Effects of Film Thickness on the Yielding Behavior of Polycrystalline Gold Films

H.D. Espinosa and B.C. Prorok
Department of Mechanical Engineering, Northwestern University
Evanston, IL 60208-3111, USA

ABSTRACT

A 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 1.0 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 thickness plays a key role in deformation behavior with a major transition in the material inelastic response occurring between a thickness of 0.5 and 1.0 µm. The size effects here reported are the first of their kind in the sense that the measurements were performed under a macroscopically homogeneous axial deformation, i.e., in the absence of macroscopic deformation gradients.

INTRODUCTION

Thin films with thickness of a fraction of a micron are commonly employed in microelectromechanical systems (MEMS) and microelectronic devices. These films frequently serve essential device functions. In these applications, the films are subjected to various thermo-mechanical conditions that may result in fracture, plasticity, friction and wear, creep, fatigue, etc. It is well known that most knowledge of bulk material behavior fails to describe material response in the submicron size regime. Hence, several groups have experimental and theoretical programs to study such features [1-4].

Frequently, investigations involving microsystems tends to be device dependent. For instance, substrate material and etching techniques play a major role on film grain structure as well as the presence of initial defects. Whatever the application, successful development of a thin film material requires an in-depth understanding of its mechanical properties through carefully designed experiments and their analysis. The quality and mechanical response of these films depends on many factors. Of relevance is the existence of film thickness and width effects that arise because of geometrical constraints to motion of defects. This paper uses the Membrane Deflection Experiment developed by Espinosa and co-workers to examine size effects in polycrystalline thin film gold specimens [5-9].

EXPERIMENTAL PROCEDURE

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 suspend membranes. The geometry of the suspended thin-film membranes can be described best

as a double dog-bone tensile specimen. A detailed description of their fabrication and shape is given in Espinosa and Prorok [8]. Figure 1 shows an optical image of the Au membranes. Membrane in-plane dimensions were varied but geometrical ratios were preserved. The geometry was chosen to minimize stress concentrations and boundary-bending effects. Four differently sized membranes, described by a characteristic width of the gauged region, were fabricated, namely, 2.5, 5, 10 and 20 µm. Membrane thickness was also varied at values of 0.3, 0.5, and 1.0 µm.

The Membrane Deflection Experiment (MDE) [5-9] was used to achieve direct tensile stressing of the specimens. The procedure involves applying load with a nanoindenter to the center of the spanning membrane. Simultaneously, an interferometer focused on the bottom side of the membrane records the deflection (see Figure 2). The result is direct tension in the gauge regions of the membrane with load and deflection being measured independently. Details of the data reduction procedure are given in Espinosa and Prorok [8].

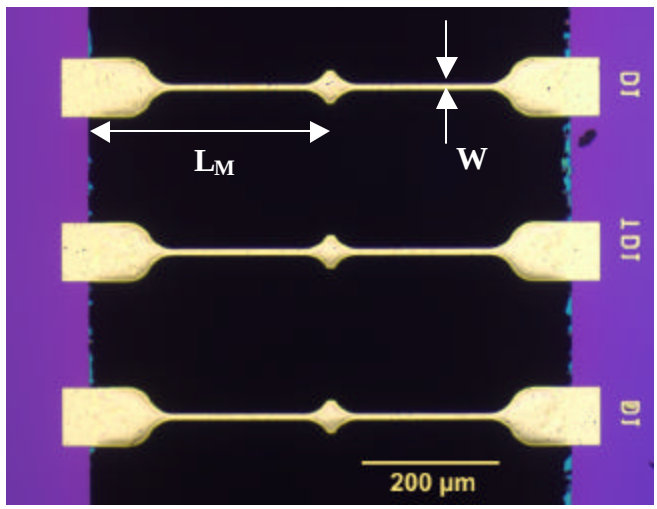


Figure 1. Optical image of three gold membranes within a die. L_M is half the membrane span and W is the membrane width.

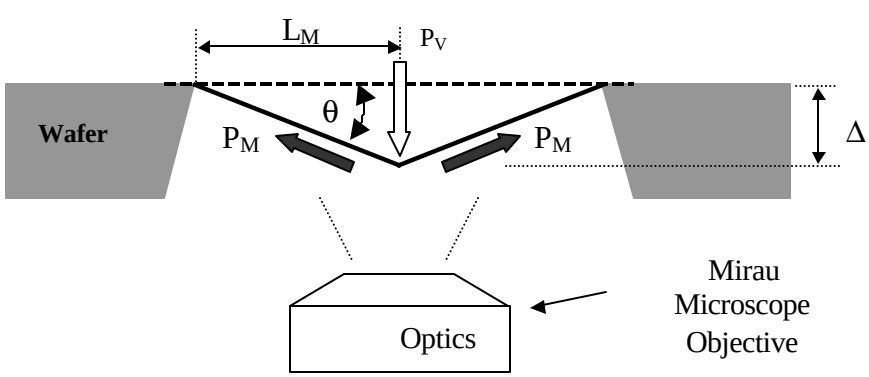


Figure 2. Side view of the MDE test showing vertical load being applied by the nanoindenter, P_V, the membrane in-plane load, P_M, and the position of the Mirau microscope objective. Δ is the deflection and θ is the angle of rotation.

RESULTS AND DISCUSSION

The microstructure of the thin film gold consisted of grains 300 nm in diameter. Since the film thickness is on the order of the grain size this translates to films of a columnar or bamboo-like structure. The standard explanation of this effect is the balance between high surface energy and grain boundary curvature. Membranes of different widths also had a characteristic numbers of grains across their width. Due to their small thickness, the membranes likely have some degree of texturing, i.e., the high surface energy of the 300 nm grains will cause preferential orientation through energy minimization. The yield stress of polycrystalline thin films can separately depend on grain size, film thickness, and crystallographic texture [10,11]. Thus, all the above characteristics are expected to play a role.

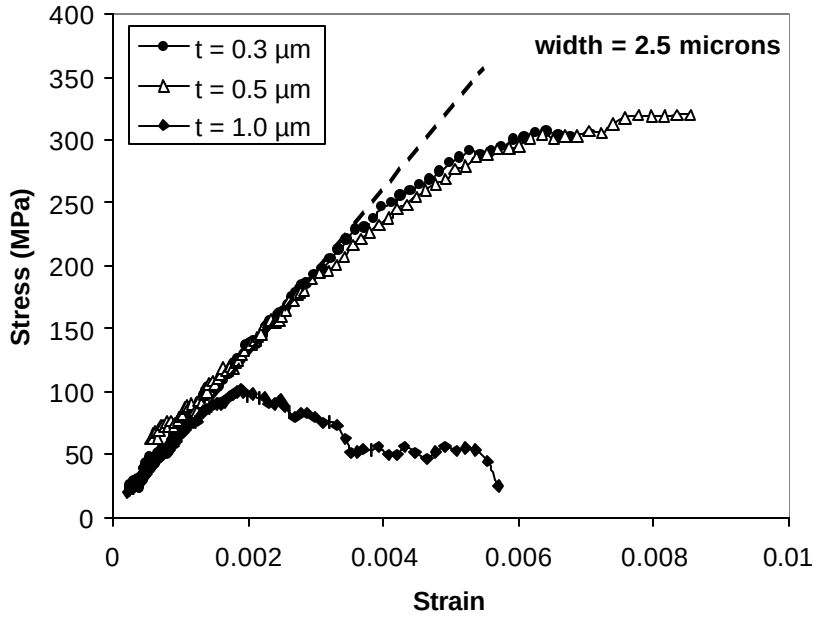
Figures 3 (a), (b), (c) and (d) are stress-strain plots obtained from membranes having a characteristic width of 2.5, 5, 10 and 20 µm, respectively. Contained in each plot are three curves each representing a thickness of 0.3 (●), 0.5 (△) and 1.0 µm (◆) where each curve is the average of five membranes of identical size and shape. The 2.5 µm wide specimens exhibited a distinct mechanical response. The curves for membranes of thickness 0.3 and 0.5 µm show nearly identical elastic and plastic behavior with the exception that the 0.5 µm membranes had a larger failure strain, possibly the result of having a larger cross-sectional area. Both possessed a Young's modulus of 53-55 GPa and a yield stress of 220 MPa. Likely, plastic deformation occurred by the same mechanisms in both cases. As thickness was increased to 1.0 µm, deformation behavior significantly changed. Young's modulus was measured at 53-55 GPa, however, yield stress decreased considerably to 90 MPa and plastic deformation occurred mostly in discrete quantities with an overall continuous decrease in stress to failure. Since grain size is around 300 nm the 1.0 µm membranes have a high probability of having more then one grain through the thickness; hence, relaxing constraints imposed on the deformation mechanisms. This feature is currently under investigation.

Stress-strain plots for a membrane width of 5.0 µm are shown in Figure 3 (b). As membrane width was increased the yield stress decreased to 170 MPa for the 0.3 and 0.5 µm thickness, possibly a result of having roughly twice as many grains across the width. In the 1.0 µm membrane the yield stress decreased to 65 MPa. It appears that in this membrane there is a mixed effect between an increase in the number of grains across the width and through the thickness to further relax the geometrical constraints on deformation mechanisms.

As membrane width is increased to 10 and 20 µm similar behavior is observed. Increases in both width and thickness further reduce the onset of plastic deformation. Table 1 lists the yield stresses for each combination of thickness and width. These data indicate the existence of two behavioral regimes with thickness or width effects acting independently or in combination. The first regime is where membrane dimensions are their smallest, width = 2.5 µm and thickness = 0.3 µm. Here, the onset of plastic yielding is the highest with grain structure likely consisting of one grain through the thickness and 8 to 10 grains across the width.

The second behavioral regime exists at the upper end of the membrane dimensions, width = 20 µm and thickness = 1.0 µm. Here, the onset of plasticity is the lowest with the grain structure consisting of approximately 66-80 grains across the width and more than one grain through the thickness. The grain assembly begins to resemble a representative volume element of polycrystalline bulk material and, therefore, it results in a reduced yield point by allowing more slip systems to become active. Of the two changes, increasing thickness above 0.5 µm appears to have the greatest effect in softening the material, although, both effects are significant.

(a)



(b)

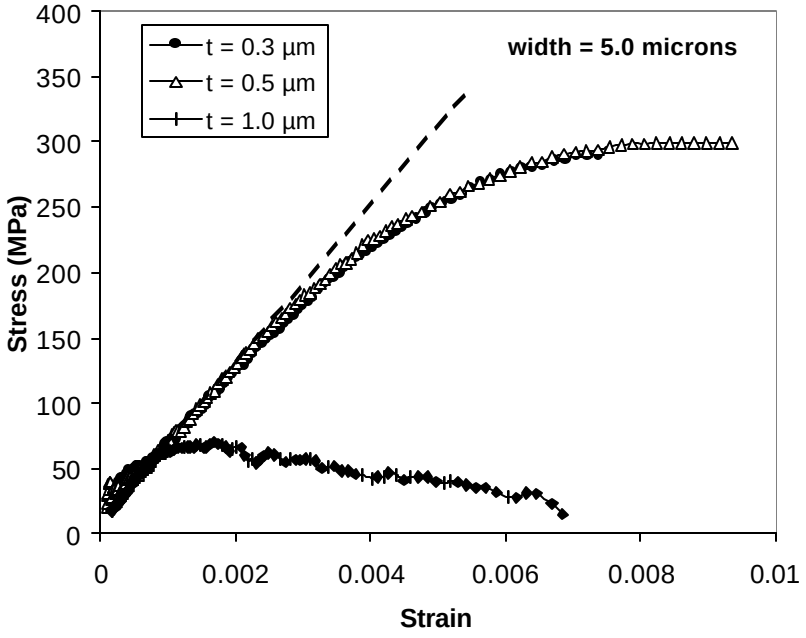
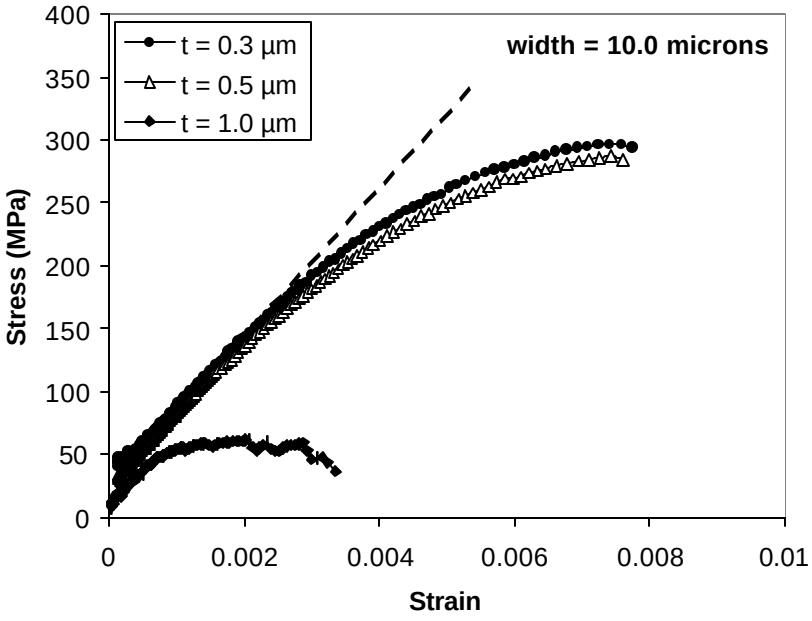


Figure 3. Stress-strain curves, for membranes 2.5 (a) and 5 µm (b) in width, as a function of film thickness. The slope of the dashed line is 55 GPa.

(c)



(d)

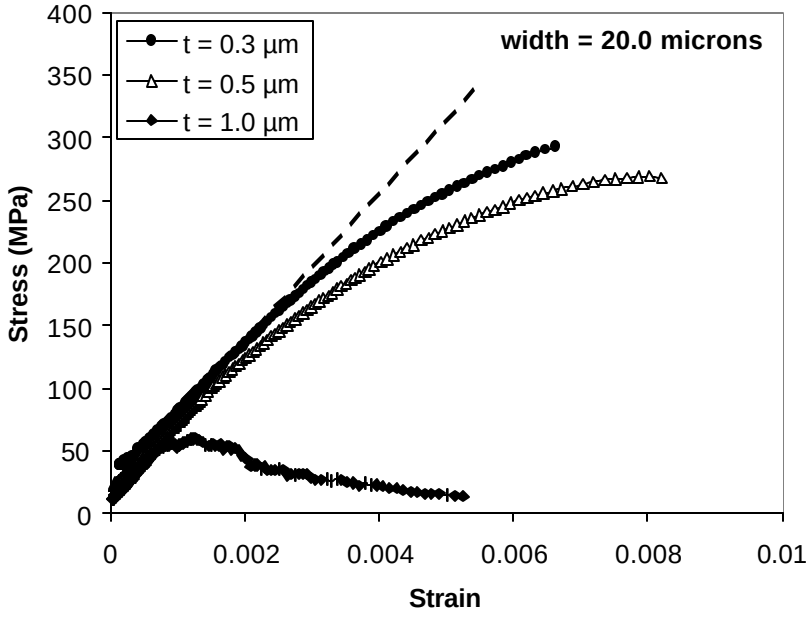


Figure 3. Stress-strain curves, for membranes 10 (c) and 20 µm (d) in width, as a function of film thickness. The slope of the dashed line is 55 GPa.

Table 1. Yield stresses for each combination of thickness and width.

Thickness ↻	**Width ➲**	**2.5 µm**	**5 µm**	**10 µm**	**20 µm**
0.3 µm		220 MPa	170 MPa	170 MPa	170 MPa
0.5 µm		220 MPa	170 MPa	170 MPa	140 MPa
1.0 µm		90 MPa	65 MPa	55 MPa	55 MPa

Given that all membranes of varying size behaved identically in the elastic regime it is clear that the specimen size has only an effect on the inelastic deformation behavior. It is interesting to note that the Young's modulus was consistently measured between 53-55 GPa in more than 100 tested membranes. This value is significantly lower than the bulk value of 78 GPa; however, values reported for thin film Au varied from 30-78 [12]. Our measurement does correlate well though with other measured values of Yong's modulus, 50-57 GPa, as reported in [13-15].

CONCLUSIONS

The Membrane Deflection Experiment [5-9] was used to evaluate size effects on the mechanical response of suspended thin film Au membranes. Young's modulus was consistently measured at 53-55 GPa. Film thickness and width were shown to significantly alter the stress-strain behavior of the membranes causing substantial changes in yield stress and film failure.

ACKNOWLEDGMENTS

This work was sponsored by the National Science Foundation under Career Award No. CMS-9624364 and the ONR-Young Investigator Program, Award No. N00014-97-1-0550.

REFERENCES

1. I. Chasiotis and W. Knauss, Proc. of SPIE - The Int. Soc. for Optical Eng. **3512**, 66 (1998).
2. M. Drory and J. Hutchinson, Mat. Res. Soc. Symp. Proc. **383**, 173 (1995).
3. A G. Evans, M. Y. He, and J. W. Hutchinson, Acta Mater., **45** (9), 3543 (1997).
4. H. Huang and F. Spaepen, Mat. Res. Soc. Symp. Proc., **405**, 501 (1996).
5. M. Fischer, Master Thesis, Purdue University, West Lafayette, IN (1999).
6. H.D. Espinosa, M. Fischer, Y. Zhu, and S. Lee, Proc. of the 4th Int. Conf. on Modeling and Simulation of Microsystems, M. Laudon and B. Romanowicz editors, 402 (2001).
7. H.D. Espinosa, B.C. Prorok, and M. Fischer, Proc. of the SEM Ann. Conf. on Experimental and Applied Mechanics, June 4-6, 2001, Portland, Oregon, 446 (2001).
8. H.D. Espinosa and B.C. Prorok, submitted to J. Mech. Phys. Solids. (2001).
9. H.D. Espinosa and B.C. Prorok, Proc. of the Symp. on Mech. Prop. of MEMS Structures, the 2001 Int. Mech. Eng. Congress in New York, New York, November 11-16 (2001).
10. J.E. Sanchez and E. Arzt, Scripta Metall. Mater. **27**, 285 (1992).
11. C.V. Thompson, J. Mater. Res. **8** (2). 237 (1993).
12. W. Nix, Met. Trans. A **20A**, 2217 (1989).
13 T.P. Weihs, S. Hong, J.C. Bravman, and W.D. Niz, J. Mater. Res. **3** (5), 931 (1988).
14 D.M. Schaefer, A. Patil and R. Reifenberger, Appl. Phys. Lett. **63** (11), 1492 (1993).
15. S. Okuda, M. Kobiyama, and T. Inami, Mater. Trans., JIM **40** (5), 412 (1999).