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Estimating the Yield Strength of Thin Metal Films through Elastic-Plastic Buckling-Induced Debonding

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Estimating the Yield Strength of Thin Metal Films through Elastic-Plastic Buckling-Induced Debonding

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Abstract

In this paper, we propose a procedure to estimate the yield strength of thin films by debonding films from their substrate by elastic-plastic buckling under thermally-induced compressive loading. The out-of-plane displacement of the metal lines under conditions of elastic-plastic buckling is dependent on the yield strength of the film. Thus, an inverse estimate of the yield strength is made from measurements of the out-of-plane displacements of the buckled metal lines. The procedure is demonstrated to estimate the yield strength of aluminum lines consistent with measurements by other techniques.

Index Terms

Yield Strength, Metal Films, Buckling, Debond

I. INTRODUCTION

The techniques that are generally used to measure the yield strength of thin metallic films are the micro-tensile tests [1], indentation tests [2], [3], [4], substrate curvature technique [5], [4] and the micro-beam bending technique [6], [7]. The micro-tensile test is similar to the tensile test used for bulk specimen except that the test is carried out on thin films. One of the challenges associated

with this technique is the difficulty in measuring strain accurately in the elongated sample [8]. It also requires a potentially difficult clamping method for at least one end. The indentation test requires one to measure the plastic zone size due to the indent. The yield strength is estimated from the geometry of the indenter and the size of the plastic zone using Johnson's formula [9]. However, the relation between the plastic zone size and yield strength is based on an idealization of the complex stress field around the indenter, which introduces uncertainty in the estimated yield strength. The substrate curvature technique is used to measure the yield strength at high temperatures. In this technique, a laser beam is scanned and reflected across the surface of a film using a rotating mirror and lens system that measures the curvature of the surface at different points [10]. The average stress in the film is then estimated using Stoney's formula [11]. When the metallic film yields, the curvature of the surface becomes constant implying a homogeneous state of stress. One of the assumptions behind Stoney's formula is uniform curvature across the surface in both directions. This assumption is typically violated, and the surface curvature is measured point-wise to obtain an average stress state in the film. Finally, in the micro-beam bending technique, a free standing micro-beam is fabricated, and is bent by the application of an electrostatic or a mechanical force to obtain a load deflection relationship. This load deflection relationship is used to estimate the plastic properties. The challenge associated with this technique is the complicated steps required to fabricate the free standing micro-beam [6]. The micro-beam bending techniques, as with all other bending related measurement techniques, also possess the drawback that the strain and stress state in the test specimen is inhomogeneous. Stress and strain gradients are known to influence the plastic deformation.

In this paper, in order to overcome limitations of current measurement techniques an alternative method for the determination of the yield strength of thin films is proposed. Here, the buckling under thermal excursion of metal lines that are *weakly bonded to stiff substrates* is used to measure the yield strength of the film material. Since the temperatures at which the debond and propagation occur are high enough to cause yielding of the metal, an elastic-plastic model is used to estimate the yield strength. This is possible since the post-buckling debond response, specifically, the out of plane displacement of the film is dependent on the yield strength of the film material. The advantages of the current method over the previous methods are that it requires relatively straightforward specimen preparation and fixturing to carryout the required

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measurements. The specimen preparation only involves simple to perform deposition and etching techniques. Measurements can be made using the ubiquitous (non-specialized) optical microscope. Additionally, the developed procedure enables one to measure the fracture toughness of the interface between the deposited metallic film and the substrate by modifying the specimen geometry to a funnel shaped one as outlined in [12].

II. MATERIALS AND METHODS

A. Experimental Procedure

The metal lines were made of aluminum, and the polymeric film on to which the metal lines were deposited was made of SU8. SU-8 is a high contrast, epoxy-based photoresist designed for micromachining and other microelectronic applications, where a thick chemically and thermally stable image is desired. Different grades of SU8 (for example, SU8-2, SU8-5, SU8-10 and SU8 -25) are available depending on the desired thickness. The SU8-2 material that was used in the present study can be spun to a maximum thickness of 2 μm depending on the spinner speed. The interface between aluminum and SU8 is known to be weak with an interfacial fracture toughness of approximately 0.3 J/m^2 [12]. In the present study, the thickness of the Al film was 2.5 μm, while the thickness of the SU8 film was 2 μm. The aluminum and SU8 films were deposited onto a (100) Si wafer with a thickness that was much higher ($t = 525 - 550\mu m$) than the two individual films. The fabrication procedure was as follows:

1) *Wafer Cleaning*: The silicon wafer was cleaned in a solution of acetone and methanol. It was then further cleaned in a (1:1) mixture of hydrogen peroxide (H$_2$O$_2$) and sulphuric acid (H$_2$SO$_4$). After performing the cleaning steps, the wafer was hot-baked at 120 °C to remove all moisture prior to SU8 deposition.

2) *SU8 Deposition and Curing*: Commercially available SU8-2002, supplied by Microchem Corporation, was spun at a speed of 3000 rpm for 30 seconds to yield a thickness of 2 μm. It was then soft-baked at 65 °C and 95 °C for 1 and 2 minutes respectively. After soft-bake, it was exposed to ultraviolet light for 8 seconds under an aligner. After exposure it was hot-baked first at 65 °C (1 minute), then at 95 °C (1 minute) to crosslink the polymer and hard-baked at 120 °C (15 minutes) to further crosslink the polymer. After each bake step,

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the wafer was cooled in a wafer carrier(> 10 minutes) so as to avoid cracking of the SU8 film.

3) *HMDS and AZ-9260 Deposition*: It is a common practice to deposit a layer of hexamethyldisiazane (HMDS) between the photoresist and the substrate to ensure good adhesion between the photoresist and the substrate. Following the established practice, HMDS was spun at a speed of 4000 rpm (30 seconds). Subsequently, a 10 μm thick positive photoresist (+PR) AZ-9260, (AZ Electronic Material), was spun at a speed of 2000 rpm (30 seconds). The deposited resist was then soft-baked at 110 °C (5 minutes) to remove moisture from the photoresist.

4) *Exposure and AZ-9260 Development*: The positive photoresist was exposed to ultraviolet light through a mask for 60 seconds. The mask used for exposure contained the patterns for lines with widths of 600-1000 μm. After exposure, the photoresist was developed in a 1:3 mixture of AZ-400K and deionized water.

5) *Aluminum Deposition and Lift Off*: Aluminum was deposited on the wafer with patterned photoresist using an e-beam evaporator at a pressure of 5×10^{-7} torr and a deposition rate of 3 Å/second. After deposition, AZ-9260 was washed off in acetone to yield the desired patterns of the metal film on the wafer.

The experimental setup to heat the patterned metal line containing chips is shown in Figure 1. The heating stage (Linkam Scientific Instruments, model THMS600) and temperature controller (Linkam TMS94) associated with it could measure temperature to an accuracy of 0.1 °C. The debonding of the metal lines was observed through a common metallugical microscopse (Olympus BX60M) as shown in the figure. The images in Figure 2 illustrate the pre-buckling and post-debond configurations of a patterned metal film subjected to thermally induced compressive stress. Upon heating, aluminum metal lines debond. The debond occurs at temperatures that are expected to be high enough to cause plastic yielding of the film [10], [13], [14]. The peak out-of-plane displacements were measured by utilizing the fact that as the magnification increases, the depth of focus decreases. Thus, due to a low depth of focus, the top surface and the edge of the debonded film in Figure 2 can be identified from their depth of focus under the optical microscope. Therefore, the top surface and the edge of the debonded film were focussed successively at a magnification of 500X. The corresponding distance (which in turn is equal
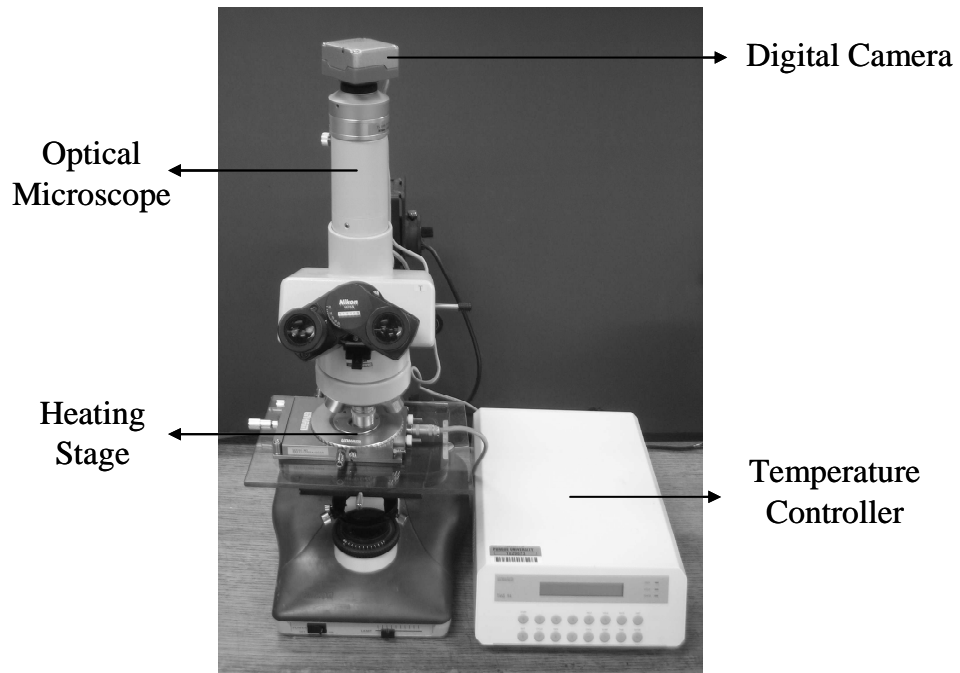
Fig. 1.   Test setup consisting of optical microscope, heating stage and temperature controller [12].
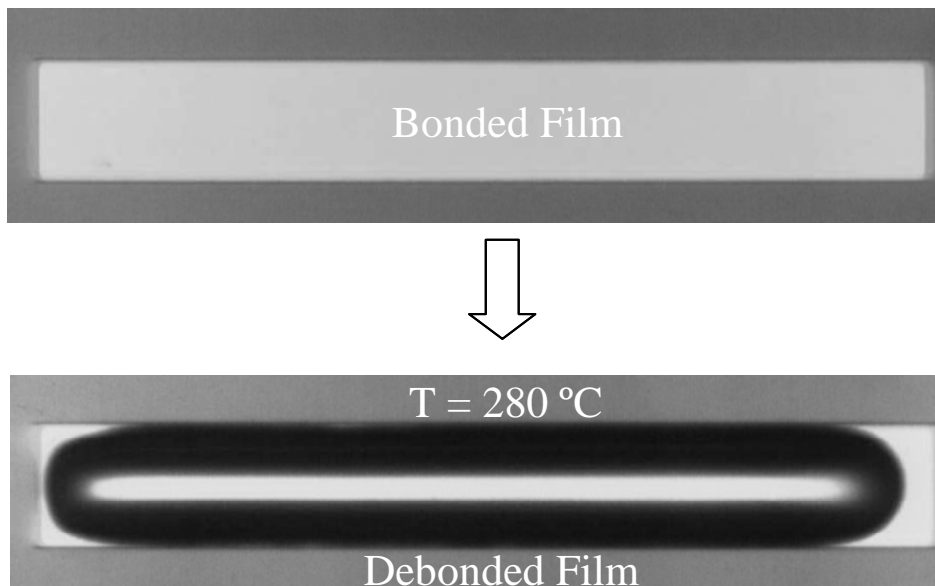

Fig. 2.   Buckling-induced debond in a thermally loaded aluminum metal line.

to the peak deflection of the debonded film) moved by the objective lens was calculated from the number of divisions (rotations) made by the fine focus knob. The distance moved by the objective lens was 1 μm for a rotation equal to 1 division on the fine focus knob. Therefore, the peak deflection that was measured was equal to the number of divisions moved by the focus knob.

B. Analytical Model

To estimate the yield strength we make use of the dependence of out-of-plane deflections on the yield strength through an elastic-plastic film deflection model motivated by reference [12]. We assume that the cross-sectional width of the film shown in Figure 2 as being $2D$ with a debonded region $2d$ wide as shown in Figure 3. The elastic, plastic strain-hardening film material has a Young's Modulus E, yield strength σ_Y, and hardening modulus H as shown in Figure 4. The film is loaded in compression by a stress σ induced by a differential thermal expansion.
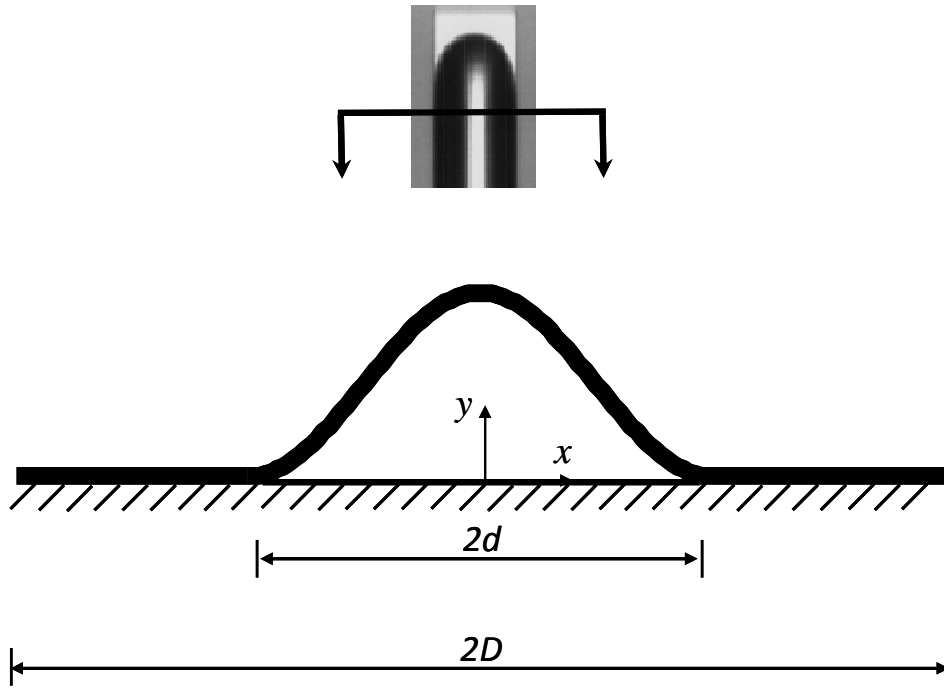

Fig. 3.   Schematic illustration of the cross-section of the debonded line.
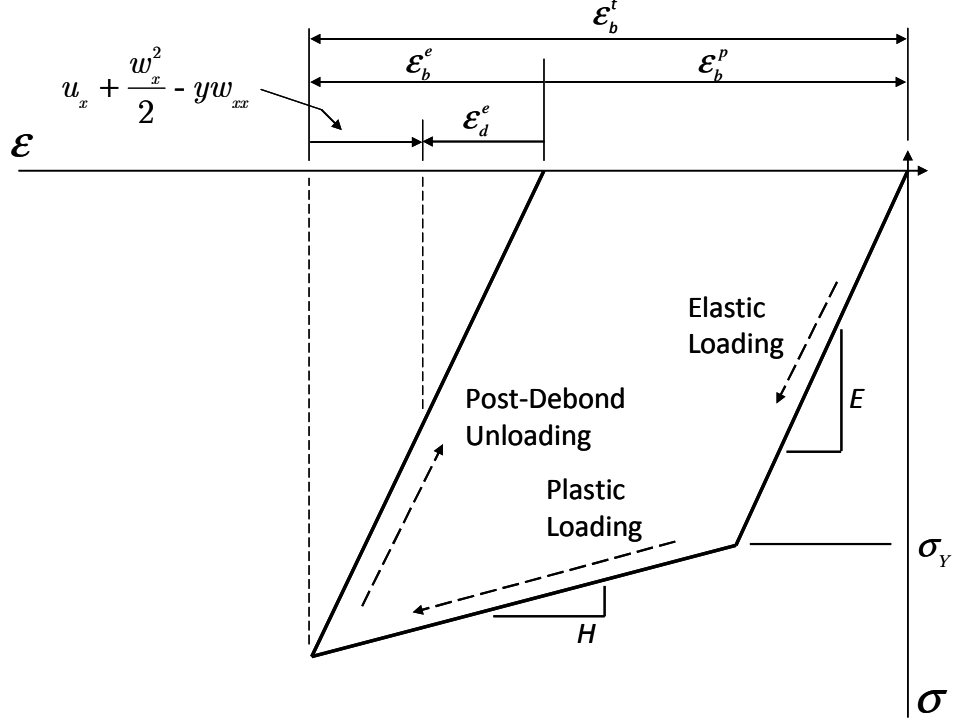
Fig. 4. The elastic plastic model utilized in the analytical description. The possesses a total strain of $\varepsilon_b^t = \Delta\alpha\Delta T$, which is composed of the elastic strain portion ε_b^e and the plastic strain portion ε_b^p. Upon debonding, the film relieves membrane stress, but increases bending stress. resulting in a net elastic strain of ε_d^e.

The analysis begins by considering a pre-existing debond of width $2d_i \ll 2d$. The stress in the film when the debond initiates is σ_d. The stress σ_d is potentially equal to or larger than the yield strength σ_Y of the film material. Therefore, the film is in a plastic state at the point when the debond initiates and remains so as the debond further propagates due to further loading. The fracture toughness ahead of the debonded region is Γ and the debond is assumed to propagate steadily.

Under conditions of steady debond propagation, the debonded region is under quasi-static equilibrium that corresponds to minimum free energy of the film column and the interface system. The configuration of minimum free energy is that at which the Helmholtz free energy of the column including a contribution from the surface energy corresponding to the region to be debonded is minimized [15]. The energy minimization principle will now be used to determine the most energetically favorable configuration, or the condition dictating steady state propagation.

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Let the in-plane displacements and out-of plane displacements of the debonded region be described by the functions $u(x)$ and $w(x)$ respectively. Hence, the configuration of the debonded region is described by $u(x)$, $w(x)$ and the debond length $2d$. For a partially-debonded film column, the free energy of the system is composed of three different parts: Helmholtz free energy stored in the bonded region, ψ_b, Helmoltz free energy stored in the debonded region, ψ_d, and the adhesion energy corresponding to the created free surface, $2\Gamma d$, which is the surface energy contribution to the total free energy of the system. That is the total free energy of the system is:

$$\psi = \psi_b + \psi_d + 2\Gamma d \tag{1}$$

Let the total strain in the bonded part of the film corresponding to the stress σ be given by ε_b^t. This strain is assumed to be decomposed into an elastic strain, ε_b^e and a plastic strain ε_b^p as:

$$\varepsilon_b^t = \varepsilon_b^e + \varepsilon_b^p \tag{2}$$

Then, the Helmholtz free energy stored in the bonded region of the film of total volume Ω is related to the stored recoverable energy in the film:

$$\psi_b = \int_\Omega \frac{E}{2}(\varepsilon_b^e)^2 \, d\Omega \tag{3}$$

and the elastic strain in the bonded film is given by:

$$\varepsilon_b^e = \frac{\sigma}{E} = \frac{1}{E}\left[\sigma_Y + \left(\varepsilon_b^t - \frac{\sigma_Y}{E}\right) H\right] \tag{4}$$

The yield stress σ_Y may be positive or negative depending on the loading direction. The total strain is the result of the thermal strain, that is, $\varepsilon_b^t = \Delta\alpha\Delta T$. This is expected to be a negative quantity due to the fact that the film is expected to expand at a higher rate compared to the rigid silicon substrate. The total strain at the debond temperature excursion, ΔT_d, is given by $\varepsilon_b^t = \Delta\alpha\Delta T_d$ where $\Delta\alpha$ is the difference in coefficient of thermal expansion between the film and the silicon substrate.

The free energy in the debonded region is related to the elastic strain energy given by:

$$\psi_d = \int_\Omega \frac{E}{2}(\varepsilon_d^e)^2 \, d\Omega \tag{5}$$

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where, the elastic strain in the debonded film at temperatures greater than the debond temperature is the difference between the thermally induced compressive strain and the relief due to bending:

$$\varepsilon_d^e = \Delta\alpha\left(\Delta T - \Delta T_d\right) + \varepsilon_b^e - \left(u_x + \frac{w_x^2}{2} - yw_{xx}\right) \tag{6}$$

In the above equation, $\Delta\alpha\left(\Delta T - \Delta T_d\right)$ is the additional elastic strain that is imposed at temperatures greater than the debond temperature. We assume here that additional elastic strain is equal to the additional total strain $\varepsilon_e^t = \Delta\alpha\left(\Delta T - \Delta T_d\right)$ or that the temperature rise beyond debond is insufficient to load the film plastically. Using the fact that the out-of-plane deflection is of the form $w\left(x\right) = \frac{w_0}{2}\left[1 + \cos(\frac{\pi x}{d})\right]$, substituting Eq. (6) into Eq. (5) and minimizing the free energy, it is easy to show [12] that the minimum energy in the debonded portion is obtained when:

$$\begin{aligned}
w_0 &= t\sqrt{\frac{4}{3}\left[\frac{\Delta\alpha\left(\Delta T - \Delta T_d\right) + \varepsilon_b^e}{\varepsilon_{cr}} - 1\right]} \\
&= t\sqrt{\frac{4}{3}\left[\frac{\Delta\alpha\left(\Delta T - \Delta T_d\right) + \frac{1}{E}\left[\sigma_Y + \left(\Delta\alpha\Delta T_d - \frac{\sigma_Y}{E}\right)H\right]}{\varepsilon_{cr}} - 1\right]}
\end{aligned} \tag{7}$$

where, w_0 is the peak out-of-plane deflection, and ε_{cr} is the elastic critical buckling strain given by

$$\varepsilon_{cr} = -\frac{\pi^2 I}{td^2} \tag{8}$$

where, $I = t^3/12$ and t is the thickness of the film. Once w_0 is measured, σ_Y can be calculated from Eq. 7. If the debond occurs over the entire width, then the value of the critical strain is $\varepsilon_{cr} = -\frac{\pi^2 I}{tD^2}$.

III. RESULTS AND DISCUSSION

The peak deflections of the debonded aluminum film for all of the line widths considered here were measured at a temperature of 282 °C, which was greater than the debond temperature. The measured peak deflections are given in Table I. To estimate the peak deflection using Eq. (7), the following values were used: $t = 2.5\ \mu m$, $E = 70$ GPa, $H = 7$ GPa, $\Delta\alpha = 22\times10^{-6}$ °C^{-1}, $\Delta T = 282 - 30 = 252$ °C, where the reference temperature for thermal expansion was 30 °C. Similarly, ΔT_d was estimated by subtracting the reference temperature from the debond temperature listed in Table I. The yield strength calculated using the above values are listed in Table I.

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TABLE I

PEAK DEFLECTIONS AND YIELD STRENGTH OBTAINED FOR DIFFERENT LINE WIDTHS.

Line Width, $2D$, (μm)	Debond Temperature, T_d, (°C)	Peak Deflection, w_0, (μm)	Yield Strength, σ_Y, (MPa)
600	282	17	60
700	282	21	72
800	258	27	64
1000	254	34	59

The estimated average yield strength of the aluminum film was 64 MPa with a standard deviation of 6 MPa.

There are several plausible sources of uncertainty associated with the measured yield strength. The first possible source of uncertainty is the bilinear elastic-plastic model used in this study, and one that is very commonly used in practice for metals. The form of the model used in the present study is consistent with the stress-strain curve illustrated for aluminum thin films in the references [16], [17]. However, any deviation of the actual material behavior would induce uncertainty in the estimated yield strength. Another possible source of uncertainty is the error induced by creep in the measured deflections listed in Table I. The measurements were made at a temperature of 555 K (282 °C), which is greater than half the homologous temperature of Aluminum. Potentially this temperature could cause creep of the film, which could alter the measured peak deflection if significant time elapses between occurrence of buckling and the time of observation. Lastly, the form of the trial function used for the deflection, as a cosine function, is an exact solution to the buckling problem that takes into account bending and large axial strains but not the shear strains. A common assumption in mechanics of thin plates is to ignore shear strains (i.e., it is justified to do so) when the ratio of the length to the thickness of the film is very large (in other words because of the thin film assumption). As film thickness increases, the form of the trial function may induce uncertainty.

The value of yield strength obtained in this study and reported above lies within the range of values reported for aluminum films in the literature. The yield strength of aluminum films generally shows variability dependent on factors such as the temperature, grain size, purity, cold working, and film thickness. For bulk aluminum, yield strength values are observed to lie in the

range 15-140 MPa if the purity of Aluminum is 99.999% and between 30-280 MPa if the purity level is 99-99.7% [13]. Specifically, for 2 μm thick films, the values of yield strength that have been reported at room temperature are 140 MPa [6] for sputtered aluminum films and 124 MPa [18] for evaporated aluminum films. However, at a temperature of 300 °C a value of 60 MPa was reported for 2 μm thick sputtered aluminum films by Edison [10], which is consistent with the values reported here.

The proposed experiment can be conducted such that the temperature dependence of the yield strength is investigated. Such a sequence of experiments can be designed by modification of the adhesion conditions of the aluminum film. If the adhesion between film and substrate is low, then debonding will occur early on at a low temperature, while for stronger adhesion values, a measurement of yield strength at higher temperatures is achieved. Similarly, the appropriate selection of the line width can be performed with similar goals in mind. Thereby, a lower limit on the line width should be avoided as the shear lag effect will lead to lowered in-plane stresses once a critical film thickness is surpassed [12].

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