1 μm Al-1%Si film on a Si substrate using a σ_o of 280 MPa, and an m of 8.

2.2 Average Stress Model

Although the Ramberg-Osgood model gives a good estimate of the yield stress and the work hardening exponent, it forces the film to behave in a Ramberg-Osgood manner. A more flexible description of the yielding behavior of the film can be found by using the following average stress and strain model. The average incremental stress in the film on a triangular beam can be expressed as

$$\delta < \sigma > = \frac{L\delta P_{film}}{w_o \int_{t_s}^{t_s+t_f} (y - y_o) dy} = \frac{2L\delta P_{film}}{w_o t_f \left[2t_s + t_f - 2y_o\right]}, \qquad (10)$$

where δP_{film} is the incremental load associated with the film alone, and y_o is the position of the neutral plane for bending. For the Ramberg-Osgood analysis described above, the neutral plane positions are continuously calculated as the film yields. Since this model is fitted to the experimental data, it can be used to provide very good estimates of the neutral plane positions during bending. Since the other quantities in eqn. (10), are experimentally determined, the

Ramberg-Osgood analysis is here used only to estimate the position of the neutral plane for bending. It does not constrain the film to follow the Ramberg-Osgood law.

The measured load, δP ,includes load contributions from the substrate. Thus δP_{film}, needed for the implementation of eqn.(10), is found using,

$$\delta P_{film} = \delta P - \delta P_{substrate}, \tag{11}$$

where $\delta P_{substrate}$ is calculated from the elastic properties and dimensions of the substrate and knowledge of the current position of the neutral plane,

$$\delta P_{substrate} = \frac{2w_0}{L^3} B_s \delta u \int_0^{t_s} \left(y - y_o\right)^2 dy = \frac{2w_0}{L^3} B_s \left[t_s^3 - 3t_s^2 y_o + 3t_s y_o^2\right] \delta u \,. \tag{12}$$

The average incremental strain in the film can also be expressed as

$$\delta < \varepsilon_{xx} >= \frac{2\delta u}{t_f L^2} \int_{ts}^{ts+tf} (y - y_o) dy = \frac{\delta u}{L^2}\left[2t_s + t_f - 2y_o\right]. \tag{13}$$

Thus, using eqns (10) and (13), the average stress and strain the film can be extracted directly from the beam bending experiment. These equations are mainly a function of the known geometry, and the experimental loads and displacements. The only unknowns are the neutral plane positions, which can be estimated using the Ramberg-Osgood analysis, as described above. Knowledge of the movement of the neutral plane position permits a calculation of the average stress and strain in the film. The average stress-strain relation obtained using this method is compared to the corresponding Ramberg-Osgood model in Fig. D.3. While there is good overall agreement, justifying the use of the Ramberg-Osgood model, the average stress-strain relation is considered more accurate as it is not constrained to follow a particular form.

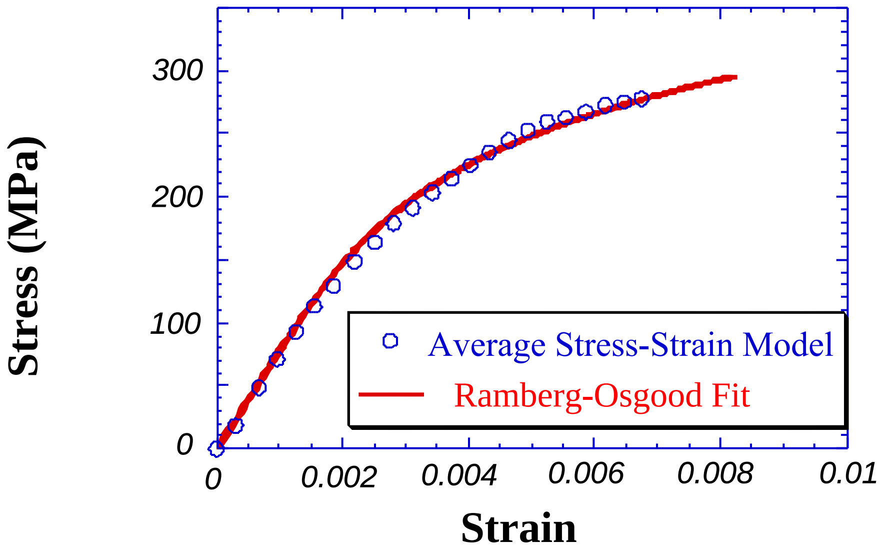

Fig. D.3. Comparison of the average stress and strain relation with the Ramberg-Osgood model. The 1 μm Al-1%Si film is deposited on a 30º 46 μm long triangular Si beam.

2.3 Verifying the average stress-strain approximation

The model for the average stress-strain relation assumes that there is no variation in stress through the thickness of the film, and treats the film as if it were subjected to a homogeneous stress state. However, in bending there is a linear variation in strain through the thickness of the film, with the highest strain occurring at the top of the film. Since the film will yield at the top of the film first and then through the thickness of the film, the average equations may be insensitive sharp features in the stress-strain relation. For example, in the beam bending experiment the load-displacement data will be inherently gradual even if the film exhibits a sharp yield point. Since the average stress and strain in the film is extracted from this load-displacement data, the stress strain behavior of the film would be predicted to be gradual as well, even if the film's "real" stress strain behavior has a sharp yield point. Therefore the gradual yielding shown in Fig. D.3 could be an effect of the bending experiment and the average stress and strain in the film. This potential error can be checked by assuming that the film behaves in a manner that will produce a very abrupt yield point, such as in the case of a film characterized by an elastic-linear strain hardening law. Assuming that the film behaves in this manner, a theoretical load-displacement relationship can be derived, using expressions similar to eqn. (3). The calculated loads and displacements are then treated as "data" and inserted into the average stress-strain equations to determine if the original linear-hardening law can be recovered. Figure D.4 shows that the mean stress-strain equations can reproduce the original linear hardening curve quite well. Therefore, the approximation of treating the film as having a uniform stress state through the thickness of the film is valid. All stress-strain plots throughout the remainder of this report use average stress-strain equations to determine the stress-strain behavior of the film.

3. Description of testing method

Representative Si microbeams of the kind used in this research are shown in Fig. D.5. The processing steps needed to create these structures will not be given here. This information can be found in the Ph.D. dissertation by J.N. Florando [7] and will appear in a forthcoming paper. Both Al and Cu metal films were sputter deposited on top of the beams and subsequently tested. The microbeams were deflected using the Nano II nanoindenter from the MTS Nano Innovation Center. This particular instrument has a load resolution of 10 nN, and a displacement resolution of 0.3 nm, which allows the microbeam bending technique to have a strain resolution on the order of 0.1 microstrain. Instead of using the traditional point load, a line load was applied to the beams using a diamond wedge tip that is 10 μm long and has a 90^{o}-included angle. The line load was used to reduce the torsional bending of the beam.

As seen in Fig. D.5, the triangular beams have a rectangular pad at the end to provide a location for the nanoindenter to bend the beam. This pad makes the beam stiffer than the ideal triangular geometry. An analysis performed to account for this effect [7] shows that by using sufficiently long beams (60 μm or longer), the results converge to the ideal triangular geometry. The triangular beams used in this paper are longer than 60 μm and were approximated as beams with an ideal triangular geometry. The beams were loaded at a constant displacement rate, which is equivalent to a constant strain rate, held for 100 seconds at the maximum load, and then unloaded at the same displacement rate as the loading segment. All the beam tests were performed at a strain rate on the order of 10^{-5}/sec.

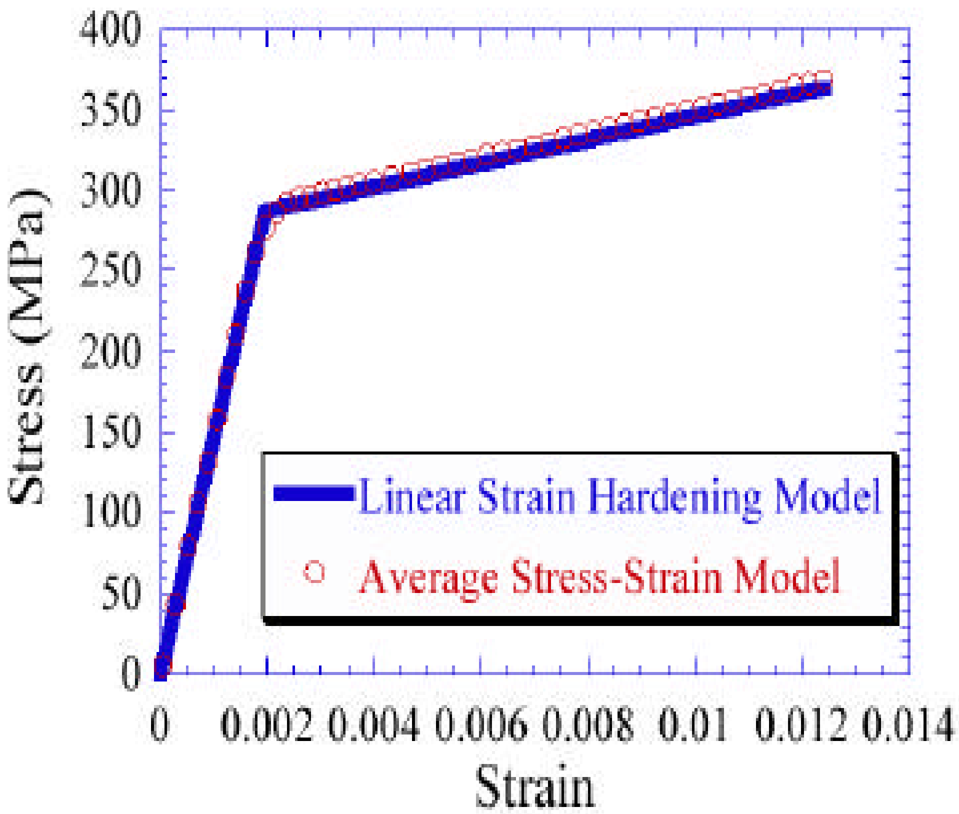

Fig. D.4. Stress-Strain plots for a theoretical "film" that follows a linear hardening model, comparing the average stress-strain equations to the stress-strain law.

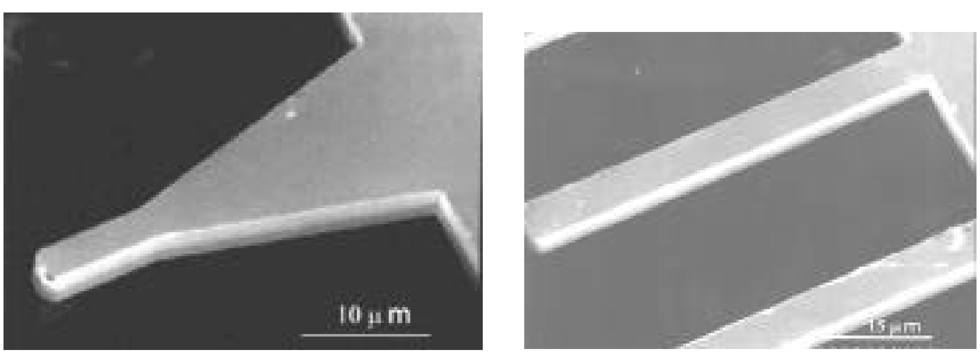

Fig. D.5. SEM picture of Si microbeams.

Since a sharp indenter tip is used to deflect the beams, the tip makes an impression in the surface of the film as the beam is deflected. The indentation displacements can be removed by assuming that for a given load the indentation displacement into the film will be the same as the displacement in the film where it is well-supported by the substrate. This indentation response is then subtracted from the experimental displacement data to obtain the displacement of the beam. The compliance of the support also adds additional displacements to the end of the beam that must be taken into account. The support compliance is calculated using the measured support compliances for rectangular beams [6,8] and the assumption that the support compliance varies linearly with the width of the base of the beam. Knowledge of the support compliance, the length of the beam, and the load allows for the displacements at the end of the beam due to the support compliance to be calculated. These calculated displacements are then subtracted from the experimental data to obtain the beam displacements due to bending.

4. Film Characterization

The grain sizes for the Cu films grown on SiO_2 and Ta were measured using Focused Ion Beam (FIB) images and the Heyn intercept method. The average grain size for the *1* μ*m* thick as-deposited film grown on SiO_2 was measured to be 0.75 μm. To study the effect of grain size, the as-deposited film was first tested, annealed in vacuum at 500°C, and tested again. After annealing, the grain size was measured to be *1.0* μ*m*. For the Cu films grown on Ta, the average grain size was *0.2* μ*m* for the *0.5* μ*m* thick film, *0.4* μ*m* for the *1.0* μ*m* thick film, and *0.5* μ*m* for the *1.7* μ*m* thick film. Symmetric x-ray diffraction was used to determine the texture of the films. The results of the scans are shown in Fig D.6. The film grown on a SiO_2 layer (dash line) shows two peaks, which correspond to the (111) and the (200) reflections. The film grown on Ta (solid line) shows a strong (111) peak, and no observable (200) peaks.

The residual stress in the dual textured Cu film was measured using asymmetric x-ray diffraction and the $\sin^2 \psi$ method [9]. For the <111> textured Cu films, the residual stress was measured using the wafer curvature method. These methods, however, give the residual stress in the film where it is supported by the massive substrate. For the bi-layer beam, the relatively thin substrate layer will accommodate some of the misfit strain. The residual stress in the film on the beams can then be calculated using the results from these techniques, and the assumption that the total misfit strain is the same on the beam as on the well-supported regions. Using this assumption, the amount of misfit strain accommodated by the substrate can be determined, and the residual stress in the film on the beams can be calculated.

5. Results and Discussion

5.1 Grain size effect

Figure D.7 shows a plot of the stress-strain behavior determined for both the as-deposited and annealed film with the dual texture. The annealed film has a higher residual tension stress, which is expected since the film deforms plastically during heating, and this deformation causes an increase in the residual tension stress at room temperature. Even though the annealed film was initially in a higher state of biaxial tension, it yielded at a lower stress than the as-deposited film. This behavior is expected since the yield stress is expected to vary inversely with the square root of the grain size.

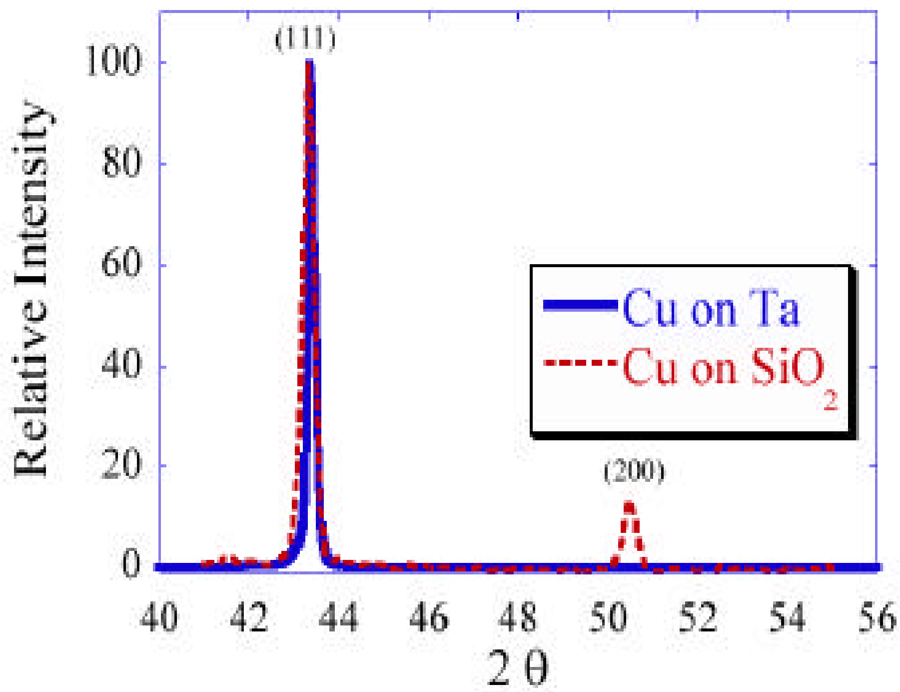

Fig. D.6. Symmetric x-ray diffraction scans for a Cu film deposited on SiO_2 (dashed) and Ta (solid line) showing a dual <111>/<200> texture for the film grown on SiO_2 and a strong <111> texture for the film grown on Ta.

5.2 Effect of Film Thickness

To study the effect of film thickness, two additional Cu films with strong <111> texture, in addition to the *1.0* μ*m* film, were deposited. The two additional films have a thickness of *0.5* μ*m*, and *1.7* μ*m*. Fig. D.8 shows the stress-strain behavior for the three film thicknesses. It should be noted that these stress-strain curves are reproducible, and the data shown here are representative of the samples.

Since the texture should be similar for all three films, the elastic loading slope should be equal. The slope for the *1.7* μ*m* thick film is a little lower than for the other two films, but the difference is within the range of experimental variation. The yield stress for the three films is different, with the thinnest film having the highest yield stress. This result is expected as Freund [10] and Nix [4] have shown that the yield stress is inversely related to the film thickness. The grain size measurements for the three films shows that the thinnest film has the smallest average grain size. Since the smaller grain size can also contribute to the increase in the flow stress, is it difficult to separate the effect of film thickness and grain size in these experiments.

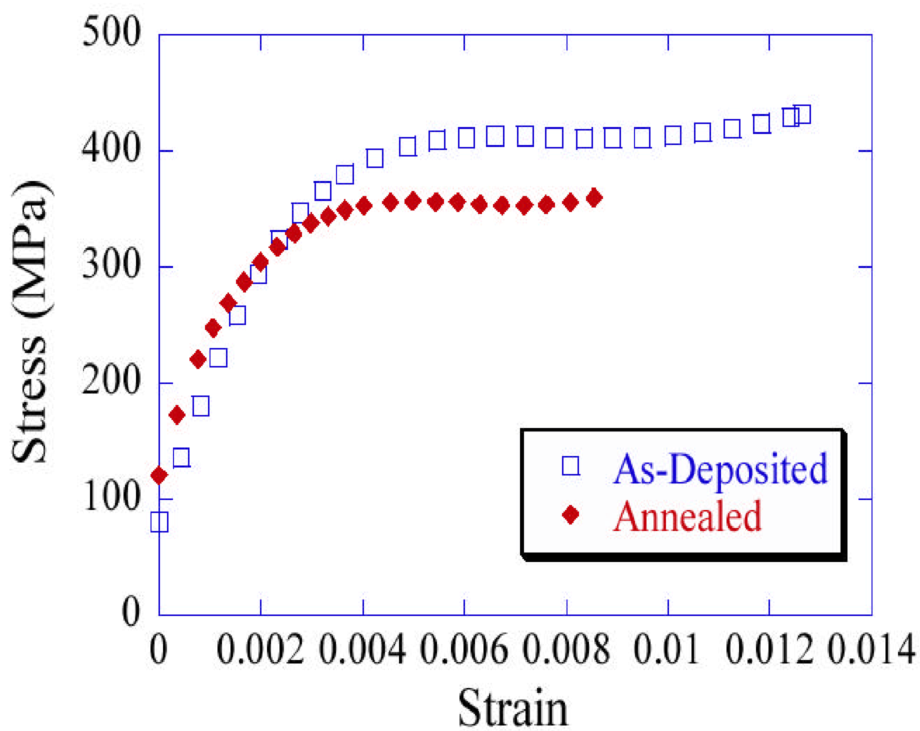

Fig. D.7 Stress-strain behavior for an as-deposited and annealed Cu film.

The most striking feature in Fig. D.8, however, is the large difference in the work hardening rates between the three films. The *1.7* μ*m* thick film has a linear strain-hardening rate of about E/ 9, while the *1.0* μ*m* film has a hardening rate of E/4. The *0.5* μ*m* thick film has an initial hardening rate for E/2.2, but the slope increases to a hardening slope of E/1.3 at a strain of about 0.006. The systematic increase in the strain hardening rates for decreasing film thickness, as well as the bi-linear strain hardening behavior seen in the *0.5* μ*m* thick film, has been predicted by Nicola, Van der Giessen, and Needleman [11] using a 2-D plane strain dislocation simulation. In their simulation, dislocations pile up near the film-substrate interface, which forms a boundary layer that has a much higher dislocation density and in-plane stress than the rest of the film. This boundary layer acts as an obstacle for other dislocations to move past, and the size of the boundary layer is independent of film thickness. Therefore, the thinner the film, the larger the effect of the boundary layer. This type of argument leads to an increase in the strain hardening rates for decreasing film thickness. For films that are sufficiently thin, as the film yields and more dislocations pile up at the interface, there develops a large enough back-stress to prevent the nucleation of new mobile dislocations. This effect leads to a secondary hardening effect, and an increase in the slope of strain hardening rate or bi-linear strain hardening, similar to the effect seen in our thinnest textured copper films.

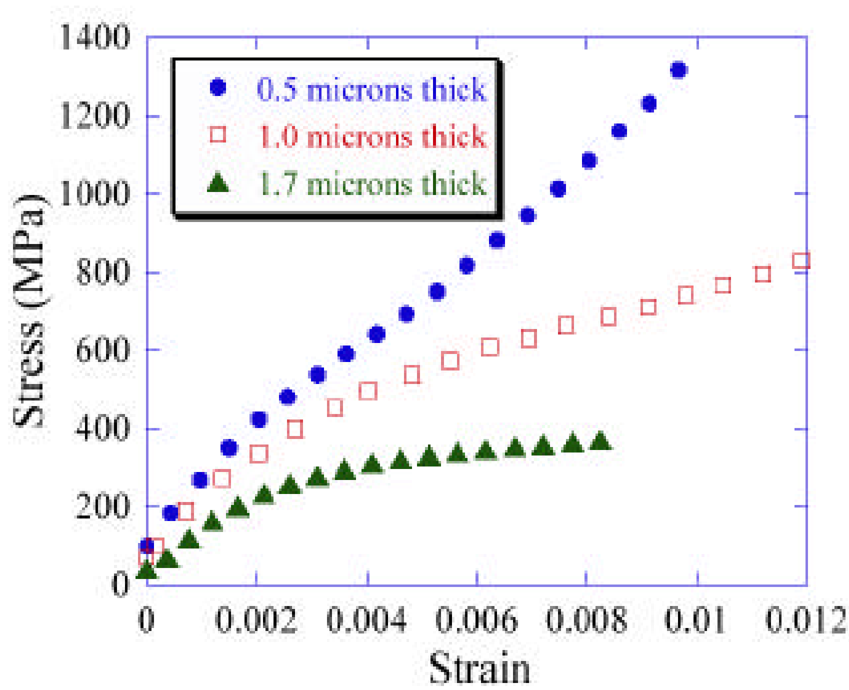

Fig. D.8 Stress-strain behavior for <111> textured Cu films of three different film thicknesses.

References

2. E.J. Garcia, and J.J. Sniegowski, "Surface Micromachined Microengine," Sensors and Actuators A-Physical, **48**(3): 203-214, (1995).

3. D. DiLella, L.J. Whitman, R.J. Colton, T.W. Kenny, W.J. Kaiser, E.C. Vote, J.A. Podosek, and L.M. Miller, "A micromachined magnetic-field sensor based on an electron tunneling displacement transducer," Sensors and Actuators A-Physical, **86**(1-2): 8-20, (2000).

4. S.M. Spearing, "Materials issues in microelectromechanical systems (MEMS)," Acta Materialia **48**(1): 179-196, (2000).

5. W.D. Nix, "Mechanical-Properties of Thin-Films," Metallurgical Transactions A-Physical Metallurgy and Materials Science **20**(11): 2217-2245, (1989).

6. T.P. Weihs, S. Hong, S., J.C. Bravman, and W.D. Nix, "Mechanical Deflection of Cantilever Microbeams : a New Technique For Testing the Mechanical-Properties of Thin-Films," Journal of Materials Research , **3**(5): 931-942, (1988).

7. S.P. Baker and W.D. Nix, "Mechanical-Properties of Compositionally Modulated Au-Ni Thin-Films : Nanoindentation and Microcantilever Deflection Experiments," Journal of Materials Research , **9**(12): 3131-3145, (1994).

8. J.N. Florando, "Measurement of the Mechanical Properties of Copper Thin Films by Microbeam Bending," Ph.D. Dissertation, Stanford University, (2003).

9. J. Florando, H. Fujimoto, Q. Ma, O. Kraft, R. Schwaiger and W.D. Nix, "Measurement of thin film mechanical properties by microbeam bending," Materials Reliability in Microelectronics IX., Warrendale, PA, Mater. Res. Soc. Symp. Proc. (1999).

10. B.M. Clemens and J.A. Bain, "Stress Determination in Textured Thin-Films Using X-Ray-Diffraction," MRS Bulletin, **17**(7): 46-51, (1992).

11. L.B. Freund, "The Stability of a Dislocation Threading a Strained Layer On a Substrate," Journal of Applied Mechanics-Transactions of the ASME, **54**(3): 553-557, (1987).

12. L. Nicola, E. Van der Giessen and A. Needleman, "2D dislocation dynamics in thin metal layers," Materials Science and Engineering A - Structural Materials, **309**, 274-277, (2003).

E. Studies of dynamic mechanical properties of metallic thin films on substrates

(Dae-han Choi)

1. Introduction

Materials used in microelectromechanical systems (MEMS) and electronic devices often take the form of thin metal films on rigid substrates; some of these structures are subjected to dynamic loading, where internal friction and damping may be important. Many testing methods have been developed to investigate the mechanical properties of these structures and extensive studies have been performed in many research groups [1]. However, the study of strength and plasticity of thin metal films on substrates is dominated by quasi-static and athermal models. Hence, our understanding of plasticity and other inelastic properties of thin films has been limited by the absence of information about the dynamic mechanical properties of these materials.

A new dynamic measurement system has been developed to investigate damping in thin metal films. This system includes a vacuum chamber, in which a free-standing bilayer cantilever sample is vibrated using an electrostatic force, and a laser interferometer to measure the displacement and velocity of the sample. With this equipment, internal friction as low as 10^{-5} in micrometer thick metal films in a temperature range of 300K to 750K can be measured. Free-standing cantilevers with different frequencies have been fabricated using well established IC fabrication processes. The cantilevers consist of thin metal films on thicker Si substrates, which exhibit low damping.

2. Measurement System

A schematic of the measurement system developed to study dynamic mechanical properties of metal thin films on substrates is shown in Fig. E.1. This measurement system consists of a vacuum chamber with a temperature-controlled heater, a laser interferometer to measure the free-decay of vibration and a data-acquisition part to record the data.

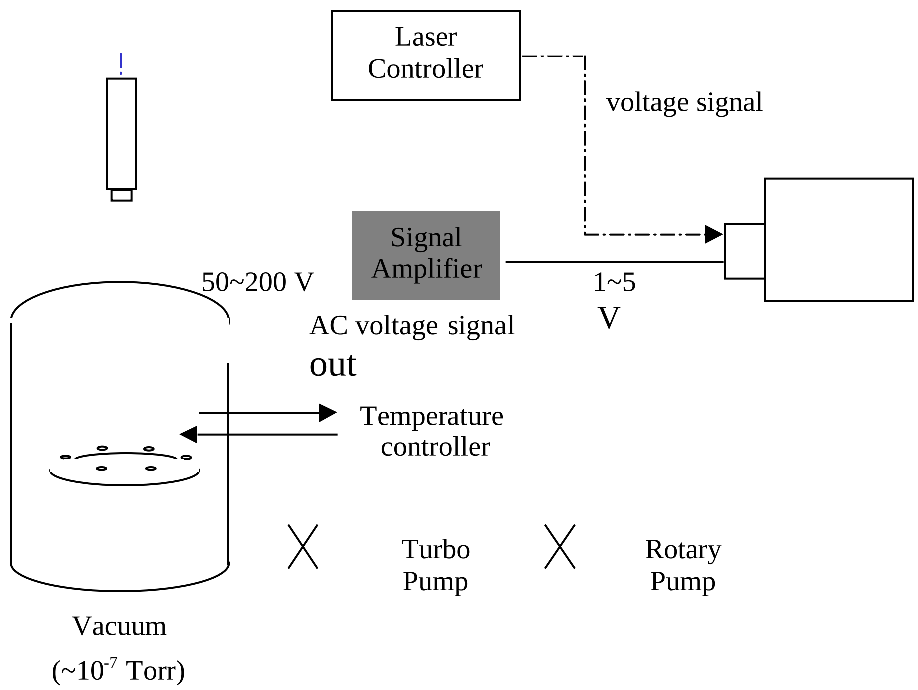

Fig. E.1. A schematic of the dynamic mechanical testing system.

A vacuum system was necessary to measure damping in the film; otherwise, air damping would prevail and the small damping in the film would be washed out. Thus, a vacuum system with a base pressure of about 5×10^{-7} torr was built. When the temperature inside the chamber increases, the pressure also increases but is never higher than 10^{-5} torr. Samples are placed on a stage within the chamber where the temperature is controlled by a heater in the temperature range from room temperature to 800K.

Each cantilever sample on the stage is driven to vibrate by an electrostatic force applied by an AC voltage of approximately 100V. This signal is generated by a data acquisition board and amplified with a current limited amplifier. Since the electrostatic force produced by the potential difference is very small, the frequency of an AC signal is controlled to match with the natural frequency of the cantilever sample to maximize displacement.

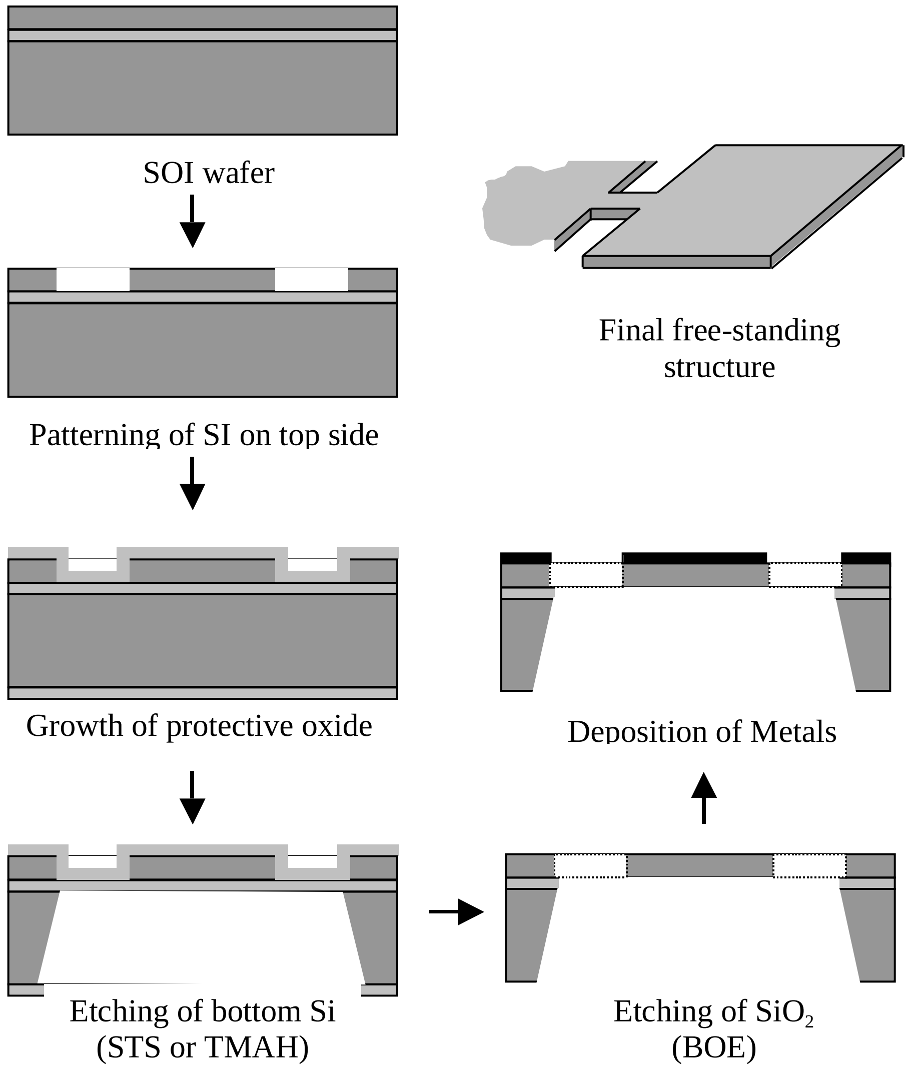

Fig. E.2. Processing steps to fabricate a free-standing cantilever.

After the voltage signal is turned off, the free decay of vibration is measured and recorded with a Polytec PI™ laser Doppler vibrometer pointed at the sample through an anti-reflection coated view-port from outside the chamber. This device is capable of measuring displacements as small as 0.5 μm/V with a resolution of 0.002 μm and velocities as low as 5mm/s/V with a resolution of 0.5 μm/s. According to our measurements, the vibration amplitude is on the order of 1 μm and the maximum strain imposed by this displacement is less than 10^{-5}, ensuring that the film does not experience any permanent deformation.

With this system, we can measure damping at various temperatures for samples with different natural frequencies. The natural frequency of each sample was determined from its geometry, mainly the size of a plate at the end of the cantilever. Thus, we can study the dependence of damping on temperature and frequency. We also can study the dependence of damping on the maximum strain imposed on films by changing the driving voltage.

3. Sample Fabrication

Micromachining techniques have been used to fabricate free-standing cantilevers for the study of anelasticity. A schematic diagram of the processing steps to fabricate bi-layer cantilevers is shown in Fig. E.2. A bonded silicon-on-insulator (SOI) wafer was used to make free-standing cantilevers. The SOI wafer consists of a 500 μm bottom Si layer, a 1 μm middle oxide layer and an 80 μm top Si layer. The top Si layers were patterned into cantilevers using standard photolithography processes and a deep reactive ion etcher (DRIE). The cantilevers have large plates at the free ends to maximize the electrostatic force applied by AC voltage. The straight sides of the mask pattern were aligned parallel to the <110> direction of the substrate to achieve sharp corners. The high selectivity of a DRIE between Si and SiO_2 made it possible to use the middle oxide layer as an etch stop. After patterning the top Si layers, wet-thermal SiO_2 layers with a thickness of 0.5 μm were grown on both sides of the wafers to protect the patterned cantilevers on the front side and to make etch windows for later processing steps on the back side. In order to release the cantilevers, etch windows were patterned into the back SiO_2 using standard lithography techniques and dry plasma etching. Once windows were etched into the backside oxide layers, the wafers were cut into 1.5 mm by 1.5 mm pieces so that each piece had one cantilever. These pieces were then submerged into an anisotropic etchant for the Si, a 25% tetramethyl ammonium hydroxide (TMAH) solution at 80 °C. The etch rate for silicon in TMAH is approximately 25 μm/hour at 80 °C. In addition, TMAH has a high selectivity with the oxide (~2000:1), so the middle oxide layer is used as an etch stop. After about 20 hours, the bottom Si layer was completely etched through from the backside. At the end of this etching process, the middle oxide layer remained intact and could be removed using a Buffered Oxide Etcher (BOE), after which free-standing cantilevers were obtained. The final shape and dimension of a sample are also illustrated in Fig. E.2.

Metal films were deposited onto the Si cantilevers by magnetron sputter deposition in order to produce structures of thin metal films on Si substrates. The films were deposited at room temperature with a base pressure of 10^{-8} torr and the Ar pressure was maintained at 3 mTorr during deposition. The samples were then thermally cycled from room temperature to 500 °C in vacuum of 10^{-7} torr in order to stabilize the grain structures in the films.

4. Research Progress

Aluminum films were chosen for the first study with a dynamic mechanical testing system. The anelastic properties of Al thin films have been studied by several groups and there is general agreement on the atomistic mechanism for this behavior [2-4]. Therefore, the internal friction measurement of Al thin films helped us verify that our measurement system worked properly and gave a consistent result. Moreover, there had been few efforts to develop a rigorous model to explain this mechanism and this measurement helped us develop a physically sensible model.

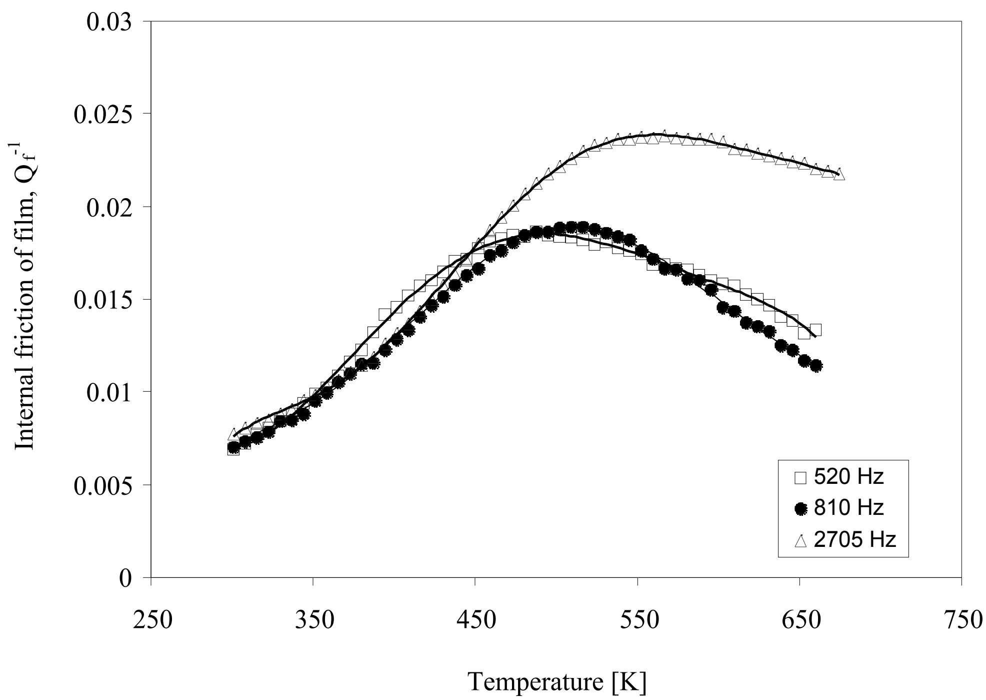

Fig. E.3. Internal friction of 2 μm Al films.

Damping of a thin Al film on a thicker Si substrate was measured and the internal friction of the film was extracted. Samples with three different frequencies were used and the measurement data for each sample were plotted in Fig. E.3. The activation energy calculated

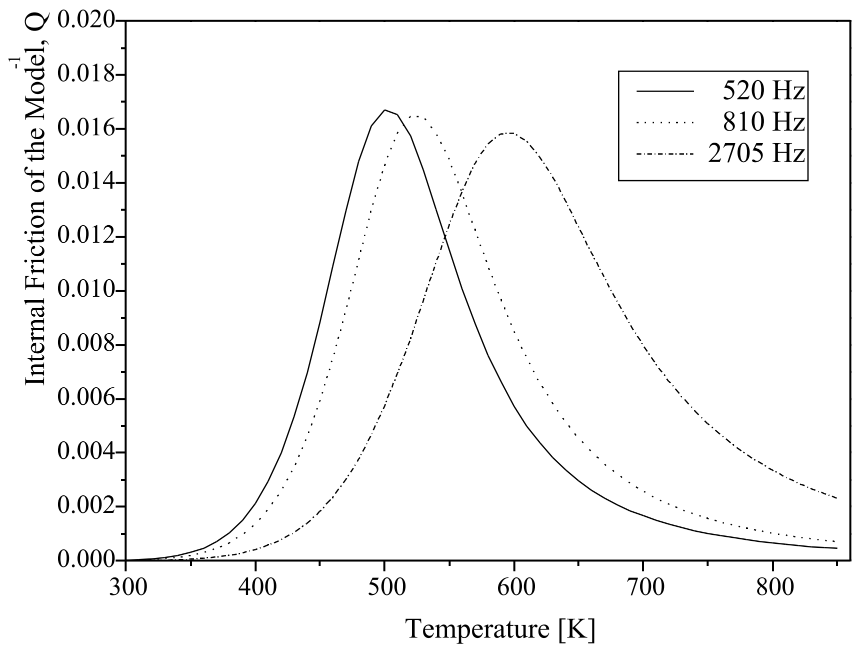

Fig. E.4. A plot of internal friction derived from the model.

from the damping data was 0.57eV, which is consistent with previous research [2-4]. This value suggested that the mechanism of internal friction in pure Al films involve grain boundary diffusion controlled grain boundary sliding. A model to describe these damping effects was developed. By deriving an expression for the diffusional strain rate using a two-dimensional Coble creep model, and modifying the conventional standard linear solid model for the case of bending, it was possible to give a good account of the observed damping. The model developed showed good agreement with experimental data. A plot of internal friction derived from the model is shown in Fig. E.4.

5. Future Plans

It is proven that our measurement system gives reliable information on the dynamic mechanical properties in thin metal films. Many different mechanisms for anelastic behavior have been reported in various bulk materials [5, 6]. In the same way, we expect there will be many different causes for the anelastic behavior of thin metal films; one of these causes was studied using Al thin films. Now, we plan to explore various alloy systems to investigate different mechanisms and to develop models for each mechanism observed. These material systems include pure metals such as Cu and Au, as well as alloys. We hope that future studies of anelasticity might benefit from our developed testing system and that eventually a more complete understanding of the mechanical properties of thin metal films is achieved from this study.

References

1. W.D. Nix, *Metall. Trans.*, **20A**, 2217 (1989)

2. B.S. Berry, "Anelastic Relaxation and Diffusion in Thin Layer Materials", in Diffusion Phenomena in Thin Films and Microelectronic Materials, D. Gupta and P.S. Ho eds., Noyes, Park Ridge, NJ, 73 (1988)

3. M. Prieler, H.G. Bohn, W. Schilling and H. Trinkaus, *J. Alloys and Compounds*, **211/212**, 424 (1994)

4. B.S. Berry and W.C. Pritchet, *J. de Physique*, **42**, C5-1111 (1981)

5. C.M. Zener, "Elasticity and Anelasticity of Metals", Univ. of Chicago Press, Chicago (1960)

6. A.S. Nowick and B.S. Berry, "Anelastic Relaxation in Crystalline solids", Academic Press, N.Y., (1972)

III. Ph.D Dissertations Supported by DOE Grant No. DE-FG03-89ER45387

20. Martha Kennedy Small (USA) 1992
"Use of the Bulge Test in Measuring the Mechanical Properties of Thin Films"

Title: Staff Scientist
Organization: Hewlett Packard
Location: Colorado Springs, CO

21. Shefford Paul Baker (USA) 1993
"Understanding Depth-Sensing Indentation Experiments and Mechanical Properties of Compositionally Modulated Au-Ni Thin Films"

Title: Associate Professor
Organization: Department of Materials Science and Engr., Cornell University
Location: Ithaca, New York

22. Joost J. Vlassak (Belgium) 1994
"New Experimental Techniques and Analysis Methods for the Study of Materials in Small Dimensions"

Title: Assistant Professor
Organization: Harvard University
Location: Cambridge, MA

23. Robert J. Hohlfelder (USA) 1998
“Bulge and Blister Testing of Thin Films and their Interfaces”

Title: Staff Scientist
Organization: Sandia National Laboratories
Location: Albuquerque, NM

24. Omar S. Leung (USA) 2001
“Studies in Strengthening Mechanisms of Thin Gold Films”

Title: Staff Scientist
Organization: Silicon Light Machines
Location: Sunnyvale, CA

25. **Ranjana Saha** (India) 2001
"Determination of Intrinsic Film Properties from Nanoindentation of Film/Substrate Composites"

Title: Research Engineer
Organization: Applied Materials
Location: Sunnyvale, CA

26. **Erica Lilleodden** (USA) 2001
"Indentation-Induced Plasticity in Gold Thin Films"

Title: Post Doctoral Researcher
Organization: Lawrence Berkeley Laboratory
Location: Berkeley, CA

27. **Jeffrey Neilson Florando** (USA) 2002
"Measurement of the Mechanical Properties of Cu Thin Films by Microbeam Bending"

Title: Research Engineer
Organization: Lawrence Livermore Nationa Laboratory
Location: Livermore, CA

28. **Mark Andrew Phillips** (Australia) 2002
"Microstructure and Mechanical Properties of Al/Al_3Sc Metallic Multilayers - Deformation Mechanisms at the Nanoscale"

Title: Research Engineer
Organization: Intel Corporation
Location: Santa Clara, CA

29. **Dae-Han Choi** (Korea) 2004
"Dynamic Mechanical Properties of Metal Thin Films on Si Substrates"

Title: Research Engineer
Organization: Lam Research
Location: Miliptas, CA

IV. Oral Presentations Resulting from DOE Grant No. DE-FG03-89ER45387

1. A.F. Jankowski, Lawrence Livermore National Laboratory, Livermore, CA; S.P. Baker, W.D. Nix, Department of Materials Science and Engineering, Stanford University, Stanford, CA; K.A. Green and S.R. Nutt, Brown University, Providence R.I., “Gold-Nickel Multilayer Films: Structure-Property Correlations”, Symposium C, Thin Films: Stresses and Mechanical Properties, 1988 Fall Meeting of MRS, Boston MA, November 1988.

2. S.P. Baker, W.D. Nix, S. Hong and A.F. Jankowski, "Mechanical Properties of Compositionally Modulated Au-Ni Thin Films Using Indentation and Microbeam Deflection Techniques", Symposium on Thin Films: Stresses and Mechanical Properties, Spring Meeting of the Materials Research Society, San Francisco, April 16, 1990.

3. S.P. Baker and W.D. Nix, "Mechanical Properties of Thin Films on Substrates", Symposium on Optical Thin Films New Developments, SPIE - The International Society for Optical Engineering, San Diego, CA, July 9-11, 1990.

4. W.D. Nix, "Mechanical Properties of Thin Films", Special Foreigner's Lecture, Japan Institute of Metals, Tohoku University, Japan, September 24, 1990.

5. J.J. Vlassak, T. Nakayama and W.D. Nix, "Structure and Mechanical Properties of Fe/Zr Multilayers" Symposium on Thin Films: Stresses and Mechanical Properties III, Fall Meeting of the Materials Research Society, Boston, MA, December 2-6, 1991.

6. M.K. Small, J. Vlassak and W.D. Nix, "Re-Examining the Bulge Test: Methods for Improving Accuracy and Reliability" Symposium on Thin Films: Stresses and Mechanical Properties III, Fall Meeting of the Materials Research Society, Boston, MA, December 2-6, 1991.

7. S.P. Baker and W.D. Nix, "Time-Dependent Deformation in Room-Temperature Indentation Experiments using a Nanoindenter" Symposium on Thin Films: Stresses and Mechanical Properties III, Fall Meeting of the Materials Research Society, Boston, MA, December 2-6, 1991.

8. W.D. Nix, "Mechanical Properties of Thin Films", Symposium X, Frontiers of Materials Research, Spring Meeting of Materials Research Society, San Francisco, CA, May 1, 1992.

9. S.P. Baker, M.K. Small, J.J. Vlassak, B.J. Daniels and W.D. Nix, "The Search for the Supermodulus Effect", NATO Advanced Study Institute: "Mechanical Properties and Deformation Behavior of Materials Having Ultra Fine Microstructures", Praia do Porto Novo, Portugal, June 29 - July 10, 1992.

10. W.D. Nix, "Analysis and Development of New Bulge Testing Techniques for Studying Mechanical Properties of Thin Films", Department of Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan, December 4, 1992.

11. W.D. Nix, M.K. Small and J. Vlassak, "Analysis and Development of New Bulge Testing Techniques for Studying Mechanical Properties of Thin Films", Symposium on This Films: Stresses and Mechanical Properties IV, Spring Meeting of Materials Research Society, San Francisco, CA, April 12-16, 1993.

12. M.K. Small, J.J. Vlassak, S.F. Powell, B.J. Daniels and W.D. Nix, "Accuracy and Reliability of Bulge Test Experiments", Symposium on This Films: Stresses and Mechanical Properties IV, Spring Meeting of Materials Research Society, San Francisco, CA, April 12-16, 1993.

13. J.J. Vlassak and W.D. Nix, "The Indentation Modulus of Elastically Anisotropic Half Spaces", Symposium on This Films: Stresses and Mechanical Properties IV, Spring Meeting of Materials Research Society, San Francisco, CA, April 12-16, 1993.

14. W.D. Nix, "Mechanical Properties of Thin Films", Workshop on Mechanical Behavior of Thin Films for Optics and Microelectronics Reliability", The Aerospace Corporation, Los Angeles, CA October 13-14, 1993.

15. J.J. Vlassak, "Advances in Measuring the Mechanical Properties of Fine Structures Using Nanoindentation", Max Planck Institut fur Metallforshung, University of Stuttgart, Germany, October 22, 1993.

16. W.D. Nix, "Plasticity and Flow Stresses in Aluminum Thin Films on Silicon", Symposium on: Thin Film Stress - Measurement and Applications, Tencor Instruments., Palo Alto, CA, May 16, 1994.

17. W.D. Nix, "Models for the Generation of Intrinsic Stresses in Thin Films", DSRC Workshop on Intrinsic Stresses in Thin Films and Layered Structures, La Jolla, CA, July 27, 1994.

18. W.D. Nix, Mechanical Properties of Thin Films, Charles S. Barrett Silver Medal Lecture, Rocky Mountain Chapter of ASM International, Golden CO, November 3, 1994.

19. W.D. Nix, "Mechanical Properties of Thin Films", Department Colloquium, Department of Mechanical Engineering, U.C. Irvine, Irvine CA, November 11, 1994.

20. G.R. English, Glenn F. Simenson, Bruce M. Clemens and William D. Nix, "Mechanical Properties of Ag/Cr Multilayered Epitaxial Thin Films", Thin Films: Stresses and Mechanical Properties V., Fall Meeting of the Materials Research Society, Boston, MA, November 28-December 2, 1994.

21. V.M. Paviot, J.J. Vlassak and W. D. Nix, "Measuring the Mechanical Properties of Thin Metal Films by Means of Bulge Testing of Micromachined Windows," Thin Films:

Stresses and Mechanical Properties V., Fall Meeting of the Materials Research Society, Boston, MA, November 28-December 2, 1994.

22. Robert J. Hohlfelder, Joost J. Vlassak, William D. Nix, Huihong Liu and Christopher E.D. Chidsey, "Blister Test Analysis Methods," Fall Meeting of the Materials Research Society, Boston, MA, November 28-December 2, 1994.

23. W.D. Nix, J. J. Vlassak, G.R. English, R.J. Hohlfelder and S.L. Neumann, "Techniques for the Study of Mechanical Properties of Thin Films", Symposium on Microstructural and Mechanical Properties of Thin Films and Interfaces: Frontiers and Future Directions, Stanford University, February 22, 1995.

24. W.D. Nix, J.J. Vlassak, R.J. Hohlfelder and J.T. Sizemore, "Measurement of Adhesion of Diamond Thin Films to Substrates: Blister Testing Techniques, Mechanical Behavior of Diamond and Other Forms of Carbon, Spring Meeting of Materials Research Society, April 18, 1995.

25. W.D. Nix, J.J. Vlassak, S.L Neumann, R.J. Hohlfelder and J. Sizemore, "Bulge and Blister Testing of Thin films and Interfaces", Third International Workshop on Stress-Induced Phenomena in Metallization, Stanford University, June 21, 1995.

26. W.D. Nix, B.M. Clemens, B. J. Daniels, G.R. English and G. Simenson, "Mechanical Properties of Multilayers", Micromechanics of Layered Materials, Center for Materials Science, Los Alamos National Laboratory, June 26, 1995.

27. W.D. Nix, "Mechanical Properties of Thin Films," Luncheon Tutorial, TMS Annual Meeting , Los Angeles, CA, February 6, 1996.

28. W.D. Nix, J.J. Vlassak, R.J. Hohlfelder and J.T. Sizemore, "Metrology of Thin Films Adhesion," Advanced Metallization for ULSI, Spring Meeting of Materials Research Society, April 8, 1996.

29. W.D. Nix, J.J. Vlassak, S.L. Neumann and V.M. Paviot, "Plane Strain Bulge Testing of Thin Metal Films," Thin Films: Stresses and Mechanical Properties VI, Spring Meeting of Materials Research Society, April 8, 1996.

30. R.J. Hohlfelder, H. Luo, J.J. Vlassak, C.E.D. Chidsey and W.D. Nix, "Measuring Interfacial Fracture Toughness with the Blister Test," Thin Films: Stresses and Mechanical Properties VI, Spring Meeting of Materials Research Society, April 8, 1996.

31. W.D. Nix, "Dislocation Channeling: A Robust Concept for Understanding Strength in Thin Films, Multilayers and Other Finely Structured Materials," 1996 ASME Mechanics and Materials Conference, Johns Hopkins University, June 12-14, 1996. (Invited Keynote Address)

32. R.J. Hohlfelder, W.D. Nix and A.S. Mack, "Using the Blister Test to Measure Interfacial Fracture Toughnesses of Inorganic Film/Substrate Systems," MRS Symposium: Interfacial

Engineering for Optimized Properties, Materials Research Society, Fall Meeting, Boston, MA, December 4, 1996.

33. W.D. Nix, "Dislocation Channeling: A Robust Concept for Understanding Strength of Thin Films, Multilayers and Other Finely Structured Materials," Department Colloquium, Department of Mechanical Engineering, University of California at Santa Barbara, February 24, 1997

34. R. Saha, T.Y. Ting, C.A. Ross and W.D. Nix, "Scanning Stiffness Microscopy - A Novel Technique for Detecting Subsurface Cracks," MRS Symposium: Materials Reliability in Microelectronics VII, Materials Research Society, Spring Meeting, San Francisco, CA, March 31, 1997.

35. O. Kraft and W.D. Nix, "Quantitative Measurement of Interface Fracture Energy by Microbeam Deflection," MRS Symposium: Materials Reliability in Microelectronics VII, Materials Research Society, Spring Meeting, San Francisco, CA, March 31, 1997.

36. W.D. Nix, "Techniques for the Study of Interface Fracture in Thin Film Systems," Nano-Fracture Mechanics Symposium, International Conference on Fracture, Sydney, Australia, April 2, 1997

37. W.D. Nix, "Bulge and Blister Testing of Thin Metal Films on Substrates," Advances in Coating and Thin Film Characterization, Mechanical Properties and Film Adhesion, ICMCTF 97, April 25, 1997

38. W.D. Nix, "Mechanical Properties of Thin Films and Interfaces: Bulge and Blister Testing," Department Colloquium, Department of Materials Science and Mineral Engineering, University of California at Berkeley, May 8, 1997

39. W.D. Nix, "Bulge and Blister Testing of Thin Metal Films on Substrates," Micromechanics Session, 1997 SEM Spring Conference, Bellevue, Washington, June 3, 1997 (invited lecture)

40. W.D. Nix, "Modeling Plastic Deformation and Dislocation Interactions in Thin Metal Films," Mechanical Behavior of Small Scale Structures: Theory and Experiment, McNU 97, Northwestern University, July 2, 1997 (invited lecture)

41. W.D. Nix, "Elastic and Plastic Properties of Thin Films on Substrates: Nanoindentation Techniques," International Conference on Strength of Materials, ICSMA 11, Prague, Czech Republic, August 25-29, 1997 (invited lecture)

42. R. Hohlfelder and W.D. Nix, "Blister Testing Techniques for the Study of Interface Fracture in Thin Film Systems," ASME International Congress and Exposition, Dallas, TX, November 20-22, 1997 (invited presentation)

43. W. D. Nix and H. Gao, "Plastic Deformation in Small Volumes - The Separate Effects of Fine Structure and Strain Gradients," ASME International Congress and Exposition, Dallas, TX, November 20-22, 1997 (invited lecture)

44. J.J. Vlassak, T. Tsui and W.D. Nix, "A New Tchnique for Visualizing the Displacement Field of Indentations: The Case of a Soft Film on a Hard Substrate," MRS Symposium: Thin Films: Stresses and Mechanical Properties, Materials Research Society, Fall Meeting, Boston, MA, December 1, 1997.

45. T. Tsui, J.J. Vlassak, and W.D. Nix, "Indentation of a Hard Film on a Soft Substrate: Visualization of the Displacement Field," MRS Symposium: Thin Films: Stresses and Mechanical Properties, Materials Research Society, Fall Meeting, Boston, MA, December 2, 1997. (poster presentation).

46. G. Cornella , S-H. Lee, O. Kraft, W.D. Nix, and J.C. Bravman, "Determination of Temperature Dependent Unstressed Lattice Spacings in Crystalline Thin Films on Substrates," MRS Symposium: Thin Films: Stresses and Mechanical Properties, Materials Research Society, Fall Meeting, Boston, MA, December 4, 1997.

47. W.D. Nix, "Modeling Stress-Temperature Hysteresis Curves for Thin Metal Films on Substrates ," Defects in Crystals: A Symposium Honoring the Contributions of John P. Hirth: Mechanical Properties, TMS Annual Meeting, San Antonio TX, February 18, 1998.

48. W.D. Nix, "Dislocation Channeling: A Robust Concept for Understanding Strength of Thin Films, Multilayers and Other Finely Structured Materials ," Peter G. Winchell Memorial Lecture, School of Materials Engineering, Purdue University, April 6, 1998.

49. W.D. Nix, R. Saha, E.T. Lilleodden, K.W. McElhaney and J.J. Vlassak, "Determination of Mechanical Properties of Bulk and Thin Film Materials by Nanoindentation: Contact Area Problems and Substrate Stiffness Effects," MRS Symposium: Measuring Mechanical Properties in the Nanometer Regime, Materials Research Society, Spring Meeting, San Francisco, CA, April 12, 1998.

50. O. Kraft and W.D. Nix, "Thermomechanical Behavior of Continuous and Patterned Thin Films ," MRS Symposium: Materials Reliability in Microelectronics, Materials Research Society, Spring Meeting, San Francisco, CA, April 13, 1998.

51. O.S. Leung and W.D. Nix, "Passivation and Substrate Effects on the Yield Strengths of Gold Films," MRS Symposium: Micromechanical Structures for Materials Research, Materials Research Society, Spring Meeting, San Francisco, CA, April 15, 1998.

52. O. Kraft and W.D. Nix, "Measurement of Mechanical Properties in Small Dimensions by Microbeam Deflection," MRS Symposium: Micromechanical Structures for Materials Research, Materials Research Society, Spring Meeting, San Francisco, CA, April 15, 1998.

53. W.D. Nix, "Plastic Deformation in Small Volumes – The Separate Effects of Fine Structure and Strain Gradients ," Invited Seminar, Graduate Aeronautical Laboratories, Caltech, Pasadena, CA , April 30, 1998.

54. W.D. Nix, "Interface Fracture in Thin Film Systems," International Workshop of Mechanics and Design of Small-Scale Materials and Systems," Tegernsee, Germany, June 16, 1998.

55. W.D. Nix, "Dislocation Plasticity in Thin Metal Films on Substrates," Gordon Conference on Thin Film Mechanical Behavior," Plymouth State College, Plymouth NH, June 21-25, 1998.

56. W.D. Nix, "Mechanical Properties of Thin Films," Acta Materialia Workshop on Materials Science and Mechanics of Interfaces, La Jolla, CA, October 27, 1998.

57. W.D. Nix, O.S. Leung and S.M. Brennan, "Yielding and Strain Hardening of Thin Metal Films on Substrates," ASME Symposium: Emerging Topics in Fracture and Failure - II, ASME Annual Meeting, Anaheim CA, November 20, 1998.

58. W.D. Nix, "Mechanisms of Plasticity in Polycrystalline Thin Films on Substrates," MRS Symposium: Polycrystalline Thin Films – Processing-Structure-Property Relationships, Materials Research Society, Fall Meeting, Boston, MA, December 3, 1998.

59. Y. Huang, H. Gao, W.D. Nix and J. Hutchinson "The Mechanism-Based Strain Gradient Plasticity," MRS Symposium: Fracture and Ductile vs. Brittle Behavior – Theory, Modeling and Experiment, Materials Research Society, Fall Meeting, Boston, MA, December 2, 1998.

60. W.D. Nix and O.S. Leung, "Modeling Plastic Deformation and Strain Hardening in Thin Metal Films on Substrates," Micromechanisms and Micromechanics of Deformation and Fracture: Symposium in Honor of Professor Ali S. Argon, Annual Meeting of The Metallurgical Society, San Diego, CA, March 1, 1999.

61. W.D. Nix, O.S. Leung and S.M. Brennan, "Strengthening Mechanisms in Thin Metal Films on Substrates," Mechanical Properties of Films, Coatings and Interfacial Materials, United Engineering Foundation Inc. Conference, Castelvecchio Pascoli, Italy, June 28, 1999.

62. J. Florando, Q. Ma, H. Fujimoto, O. Kraft, R. Schwaiger and W.D. Nix, "Measurement of Thin Film Mechanical Properties by Microbeam Bending," MRS Symposium: Materials Reliability in Microelectronics IX, Materials Research Society, Spring Meeting, San Francisco, CA, April 8, 1999.

63. W.D. Nix, "Plasticity of Crystalline Materials at Small Length Scales," MRS Symposium: Multiscale Phenomena in Materials – Experiments and Modeling, Materials Research Society, Fall Meeting, Boston, MA, November 30, 1999.

64. W.D. Nix, "Mechanisms of Time-Dependent Plasticity in Polycrystalline Thin Films on Substrates," TMS Annual Meeting, Nashville, TN, March 15, 2000.

65. W.D. Nix "Mechanical Properties of Thin Films," Alpha Sigma Mu Lecture, ASM International, St. Louis, MO, October 9, 2000.

66. W.D. Nix, "Mechanical Properties of Thin Films," First J.B. Cohen Memorial Lecture, Materials Science and Engineering, Northwestern University, Evanston, Illinois, October 20, 2000.

67. W. D. Nix, R. Saha, E. T. Lilleodden, D. Barbero and B. M. Clemens, "Experiments on Theoretical Strength and Size Effects in Nanoindentation," Invited Lecture, Symposia W and Q, Materials Research Society, Boston, MA, November 30, 2000.

68. W. D. Nix, O.S. Leung and D. Weiss, "Modeling Plasticity in Polycrystalline Gold Thin Films on Silicon," Invited Lecture, Symposium P, Materials Research Society, San Francisco, CA, April 17, 2001.

69. J.N. Florando and W.D. Nix, "Study of the Yielding and Strain Hardening Behavior of a Copper Thin Film on a Silicon Substrate Using Microbeam Bending," Materials Research Society, Spring Meeting, San Francisco, CA, April 17, 2001.

70. E.T. Lilleodden, J.A. Zimmerman, S.M. Foiles and W.D. Nix, "An experimental and computational study of the elastic-plastic transition in thin films," MRS Spring Meeting, San Francisco, CA, April 17, 2001.

71. M.A. Phillips, V. Ramaswamy, B.M. Clemens and W.D. Nix, "Mechanical Properties of Aluminum-Scandium Multilayers," Materials Research Society (MRS) Spring Meeting, San Francisco, April 16-20, 2001.

72. W.D. Nix, "Mechnical Properties of Thin Films," Department Seminar, Chemical Engineering and Materials Science, University of California, Davis, April 30, 2001.

73. W.D. Nix and Ranjana Saha, "Effects of the Substrate on the Determination of Thin Film Mechanical Properties by Nanoindentation," Nano User's Group Meeting, Las Vegas, Nevada, July 30-31, 2001.

74. J.N. Florando and W.D. Nix, "Measurement of Thin Film Mechanical Properties by Microbeam Bending," MTS Nano Users Meeting, Las Vegas, NV, July 30-31, 2001.

75. W.D. Nix, E.T. Lilleodden, R. Saha, D. Barbero and B. M. Clemens, "Indentation Size Effects and Length Scales for Plasticity," 2nd European Symposium on Nanomechanical Testing, Hückelhoven, Germany, September 26, 2001. (Video Presentation).

76. William D. Nix, Ranjana Saha, Erica Lilleodden, David Barbero and Bruce M. Clemens, "Indentation-Induced Dislocation Plasticity in Crystals," Multiscale Modeling of Materials Strength and Failure, Sponsored by: DOE/DP and LLNL, Bodega Bay, CA, October 7-10, 2001.

77. E.T. Lilleodden and W.D. Nix, "Microstructural length-scale effects in metal thin films," ASME International Mechanical Engineering Congress, New York City, NY November, 2001.

78. W.D. Nix, "Mechanical Properties of Thin Films," Second Lawrence Van Vlack Memorial Lecture, Department of Materials Science and Engineering, University of Michigan, Ann Arbor, MI, February 7-8, 2002.

79. W.D. Nix, "Experiments on Size Effects and Theoretical Strength in Nanoindentation," Department of Materials Science and Engineering, University of California, Berkeley, CA, February 14, 2002.

80. J.N. Florando and W.D. Nix, "Study of the Yielding and Strain Hardening Behavior of a Cu Film Using Microbeam Bending," TMS Annual Meeting, Seattle, WA, February 19, 2002.

81. M.A. Phillips, B.M. Clemens, W.D. Nix, "High strength nanoscale Al/Al_3Sc multilayers formed by interface reaction," Nanostructured Interfaces Symposium, Materials Research Society (MRS) Spring Meeting, San Francisco, April 1-5, 2002.

82. W.D. Nix, O.S. Leung and J.N. Florando, "Modeling Plasticity in Polycrystalline Gold Thin Films on Silicon Substrates - Emphasis on Strain Hardening," 14th U.S. National Congress of Theoretical and Applied Mechanics, Virginia Tech, Blacksburg, VA, June 23-28, 2002.

83. M.A. Phillips, B.M. Clemens, W.D. Nix, "Deformation behavior of high-strength nanoscale Al/Al_3Sc multilayers," USNCTAM14; 14th U.S. National Congress of Theoretical and Applied Mechanics, Blacksburg VA, June 23-28, 2002.

84. W.D. Nix, Ranjana Saha, Seung-Min Han and Mark Phillips, "Using Contact Stiffness in Nanoindentation of Thin Films and Multilayers on Substrates," Nano User's Group Meeting, Las Vegas, Nevada, August 19-20, 2002.

85. J.N. Florando and W.D. Nix, "Not Just Nanoindentation: Methods of Measuring the Stress-Strain Behavior of Materials in Small Dimensions Using a Nanoindenter," MTS Nano Users Meeting, Las Vegas, NV, August 20, 2002.

86. W.D. Nix, "Mechanical Properties of Thin Films," Edwin G. Baetjer II Colloquium, Department of Mechanical & Aerospace Engineering, Princeton University, December 12, 2002.

87. W.D. Nix, "Modeling Plasticity in Polycrystalline Thin Films on Silicon Substrates," Edwin G. Baetjer II Colloquium, Department of Mechanical & Aerospace Engineering, Princeton University, December 13, 2002.

88. W.D. Nix, "Modeling Strength and Plasticity in Thin Films on Substrates," L.B. Freund Symposium, California Institute of Technology, January 17, 2003.

89. W.D. Nix, "Mechanical Properties of Thin Films," Department Seminar, School of Materials Science and Engineering, Georgia Institute of Technology, April 15, 2003.

90. J.N. Florando and W.D. Nix, "Study of the Yielding Behavior of Copper Thin Films on Silicon Substrates Using Microbeam Bending," Materials Research Society Spring Meeting, San Francisco, CA, April 24, 2003.

91. W.D. Nix, “Mechanical Properties of Thin Films,” Department Seminar, Department of Mechanical Engineering, University of California, Riverside, May 14, 2003.

92. D.-H Choi, H. Kim and W.D. Nix, "Anelasticity and Damping of Al thin films on Si substrates,” ASME conference, Scottsdale, AZ , June 2003.

93. M.A. Phillips, B.M. Clemens, W.D. Nix, “Mechanical properties of Al/Al_3Sc metallic multilayers - deformation mechanisms at the nanoscale”, THERMEC'2003, International Conference on Processing and Manufacturing of Advanced Materials, Leganés, Madrid, Spain, July 7-11, 2003.

94. W.D. Nix, “Length Scale Effects on Mechanical Properties of Crystalline Materials,” SSRL/ALS Workshop: Probing Mechanical Deformation and Failure via Synchrotron Radiation, SSRL, Stanford CA, October 8, 2003.

95. W.D. Nix, “Size Effects in Plasticity,” Materials at the Micro and Nano Scale:The Interaction between Processing, Mechanical, and Electrical Properties, ASME International Mechanical Engineering Congress, Washington, DC, November 20, 2003.

96. Julia R. Greer and William D. Nix, “Size effects in Mechanical Testing of Gold at the Microscale,” MRS Fall Meeting Symposium U on Thin Films: Stresses and Mechanical Properties, Boston, MA, December 2003.

97. D.-H Choi and W.D. Nix, "Study of Anelasticity observed in Al and Au films on Si substrates,” MRS Fall Meeting Symposium U on Thin Films: Stresses and Mechanical Properties, Boston, MA December, 2003.

98. W.D. Nix, “Modeling Strength and Plasticity in Thin Films and Multilayers,” *Mechanical behaviour of systems at small length scales*, Indian Institute of Science, Bangalore, India, January 12-16, 2004

V. Publications Resulting from DOE Grant No. DE-FG03-89ER45387

1. S.R. Nutt, K.A. Green, S.P. Baker, W.D. Nix and A. Jankowski, "Gold-Nickel Multilayer Films: Structure-Property Correlations", Materials Research Symposium Proceedings, 130, 129 (1989)

2. M.J. Mayo, R.W. Siegel, A. Narayanasamy and W.D. Nix, "Mechanical Properties of Nanophase TiO_2 as Determined by Nanoindentation", Journal of Materials Research, 5, 1073 (1990).

3. S.P. Baker, A.F. Jankowski, S. Hong and W.D. Nix, "Mechanical Properties of Compositionally Modulated Au-Ni Thin Films Using Indentation and Microbeam Deflection Techniques", Materials Research Symposium Proceedings, 188, 289-294 (1990).

4. S.P. Baker and W.D. Nix, "Mechanical Properties of Thin Films on Substrates", in Optical Thin Films III: New Developments, Proc. SPIE, 1323, 263-276 (1990).

5. M.J. Mayo, R.W. Siegel, Y.X. Liao and W.D. Nix, “Nanoindentation of Nanocrystalline ZnO”, Journal of Materials Research, 7, 973-979 (1992).

6. J.J. Vlassak, T. Nakayama, T.J. Konno and W.D. Nix, "Structure and Mechanical Properties of Fe/Zr Multilayers", Materials Research Symposium Proceedings, 239, 493-498 (1992).

7. M.K. Small, J.J. Vlassak and W.D. Nix, "Re-Examining the Bulge Test: Methods for Improving Accuracy and Reliability", Materials Research Symposium Proceedings, 239, 257-262 (1992).

8. S.P. Baker, T.W. Barbee, Jr. and W.D. Nix, "Time-Dependent Deformation in Room-Temperature Indentation Experiments using a Nanoindenter", Materials Research Symposium Proceedings, 239, 319-324 (1992).

9. M.K. Small and W.D. Nix, "Analysis of the Accuracy of the Bulge Test in Determining the Mechanical Properties of Thin Films", Journal of Materials Research, 7, 1553-1563 (1992).

10. J.J. Vlassak and W.D. Nix, "A New Bulge Test Technique for the Determination of Young's Modulus and Poisson's Ratio of Thin Films", Journal of Materials Research, 7, 3242-3249 (1992).

11. S.P. Baker, M.K. Small, J.J. Vlassak, B.J. Daniels and W.D. Nix, "The Search for the Supermodulus Effect", Proceedings of the NATO Advanced Study Institute: "Mechanical Properties and Deformation Behavior of Materials Having Ultra Fine Microstructures",

Eds.: M. Nastasi, D.M. Parkin and H. Gleiter, Kluwer Academic Publishers, NATO ASI Series, Series E: Applied Sciences, 233, 165-192 (1993).

12. J.J. Vlassak and W.D. Nix, "The Indentation Modulus of Elastically Anisotropic Half Spaces", Phil. Mag., A67, 1045-1056 (1993).

13. M.K. Small, J.J. Vlassak, S.F. Powell, B.J. Daniels and W.D. Nix, "Accuracy and Reliability of Bulge Test Experiments", Materials Research Symposium Proceedings, 308, 159-164 (1993).

14. S. Bader, P.A. Flinn, E. Arzt and W.D. Nix, "Dispersion Strengthening of Al Films by Oxygen Ion Implantation", Materials Research Symposium Proceedings, 308, 317-321 (1993).

15. M.K. Small, B.J. Daniels, B.M. Clemens and W.D. Nix, "The Elastic Biaxial Modulus of Ag-Pd Multilayered Thin Films Measured Using the Bulge Test", Journal of Materials Research, 9, 25-30 (1994).

16. S. Bader, P.A. Flinn, E. Arzt and W.D. Nix, "Mechanical Strength and Microstructure of Oxygen Ion-Implanted Al Films", Journal of Materials Research, 9, 318-327 (1994).

17. J.J. Vlassak and W.D. Nix, "Measuring the Elastic Properties of Anisotropic Materials by Means of Indentation Experiments," J. Mech. Phys. Solids, 42, 1223-1245 (1994).

18. Baker and W.D. Nix, "Mechanical Properties of Compositionally-Modulated Au-Ni Thin Films: Nanoindentation and Micro-Cantilever Beam Deflection Experiments," Journal of Materials Research, 9, 3131-3144 (1994).

19. S.P. Baker and W.D. Nix, "Intrinsic Stresses in Compositionally-Modulated Au-Ni Thin Films and the Supermodulus Effect," Journal of Materials Research, 9, 3145-3152 (1994).

20. S.P. Baker, J.A. Bain, B.M. Clemens and W.D. Nix, "Interfacial Structure and Mechanical Properties of Compositionally-Modulated Au-Ni Thin Films," Materials Research Symposium Proceedings, 343, 340-346 (1994).

21. M.K. Small and W.D. Nix, "Calculation of the [111]-texture Dependence of the Elastic Biaxial Modulus," Materials Research Symposium Proceedings, 343, 561-566 (1994).

22. G.R. English, Glenn F. Simenson, Bruce M. Clemens and William D. Nix, "Mechanical Properties of Ag/Cr Multilayered Epitaxial Thin Films" Materials Research Symposium Proceedings, 356, 363-368 (1995).

23. V.M. Paviot, J.J. Vlassak and W. D. Nix, "Measuring the Mechanical Properties of Thin Metal Films by Means of Bulge Testing of Micromachined Windows" Materials Research Symposium Proceedings, 356, 579-584 (1995).

24. Robert J. Hohlfelder, Joost J. Vlassak, William D. Nix, Huihong Liu and Christopher E.D. Chidsey, "Blister Test Analysis Methods" Materials Research Symposium Proceedings, 356, 585-590 (1995).

25. A.B. Mann, J.B. Pethica and W.D. Nix , "Nanoindentation of Epitaxial Thin Films: A Study of Pop-In Events" Materials Research Symposium Proceedings, 356, 271-276 (1995).

26. Jim Sizemore, R.J. Hohlfelder, J.J. Vlassak and W.D. Nix, "Measuring the Adhesion of Diamond Thin Films to Substrates Using the Blister Test" Materials Research Symposium Proceedings, 383, 197-207 (1995).

27. W.D. Nix, "Elastic and Plastic Properties of Thin Films on Substrates: Nanoindentation Techniques," Materials Science and Engineering A, A234-236, 37-44 (1997).

28. R.J. Hohlfelder, W.D. Nix and A.S. Mack, "Using the Blister Test to Measure Interfacial Fracture Toughnesses of Inorganic Film/Substrate Systems," Materials Research Symposium Proceedings, accepted (1997).

29. R. J. Hohlfelder, H. Luo, J. J. Vlassak, and C.E.D. Chidsey and W. D. Nix, , "Measuring Interfacial Fracture Toughness with the Blister Test" Materials Research Symposium Proceedings, Materials Research Symposium Proceedings, 436, 115-120 (1997).

30. R. Saha, T.Y. Ting, C.A. Ross and W.D. Nix, "Scanning Stiffness Microscopy - A Novel Technique for Detecting Sub-Surface Cracks," Materials Reliability in Microelectronics VII,Materials Research Symposium Proceedings. , 285-90 (1997).

31. Guido Cornella, Seok-Hee Lee, William D. Nix and John C. Bravman, "An Analysis Technique for Extraction of Thin Film Stresses from X-Ray Data", Applied Physics Letters, 97, 2949-2951 (1997).

32. W.D. Nix and H. Gao, "Indentation Size Effects in Crystalline Materials: A Law for Strain Gradient Plasticity," J. Mechanics and Physics of Solids, 46, 411-425 (1998).

33. R. Spolenak, O. Kraft, W.D. Nix and E. Arzt, "Alloying Effects in Electromigration: Modelling and Experiments," Proceedings of the Fourth International Workshop on Stress Induced Phenomena in Metallizations, AIP Conf. Proc. (USA), AIP Conference Proceedings, no.418, 147-58, (1998).

34. K.W. McElhaney, J.J. Vlassak and W.D. Nix, "Determination of Indenter Tip Geometry and Indentation Contact Area for Depth-Sensing Indentation Experiments," Journal of Materials Research, 13, 1300-1306 (1998).

35. O. Kraft and W.D. Nix, "Measurement of the lattice thermal expansion coefficients for thin metal films on substrates," Journal of Applied Physics, 83, 3035-3038 (1998).

36. Guido Cornella, Seok-Hee Lee, William D. Nix and John C. Bravman, "Determination of Temperature Dependent Unstressed Lattice Spacings in Crystalline Thin Films on

Substrates," Materials Research Symposium Proceedings, VII, MRS Proc. Vol. 505, p. 527-532, (1998)

37. O. Kraft and W.D. Nix"Thermomoechanical Behavior of Continuous and Patterned Al Thin Films" in Materials Reliability in Microelectronics VIII edited by J.C.Bravman, J.R. Lloyd, M. Korhonen, and T. Marieb, Materials ResearchSociety Symp. Proc. 516, Pittsburgh, PA, 201-212 (1998).

38. O. Kraft, R. Schwaiger, and W.D. Nix, "Measurement of Mechanical Properties in Small Dimensions by Microbeam Deflection" in Microelectromechanical Structures for Materials Research edited by S. Brown, H. Guckel, G. Johnson, J. Gilbert, R. Howe, and P. Krulevitch, Materials Research Society Symp. Proc. 518, Pittsburgh, PA, pp. 39-44 (1998).

39. W.D. Nix, "Yielding and Strain Hardening of Thin Metal Films on Substrates," Scripta Materialia, 39, 545-554 (1998).

40. W.D. Nix and H. Gao, "An Atomistic Interpretation of Interface Stress," Scripta Materialia, 39, 1653-1661 (1998).

41. Joost J. Vlassak, T.Y. Ting and W.D. Nix, "A New Technique for Visualizing the Displacement Field of Indentations; The case of a Soft Film on a Hard Substrate," Thin-Films-Stresses and Mechanical Properties VII, Materials Research Symposium Proceedings, 505, 71-76 (1998).

42. H. Gao and W.D. Nix, "Surface Roughening of Heteroepitaxial Thin Films," Annual Reviews of Materials Science 29, 173-209 (1999).

43. T.Y. Tsui, J.J. Vlassak and W.D. Nix, Indentation plastic displacement field: Part I. The case of soft film on hard substrates," J. Materials Research, 14, 2196-2203 (1999).

44. T.Y. Tsui, J.J. Vlassak and W.D. Nix, Indentation plastic displacement field: Part II. The case of hard film on soft substrates," J. Materials Research, 14, 2204-2209 (1999).

45. H. Gao, L. Zhang, W.D. Nix, C.V. Thompson and E. Arzt, "Crack-Like Grain Boundary Diffusion Wedges in Thin Metal Films," Acta Materialia, 47, 2865-2878 (1999).

46. H. Gao, Y. Huang, W. D. Nix, "Modeling Plasticity at the Micrometer Scale," Naturwissenschaften Vol. 86, No. 11, 507-515 (1999).

47. J. Florando, H. Fujimoto, Q. Ma, O. Kraft, R. Schwaiger and W.D. Nix, "Measurement of Thin Film Mechanical Properties by Microbeam Bending," Materials Reliability in Microelectronics IX, Materials Research Symposium Proceedings. 563, 231-236 (1999).

48. H. Gao, Y. Huang, W.D. Nix and J.W. Hutchinson, "Mechanism-Based Strain Gradient Plasticity - I. Theory," Journal of Mechanics and Physics of Solids, 47, 1239-1263 (1999).

49. M. Schultz and W.D. Nix, "Finite Element Analysis of the Wedge Delamination Test," Int. Journal of Solids and Structures, 37, No. 7, 1045-1063 (2000).

50. Y. Huang, H. Gao, W.D. Nix and J.W. Hutchinson, "Mechanism-Based Strain Gradient Plasticity - II. Analysis," Journal of Mechanics and Physics of Solids, 48, 99-128 (2000).

51. Deok-kee Kim, Birgit Heiland, William D. Nix, Eduard Arzt, Michael D. Deal and James D. Plummer, "Microstructure of Thermal Hillocks on Blanket Al Thin Films," Thin Solid Films, 371: (1-2) 278-282 (2000).

52. Deok-kee Kim, William D. Nix, Michael Deal and James D. Plummer, "Creep-controlled diffusional hillock formation in blanket aluminum thin films as a mechanism of stress relaxation," J. Materials Research, 15 (8), 1709-1718 (2000).

53. T.R. Dalbeck, O.S. Leung and W.D. Nix, "Mechanisms of Time-Dependent Plasticity in Polycrystalline Thin Films on Substrates," Deformation, Processing, and Properties of Structural Materials, E.M. Taleff, C.K. Syn and D.R. Lesuer (Eds.), The Metallurgical Society, 2000, pp. 95-108.

54. Y. Huang, Z. Xue, H. Gao, W.D. Nix, Z.C. Xia, "A study of microindentation hardness tests by mechanism-based strain gradient plasticity," Journal of Materials Research, 15, 1786-1796 (2000).

55. Y. Huang, H. Gao, W.D. Nix and J.W. Hutchinson, "Mechanism-Based Strain Gradient Plasticity - II. Analysis," Journal of Mechanics and Physics of Solids, 48, 99-128 (2000).

56. M. Schulze and W.D. Nix, "Finite Element Analysis of the Wedge Delamination Test," Int. Journal of Solids and Structures, 37, No. 7, 1045-1063 (2000).

57. O.S. Leung, A. Munkholm, S. Brennan and W.D. Nix, "A Search for Plastically-Induced Strain Gradients in Gold Thin Films on Silicon Substrates using Depth-sensitive X-Ray Diffraction," Journal of Appl. Phys., 88, 1389-1396 (2000).

58. S. Brennan, A. Munkholm, O.S. Leung and W.D. Nix, "X-ray Measurement of the Depth Dependence of Stress in Gold Thin Films," Physica B. 283, 125-129 (2000).

59. M. Goken, M. Kempf and W.D. Nix, "Hardness and modulus of the lamellar microstructure in PST-TiAl studied by nanoindentations and AFM," Acta Materialia, 49 (5): 903-911 (2001).

60. M. Goken, R. Sakidja W.D. Nix and J.H. Perepetzko, "Microstructural Mechanical Properties and Yield Point Effects in Mo Alloys," Materials Science and Engineering A, 319, 902-908 (2001)

61. R. Saha and W.D. Nix, "Soft Films on Hard Substrates – Nanoindentation of Tungsten Films on Sapphire Substrates," Materials Science and Engineering A, 319, 898-901 (2001)

62. Deok-kee Kim, William D. Nix, Richard P. Vinci, Michael Deal and James D. Plummer, "Study of the effect of grain boundary migration on hillock formation in Al thin films," J. Appl. Phys., 30 (2), 781-788 (2001).

63. Y. Huang, M.J. Aziz, J.W. Hutchinson, A.G. Evans, R. Saha and W.D. Nix, “Comparison of Mechanical Properties of Ni3Al Thin Films in Disordered FCC and Ordered L12 Phases,” Acta Materialia, 49, 2853-2861 (2001).

64. Ranjana Saha, Zhenyu Xue, Young Huang and William D. Nix, “Indentation of a soft metal film on a hard substrate: strain gradient hardening effects,” J. Mech. Phys. Solids, 49, 1997-2014 (2001).

65. Jeffrey N. Florando and William D. Nix, “Study of Yielding and Strain Hardening Behavior of a Copper Thin Film on a Silicon Substrate Using Microbeam Bending,” Mat. Res. Soc. Symp. 673, 1.9.1-1.9.6 (2001).

66. Erica T. Lilleodden, Jonathan A. Zimmerman, Stephen M. Foiles and William D. Nix, “An Experimental and Computational Study of the Elastic-Plastic Transition in Thin Films,” Mat. Res. Soc. Symp. 673, 1.3.1-1.3.6 (2001).

67. W.D. Nix and O.S. Leung, “Thin Films: Plasticity,” Encylopedia of Materials: Science and Technology, Elsevier Science Ltd. pp. 9262-9274 (2001).

68. Ranjana Saha and William D. Nix, “Effects of the substrate on the determination of thin film mechanical properties by nanoindentation,” Acta Materialia, 50, 23-38 (2002).

69. N. Huber, W. D. Nix, and H. Gao, “Identification of elastic-plastic material parameters from pyramidal indentation of thin films,” Proceedings of the Royal Society of London Series a-Mathematical Physical and Engineering Sciences 458 (2023), 1593-1620 (2002).

70. M.A. Phillips, B.M. Clemens, W.D. Nix, “High strength nanoscale Al/Al3Sc multilayers formed by interface reaction,” Nanostructured Interfaces Symposium, Materials Research Society (MRS), MRS Proceedings Volume 727 (2002).

71. E.T. Lilleodden , J.A. Zimmerman , S.M. Foiles and W.D. Nix. "Atomistic Simulations of Elastic Deformation and Dislocation Nucleation During Nanoindentation," Journal of the Mechanics and Physics of Solids, 51, No. 5 , 901-920 (2003).

72. M.A. Phillips, B.M. Clemens, W.D. Nix, "Mechanical properties of Al/Al3Sc metallic multilayers - deformation mechanisms at the nanoscale," THERMEC'2003, pts. 1-5, 426-4, 3373-3378 (2003).

73. M.A. Phillips, B.M. Clemens and W.D. Nix, "Microstructure and nanoindentation hardness of Al/Al3Sc multilayers," Acta Materialia, 51, 3157-3170 (2003).

74. M.A. Phillips, B.M. Clemens and W.D. Nix, "A model for dislocation behavior during deformation of Al/Al3Sc (fcc/L1(2))metallic multilayers," Acta Materialia, 51, 3171-3184 (2003).