THE USE OF MICROMACHINED STRUCTURES FOR THE MEASUREMENT OF MECHANICAL PROPERTIES AND ADHESION OF THIN FILMS

Mehran Mehregany, Mark G. Allen, and Stephen D. Senturia

Microsystems Technology Laboratories
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139

This paper reports the application of silicon micromachining to the measurement of mechanical properties of thin films such as intrinsic stress, Young's modulus, and adhesion. The measurement is based on the deflection and subsequent peeling of suspended membrane sections of the film. The original goal of the work was to make a quantitatively reproducible adhesion test by applying micromachining techniques to the blister peel test described by Hinkley [1]. Our initial measurements demonstrated the importance of residual stress in the films, which resulted in an expanded emphasis on the basic mechanical properties of the membrane as a prelude to accurate adhesion measurements. We will briefly discuss the process for micromachining suspended membranes, the theory leading to the determination of mechanical properties of the films, our results, and the present status of the adhesion work.

Square suspended membrane sites are fabricated using standard micromachining processes [2]. Figure 1 gives a schematic of the fabrication process. First, a p+ etch stop layer is formed by boron deposition from high temperature solid sources [3]. The deposition is at 1175 °C for 120 minutes in an environment of 90% nitrogen and 10% oxygen. A thermal oxide is grown at 990 °C for a total time of 75 minutes (15 minutes dry O_2, 45 minutes steam, 15 minutes dry O_2) giving a resulting oxide thickness of 3200 Å. Using standard photolithographic techniques, the test site pattern is defined on the back oxide while the front oxide is protected with photoresist. A 50/50 hydrazine/water solution [4] is used under reflux at 115 °C to form the silicon diaphragms, which are approximately 4.7 microns thick at this boron doping level. The polyimide (a BTDA-ODA/MPDA polymer obtained from Dupont) is spin cast on the wafer in multiple coats. After each coat, a prebake is done in air at 135 °C for 14 minutes. After the final coat, the film is cured in nitrogen at 400 °C for 45 minutes. Finally, the diaphragm supporting the film is etched away in a SF_6 plasma to form the free-standing polyimide membranes. Films ranging in thickness from 5 to 11 microns have been produced in this manner.

Theoretical analyses of the load-deflection behavior of elastic membranes have been done by many authors [5], [6], [7]. In this work, we have used the energy method described by Timoshenko [5]. Preliminary load-deflection results indicated that the intrinsic tensile stress in the film (due to shrink-
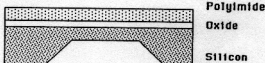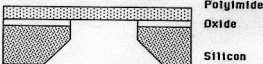

Figure 1. Process schematic

age during cure and/or thermal expansion coefficient mismatch between film and substrate) is not negligible, so the energy method was modified to include the contribution of this stress. In the energy method, which is necessarily approximate, functional forms for the displacements of the deflected surface are assumed. These functions contain several undetermined constants. The constants are found by minimizing the total system energy, leading to an expression for the deflected surface as a function of pressure and intrinsic stress.

Figure 2 shows the coordinate system for the deflected membrane. The origin is located in the plane of the oxide surface at the center of the square membrane. The functional forms we chose for the deflections are the lowest order components of the Fourier series expansion of the true solution:

$$u = c \sin(\frac{\pi x}{a}) \cos(\frac{\pi y}{2a})$$

$$v = c \sin(\frac{\pi y}{a}) \cos(\frac{\pi x}{2a})$$

$$w = w_0 \cos(\frac{\pi x}{2a}) \cos(\frac{\pi y}{2a})$$

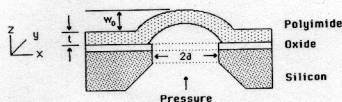Figure 2. Definitions of blister parameters

where u, v, and w are the deflections along the x, y, and z axes respectively (see Fig. 2); w_0 and c are the two arbitrary constants to be determined; and a is the halflength of the square. These assumed functional forms satisfy the zero-strain boundary conditions at the edges of the membrane and lead to nearly hemispheric deflection near the membrane center. Performing the energy minimization leads to the following expressions for w_0 and c:

$$c = \frac{51\pi^2}{315\pi^2+320}\frac{w_0^2}{a}$$

$$E\left[\frac{w_0}{t}\right]^3 + 1.6649\left[\frac{a}{t}\right]^2\frac{N_0}{t}\frac{w_0}{t} = 0.5469\frac{pa^4}{t^4}$$

where N_0 is the intrinsic stress, E is Young's modulus, p is the differential pressure and t is the film thickness. The expression for w_0, the deflection at the center of the film, is of particular interest since this is the quantity we measure. The expression predicts a linear dependence of deflection on pressure at low pressure due to intrinsic film stress, and a cubic dependence at higher pressure. What constitutes low and high pressure is determined by the magnitude of the intrinsic stress. The linear term depends only on residual stress and the cubic term depends only on elastic film constants (i.e., Young's modulus). In this model, therefore, the residual stress can be determined by the initial slope of the load-deflection curve, while Young's modulus can be determined by the curvature of this same curve. However, it must be recognized that more exact solutions for the membrane deflection may not separate so cleanly into two independent terms.

Load-deflection measurements of the suspended polyimide films are made by mounting the wafer in a specially designed chuck which seals the trapezoidal cavity in the wafer and permits the application of differential pressure by injecting volumes of air into that cavity with a microliter syringe. The cavity pressure is measured with a silicon pressure transducer which is built into the chuck. The deflection of the film at the center of the test site (x=y=0 in Fig. 2) is measured using an optical microscope. Typical data are shown in Figure 3 (circles), to which we have fit the model previously described (solid line). As can be seen, the data appear mostly linear with slight curvature. The almost linear behavior demonstrates the dominance of the intrinsic stress component of the deflection over the elastic component. Thus, we expect to be able to extract values for intrinsic film stress with greater precision than values for Young's modulus. This is indeed what is found.

Table I gives a summary of the data we have collected on various sized test sites. In this first study, each wafer contained only one test site at its center. Thus, each entry in the table represents a
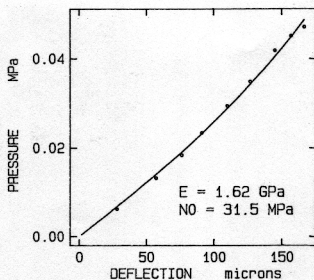

Figure 3. Model fitted to typical load-deflection data

different wafer. As can be seen, except for the 3x3 wafers, values for the intrinsic film stress agree rather well for various geometries. However, the values of Young's modulus obtained, although of the correct order of magnitude, vary substantially. We attribute this to poor resolution of the curvature measurement in such a residual stress dominated regime. This has led us to seek alternative approaches to determine Young's modulus. Note that the ratio of intrinsic stress to Young's modulus, which can be thought of as an 'intrinsic strain', is large, of the order of 1%. In general, the elastic component of the load-deflection behavior will be small until the elastic strain reaches 1%. This is dif-

SIZE (2a)	Thickness (microns)	E (GPa)	N_0 (MPa)
2	10.6	0.71	29.7
2	10.5	-	31.4
3	10.6	6.9	43.8
3	6.3	8.6	49.4
3	10.3	3.4	45.3
3	10	1.6	31.5
6	10		33.2
6	10	1.5	28.7

Table I. Intrinsic stress and modulus data

ficult to achieve in a membrane deflection. In other
work which is being reported separately [8], we have
developed an independent method which exploits this
"intrinsic strain" in micromachined structures to
determine the ratio of Young's modulus to intrinsic
stress. With that ratio and with the value for the
intrinsic stress determined by the method described
here, a value for Young's modulus of these films can
be determined with better precision than with the
present measurements.

As mentioned above, the suspended membranes are
also suited for a measurement of the adhesion of a
polymer film to a silicon wafer. By increasing the
differential pressure on the test site, the film will
peel off the substrate, forming a blister. Such a
test has been reported by Hinkley [1], using test
sites fabricated by a non-lithographic process. How-
ever, the test performed by Hinkley used a constant
pressure source to peel the film. Since the critical
pressure for peel (the pressure at which peel in-
itiates) decreases with increasing radius, the con-
stant pressure peel is inherently unstable. Once
initiated, the blister either will peel to the edge
of the wafer, or burst if the ultimate tensile stress
of the film is exceeded. Our test chuck permits the
use of controlled-volume peel. Since we are increas-
ing pressure by injection of known volumes, the vol-
ume of the blister formed is constrained and control-
lable, both at peel initiation and incrementally
thereafter. We have demonstrated that this method
can be used to follow the peeling of polyimide on
silicon. Quantitative measurements of adhesion using
this technique are now under way.

ACKNOWLEDGEMENT

REFERENCES

[1] J.A. Hinkley, "Adhesion of Polymer Films to SiO$_2$"
Journal of Adhesion, 16, 115 (1983)

[2] K. Peterson, "Silicon as a Mechanical Material"
Proceedings of the IEEE, 70, 420 (1982)

[3] Boron+ solid sources, model GS-245
Owens-Illinois, Inc.

[4] Silicon Anisotropic Etchant, PSE-100,
Transene Company, Inc.

[5] S. Timoshenko, Theory of Plates and Shells
McGraw-Hill (1940) Ch. 8,9

[6] S. Way, "Uniformly Loaded, Clamped, Rectangular
Plates With Large Deflection", Proceedings of
the Fifth International Conference for Applied
Mechanics, Cambridge, Ma. 123 (1938)

[7] J.G. Williams, Fracture Mechanics of Polymers
Holsted Press, 1984, Ch. 2

[8] M. Mehregany, R. Howe, S. Senturia, "Novel
Microstructures for the Study of Residual Stress
in Polyimide Films", Paper submitted to the
Electronic Materials Conference, Amherst, Ma.
June, 1986