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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/Al₃Sc 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$_3$Sc multilayers. We have made and studied the mechanical properties of Al/Al$_3$Sc 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$_3$Sc, 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$_3$Sc layers are perfectly coherent with the Al layers, making these alloys analogous to Ni/Ni$_3$Al 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$_3$Sc layers, suggesting that shearing of the Al$_3$Sc 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$_3$Sc 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/Al₃Sc 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 Al₃Sc, 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/Al₃Sc 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/Al₃Sc 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$_3$Sc 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$_3$Sc multilayers as a function of Al layer thickness. The different groups of data correspond to different Al$_3$Sc layer thicknesses.

The accompanying hardness results show that layering with nanoscale coherent Al$_3$Sc 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$_3$Sc thicknesses. While the strength clearly increases with decreasing Al layer thickness, as expected, the multilayers with
the thinnest Al$_3$Sc are weakest. As described in the paper cited below, we believe this
dependence on the thickness of the Al$_3$Sc 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

**B. A model for dislocation behavior during
deformation of Al/Al$_3$Sc (fcc/L1$_2$) metallic
multilayers**

*Mark A. Phillips*

In the previous paper, we described the microstructure and indentation hardness of
nanoscale Al/Al$_3$Sc 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$_3$Sc. We show that the anti-phase
boundary (APB) energy of the L1$_2$, Al$_3$Sc 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$_3$Sc
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$_3$Sc barrier layers. We see that for the largest Al layer thickness and for the
thickest Al$_3$Sc layers, the model coincides with the data. But for thinner Al layers and especially
for very thin Al$_3$Sc 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$_3$Sc
layers are cut by collapsing dislocation dipoles.

deformation of Al/Al$_3$Sc (fcc/L1(2))metallic multilayers," Acta Materialia, 51, 3171-3184
(2003).
Fig. B.1  Hardness of Al/ Al$_3$Sc multilayers as a function of Al layer thickness. The different groups of data correspond to different Al$_3$Sc layer thicknesses. The line shows the strength predicted if dislocations are completely confined to the Al layers.

\[ H = 2.7 \sigma_y \]
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 1µm) 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°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:

\[
M = \frac{M_t}{w} = \frac{P(L-x)}{w} = \frac{P(L-x)}{w_o} \left( \frac{L-x}{L} \right) = \frac{PL}{w_o} = \text{constant}.
\]  

(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, \( \delta u \), may then be regarded as a time derivative for a time-independent process. Corresponding increments in the load, \( \delta P \), moment (per unit width), \( \delta M = (L/w_o)\delta P \), and curvature, \( \delta k = (2/L^2)\delta u \), 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.
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,

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

(2)

where $\bar{\varepsilon}^f$ is the equivalent strain, $\bar{\sigma}^f$ is the equivalent stress, $E_f$ is Young’s modulus, $\sigma_o$ 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 ($\sigma_o$ and $m$) from the experimental load-displacement data. To make this determination we first assume values for $\sigma_o$ and $m$ and calculate the incremental loads and displacements during bending. We choose those values of $\sigma_o$ and $m$ that provide the best fit to the experimental load-displacement data. For a given set of $\sigma_o$ and $m$, the incremental bending loads can be related to the incremental bending displacements using

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

(3)
where $B_s$ is the plane strain modulus of the substrate and where the in-plane stresses in the film, $\sigma_{xx}^f$ and $\sigma_{zz}^f$, are found for each increment of bending using

$$
\delta \sigma_{xx}^f = \left[1 - K_2 \nu_f \left(2 \sigma_{xx}^f - \sigma_{zz}^f\right)ight]^{-1} E_f \delta \epsilon_{xx},
$$

and

$$
\delta \sigma_{zz}^f = K_2 \delta \sigma_{xx}^f,
$$

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

$$
\delta \epsilon_{xx}(y) = \delta \kappa (y - y_o),
$$

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

The parameter $K_2$ in these relations is found using

$$
K_2 = \left(\nu_f \bar{\sigma}_{xx}^f - K_1 \left(2 \sigma_{xx}^f - \sigma_{zz}^f\right)\right) / \left(\bar{\sigma}_{xx}^f + K_1 \left(2 \sigma_{zz}^f - \sigma_{xx}^f\right)\right),
$$

where

$$
K_1 = \frac{m}{2} \left(\frac{\sigma_{zz}^f - \frac{1}{2} \sigma_{xx}^f}{\bar{\sigma}_{xx}^f}\right)^{-1}.
$$

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

$$
0 = \delta M = \int_{0}^{L_s} B_s (y - y_o) dy + \int_{L_s}^{L_s + t_f} \left[1 - K_2 \nu_f \left(2 \sigma_{xx}^f - \sigma_{zz}^f\right)ight]^{-1} E_f \delta \epsilon_{xx}(y - y_o) dy
$$

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 $\sigma_o$, and the strain hardening exponent
I/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.

![Experimental Data vs Ramberg-Osgood Fit](image.png)

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 $\sigma_o$ 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

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

where $\delta P_{\text{film}}$ 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, $\delta P$, includes load contributions from the substrate. Thus $\delta P_{\text{film}}$, needed for the implementation of eqn.(10), is found using,

$$\delta P_{\text{film}} = \delta P - \delta P_{\text{substrate}},$$  \hspace{1cm} (11)

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

$$\delta P_{\text{substrate}} = \frac{2w_0}{L^3} B_s \delta u \int_0^{t_s} (y - y_o)^2 dy = \frac{2w_0}{L^3} B_s [t_s^3 - 3t_s^2 y_o + 3t_s y_o^2] \delta u. \hspace{1cm} (12)$$

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

$$\delta <\varepsilon_{xx}> = \frac{2\delta u}{t_f L^2} \int_{t_s}^{t_s+tf} (y - y_o) dy = \frac{\delta u}{L^2} [2t_s + t_f - 2y_o]. \hspace{1cm} (13)$$

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.
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°-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 µm 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 µm. For the Cu films grown on Ta, the average grain size was 0.2 µm for the 0.5 µm thick film, 0.4 µm for the 1.0 µm thick film, and 0.5 µm for the 1.7 µm 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$^2$ ψ 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.

![Symmetric x-ray diffraction scans for a Cu film deposited on SiO\(_2\) (dashed) and Ta (solid line) showing a dual \(<111>/\langle200\rangle\) texture for the film grown on SiO\(_2\) and a strong \(<111>\) texture for the film grown on Ta.](image)

**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 \(\mu m\) film, were deposited. The two additional films have a thickness of 0.5 \(\mu m\), and 1.7 \(\mu m\). 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 \(\mu m\) 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, it is difficult to separate the effect of film thickness and grain size in these experiments.

![Stress-strain behavior for an as-deposited and annealed Cu film.](image)

**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 μm thick film has a linear strain-hardening rate of about $E/9$, while the 1.0 μm film has a hardening rate of $E/4$. The 0.5 μm 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 μm 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.

![Stress-strain behavior for <111> textured Cu films of three different film thicknesses.](image)

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

### References


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.
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 $5 \times 10^{-7}$ 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.
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...
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.

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