Size effects on the mechanical behavior of gold thin films

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The Membrane Deflection Experiment was used to test the mechanical response of freestanding thin film gold specimens. We present stress-strain curves obtained on films 0.3, 0.5, and 0.1 μm thick. Elastic modulus was consistently measured in the range of 53–55 GPa. Several size effects on the mechanical properties were observed including yield stress variations with membrane width and film thickness. It was observed that a width of 2.5 μm and a thickness of 0.5 μm correspond to major transitions in the material deformation behavior. © *2003 Kluwer Academic Publishers*

1. Introduction
1.1. Thin films & MEMS
Thin films with thickness of a few microns or a fraction of a micron are employed as components in MEMS and microelectronic devices. These films frequently serve as essential device functions. The demands placed on thin films in these applications can sometimes subject them to various mechanical conditions, such as; plasticity, friction and wear, creep, fatigue, etc. It is well known that methods to describe bulk material behavior fail to describe material response in this size regime. Many researchers currently have experimental programs to study such characteristics [1–4]. Frequently, each particular investigation involving microdevices tends to be device dependent and introduces new fundamental questions. For instance, substrate material and etching techniques play a major role on film's grain structure as well as the presence of initial defects.

1.2. Size effects
Over the past decade, there has been great motivation to reduce the size of many mechanical systems to the micron and sub-micron scale by fabricating devices out of thin film materials. Whatever the application, successful development of a thin film material requires a through understanding of its mechanical properties. The quality and mechanical response of these films depends on many factors. Of principal issue is the existence of film thickness effects that arise because of geometrical constraints on dislocation motion. This paper uses the Membrane Deflection Experiment to examine size effects in polycrystalline thin film gold [5–8].

2. Experimental procedure
2.1. Sample design
Specially designed thin film gold specimens were microfabricated on (100) Si wafers. Specimen shape was defined on the topside by photolithography and lift off. On the bottom side windows were etched through the wafer, underneath the specimens, with the purpose of creating freestanding membranes. The geometry of the membranes can be described best as a double dog-bone tensile specimen. A more detailed description of their fabrication and shape is given in Espinosa and Prorok [8]. Fig. 1 shows an optical image and a schematic representation of the Au membranes. Membrane size was varied in scale to preserve the geometry. This geometry was chosen to minimize stress concentrations and boundary-bending effects. Samples were fabricated such that 4 differently sized membranes had identical geometry, but with different characteristic dimensions.

2.2. Mechanical testing
The Membrane Deflection Experiment (MDE) was used to achieve direct tensile stressing of the specimens [5, 8]. The procedure involves applying a line load, using a nanoindenter, at the center of the span. Simultaneously, an interferometer focused on the bottom side of the membrane records the deflection. The result is direct tension in the gauged regions of the membrane with load and deflection being measured independently.

The data directly obtained from the MDE test must then be reduced to arrive at a stress-strain signature for the membrane. The load in the plane of the membrane is found as a component of the vertical nanoindenter load by the following equations,

$$\tan \theta = \frac{\Delta}{L_M} \quad \text{and} \quad P_M = \frac{P_V}{2 \sin \theta}, \qquad (1)$$

where (from Fig. 2) θ is the angle of deflection, Δ is the displacement, L_M is half the membrane length, P_M is the load in the membrane plane, and P_V is the load measured by the nanoindenter. Once P_M is obtained, the Cauchy stress, $^t\sigma$, can be computed from,

$$^t\sigma = \frac{P_M}{A}, \qquad (2)$$

where A is the cross-sectional area in the gauge region.

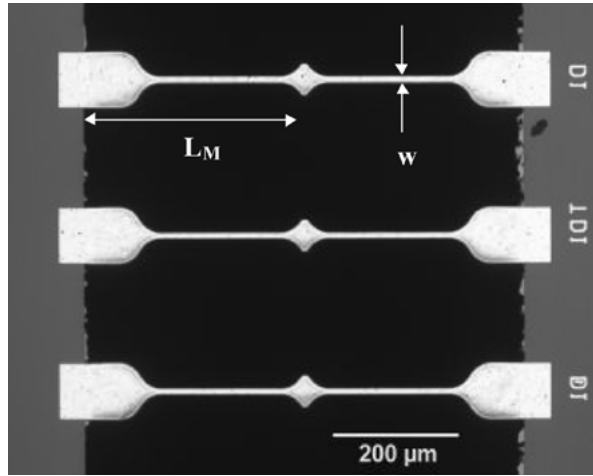The interferometer yields vertical displacement information in the form of monochromatic images taken

Figure 1 Optical image of three Au membranes. L_M is half the membrane span, and W is the membrane width.
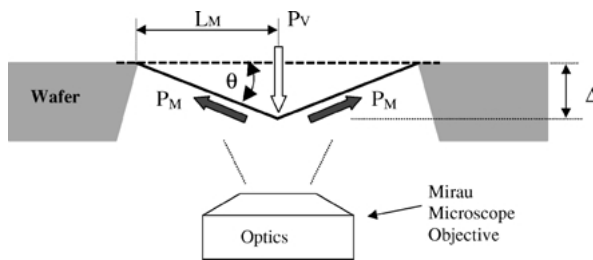

Figure 2 Side view of the MDE test showing vertical load being applied by the nanoindenter, P_V, the membrane in-plane load, P_M, and the position of the Mirau microscope objective.
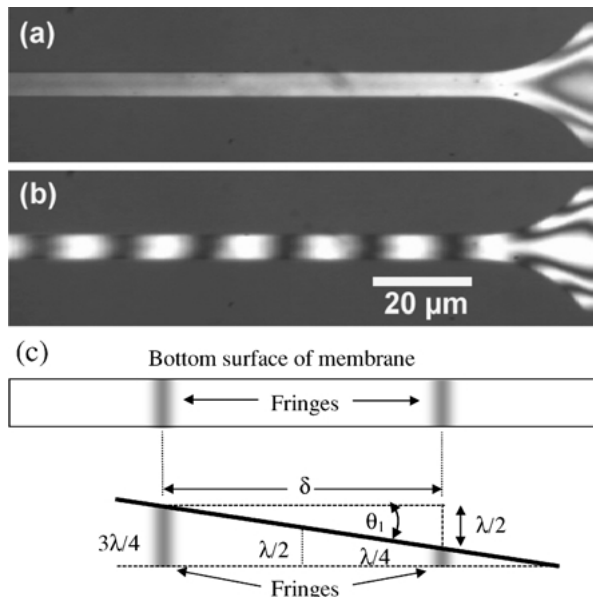

Figure 3 Monochromatic images of the membrane's bottom side showing an unloaded membrane (a) and a loaded membrane (b). The relationship between the distance between fringes, δ, and vertical displacement is shown in (c).

at periodic intervals. Fig. 3 shows two such images for a membrane before loading (a) and in the process of being loaded (b). The relationship between the distance between fringes, δ, and vertical displacement is shown in the schematic in Fig. 3c. Assuming that the membrane is deforming uniformly along its gage length, the relative

deflection between two points can be calculated, independently of the nanoindenter measurements, by counting the total number of fringes and multiplying by $\lambda/2$. Normally part of the membrane is out of the focal plane and thus all fringes cannot be counted. To solve this problem we can find the average distance between the number of fringes that are in the focal plane. Using the geometrical relations shown in Fig. 3c an overall strain, $^t\varepsilon$, for the membrane can be computed from the following relation,

$$^t\varepsilon = \frac{\sqrt{\delta^2 + (\lambda/2)^2}}{\delta} - 1 \qquad (3)$$

This relationship is valid when deflections and angles are small. Large angles require a more comprehensive relation to account for the additional path length due to reflection off of the deflected membrane. This relation is derived in Espinosa and Prorok [8]. For this study, deflection angles in all four membrane sizes are small and thus the above Equation 3, is used.

3. Results and discussion
3.1. MDE repeatability
Fig. 4a and b are load-displacement and stress-strain plots, respectively, of 5 thin film gold membranes of identical size and shape. These data offer testimony to the repeatability and reliability of the MDE test for examining mechanical properties of thin film specimens. The stress-strain curves indicate that Young's modulus is consistent at 53–55 GPa and that each membrane
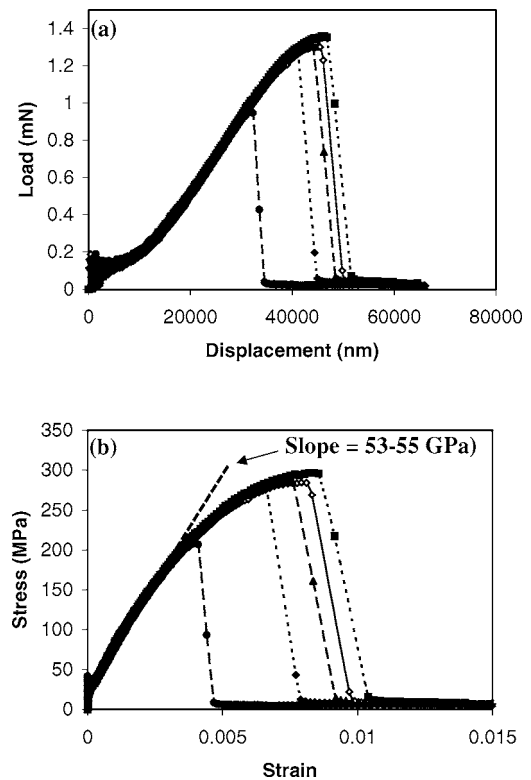

Figure 4 Load-displacement (a) and stress-strain (b) curves for 5 thin film gold membranes of identical size and shape showing the repeatability and reliability of the Membrane Deflection Experiment in evaluating thin film mechanical response.

behaves in the same manner in the elastic region as well as in the plastic regime. Failure is seen to occur at different levels of stress and is related to defect size and generation statistical effects.

3.2. Effect of membrane width

Fig. 5a and b are stress-strain curves for gold films 0.3 and 1.0 μm thick, respectively. Each plot shows the effect of membrane width on mechanical response for widths of 2.5 to 20 μm. The individual signature of each membrane width represents the average curve of 5 membranes of identical size and shape. The 0.3 μm thick specimens have a well defined elastic regime with a Young's modulus of 53–55 GPa. The specimens of widths 5, 10, and 20 μm exhibit nearly identical behavior with some variability in failure strain. The yield stress for these three widths was found to be 160–170 MPa. The 2.5 μm width membrane shows an extended elastic zone and a larger yield stress of 220 MPa. Its overall stress-strain signature rises slightly above the membranes of larger width. An explanation of this behavior may come from the knowledge that yield stress of polycrystalline thin films is a function of its crystallographic texture [9, 10]. Due to their small thickness, the membranes likely have some degree of cube texturing, i.e., the x-y plane of the film contains grains oriented in {001}⟨100⟩. Further reducing a dimension such as width may cause secondary texturing to occur, i.e., grains are not only oriented as {001}⟨100⟩ but also align along a direction in that plane. Another possibility is the effect of further geometrical constraints on deformation processes with reduction in number of grains within the specimen width.
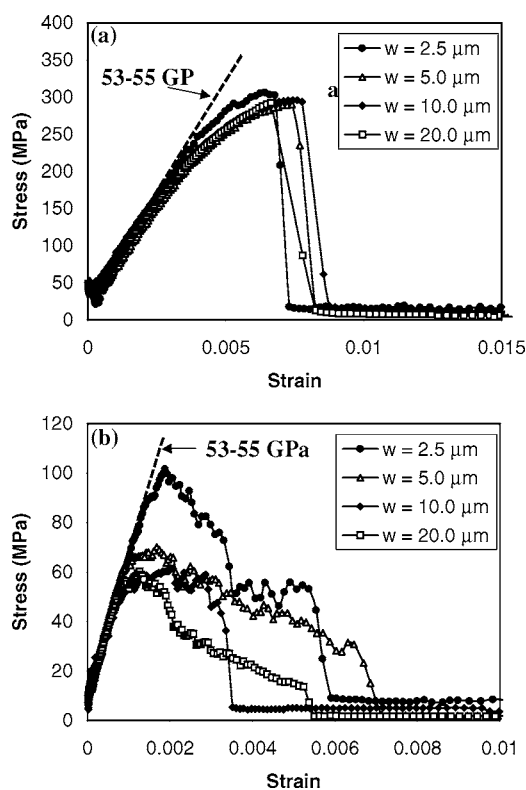
The 1.0 μm thick specimens exhibited distinctly different deformation and failure behavior. A significant change in mechanical response occurs as the films begin to plastically deform. Behavior changes such that failure is not a sharp transition but rather undergoes a period of gradual decrease in stress then followed by sharp decrease. Also present are the appearance of sharp undulations in stress that resemble *Lüders bands* observed in the deformation of some bulk materials. This indicates that plastic yielding happens in a discrete manner. Young's modulus for the 1.0 μm thick specimens was found to be 53–55 GPa, consistent with the 0.3 μm thick specimens. However, values of yield stress were significantly lower than those for thinner films and varied to some extent with width. At a width of 2.5 μm the yield stress was 90 MPa and it decreased to 65 MPa at 5.0 μm width and then to 55 MPa for 10 and 20 μm widths.

3.3. Effect of thickness

Thickness effects are examined by plotting stress-strain curves obtained from membranes of the same width. Fig. 6a and b are stress strain curves for membranes of widths 2.5 and 20 μm, respectively. Each show the stress strain curve for film thickness of 0.3, 0.5, and 1.0 μm. For the 2.5 μm width the 0.3 and 0.5 μm thick films yield nearly identical behavior while the deformation and failure of the 1.0 μm thick film is clearly governed by different deformation processes. As width is increased to 20 μm the stress-strain curve for the 0.5 μm film drops below that of the 0.3 μm film corresponding to a reduction in the onset of plastic deformation. The

Figure 5 Stress-strain curves for gold films 0.3 (a) and 1.0 μm (b) thick showing the effect of width on mechanical response.
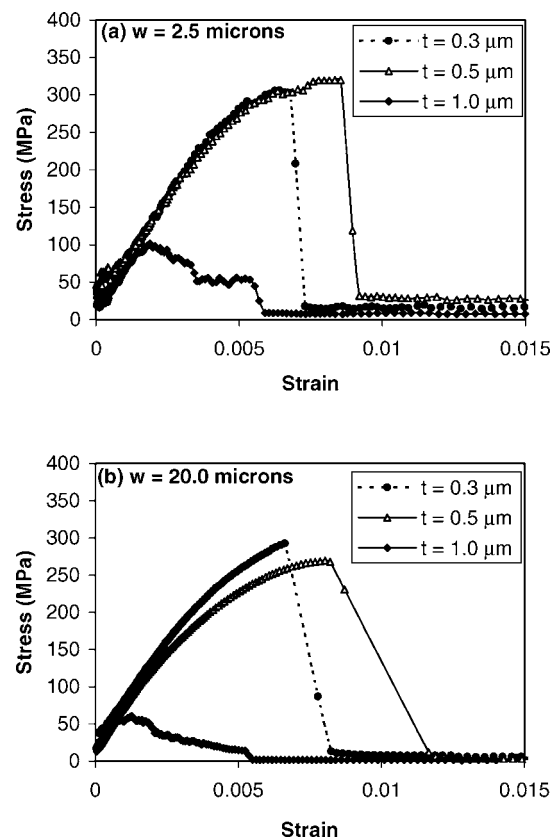

Figure 6 Stress-strain curves for membranes 2.5 (a) and 20 μm (b) in width showing the effect of film thickness.

1.0 μm thick film also shows a significant drop in yield stress.

Given that all membranes of varying size and shape behave identically in the elastic region it is clear that the specimen size has an effect on mainly the plastic deformation behavior. It is interesting to note that the Young's modulus was consistent at 53–55 GPa, which is significantly lower than the value of 78 GPa for bulk Au; however, values reported for thin film Au varied from 30–78 [11]. It does however match well with the modulus measured for nanocrystalline Au of 55 GPa [12].

4. Conclusions

The Membrane Deflection Experiment was used to evaluate size effects on the mechanical response of suspended thin film Au membranes. Young's modulus was measured repeatedly at 53–55 GPa. Film thickness and width was shown to significantly alter the stress-strain behavior of the membranes causing substantial changes in yield stress and film failure.

Acknowledgments

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