MICROFABRICATED STRUCTURES FOR THE MEASUREMENT OF ADHESION AND MECHANICAL PROPERTIES OF POLYMER FILMS

Mark G. Allen and Stephen D. Senturia
Microsystems Technology Laboratories
Massachusetts Institute of Technology
Cambridge MA 02139

Introduction

Determination of the mechanical properties and adhesive strength of thin films in microelectronic devices is important both during fabrication and in evaluation of long term device reliability. Many tests of these properties are available [1-5], but few combine the advantages of an in-situ measurement technique and compatibility with standard integrated circuit processes. Several types of microfabricated structures for the measurement of the mechanical properties of polymer films have been fabricated in our laboratory [6,7]; the structures discussed in this work are suspended (free-standing) square membranes of a polymer film supported on an oxidized silicon wafer. In this paper, we report the use of these structures for the in-situ measurement of adhesion of polymer films.

Sample Fabrication

Suspended membranes are made by first fabricating a square diaphragm 5 microns in thickness in an oxidized silicon wafer using photolithography and anisotropic etching techniques [6]. The wafer is then spin-coated with the polymer of interest and the diaphragm is removed with a backside plasma etch to create a free-standing polymer membrane. Square membranes of a BTDA-ODA/MPDA polyimide (cast without adhesion promoter) from 1 to 25 millimeters (mm) on a side and ranging from 6 to 15 microns in thickness have been fabricated using this technique.

Alternate structures which are based on the suspended membranes are 'island' structures. To fabricate this structure, the square silicon diaphragm is defined so as to leave a small island of thick silicon at the center. The polymer is then spin-cast and cured as for the suspended membranes. Upon removal of the silicon diaphragm, the suspended membrane is left with a small silicon island adhered to its center. This structure is then used as the basis for further adhesion tests.

Mechanical Property Measurements

The residual stress and Young's modulus of the film can be determined by measurement of the load-deflection behavior of the membrane (see Fig. 1) [3,7,8]. The wafer is epoxied to a substrate which seals the cavity under the membrane and placed in a chuck which permits the application of differential pressure by use of either a pressure source or a microliter syringe. The differential pressure is measured using a silicon pressure transducer mounted in the chuck. The entire assembly is placed on a microscope stage with a calibrated z-axis and the deflection d of the film at the center of the membrane is measured.

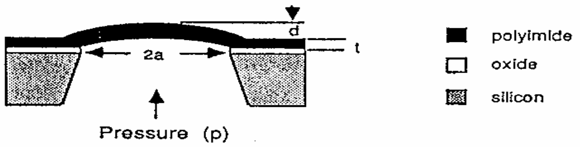

Figure 1. Membrane parameters

A theoretical analysis of the load-deflection behavior has been performed using membrane mechanics (the energy minimization approach of Timoshenko [9]), modified to account for the presence of residual stress [10]. This leads to the following relation:

$$\left(\frac{E\,t}{a^4}\right) d^3 + \left(\frac{1.66\,t\,\sigma_0}{a^2}\right) d = 0.547\, p \qquad (1)$$

where p is the applied pressure, E is Young's modulus, σ_0 is the residual stress in the film, 2a is the length of the square side, t is the film thickness, and d is the deflection at the center of the membrane. Using this approach, we have previously found values of E=3 GPa and σ_0=30 MPa for this polyimide [7]. Knowledge of both the above equation and the mechanical property data are necessary for the study of polymer adhesion using these structures.

Adhesion Measurements - Suspended Membranes

The suspended membranes can be used for a measurement of the work of adhesion γ_a of the polymer film to the silicon dioxide substrate. By increasing the differential pressure on the test site, the film will peel off the substrate, forming a blister. Computer simulations of the blister volume or radius as a function of critical pressure at constant γ_a indicate that the blister pressure-volume characteristic is unstable; once peel has been initiated at a fixed pressure, the blister will grow without bound. This phenomenon has been observed experimentally in previous applications of the blister test [11]. Our experiment uses a controlled-volume loading to initiate and limit peel. This is accomplished by injecting pressurizing fluid (air) into the space under the blister using a calibrated microliter syringe. The PV work necessary to peel (and stretch) the blister from its initial to final radius is measured. The injected fluid is then withdrawn, and the portion of the PV work that went into stretching the blister is measured. This procedure is illustrated graphically in Figure 2; the shaded area is the average γ_a times the total area peeled.

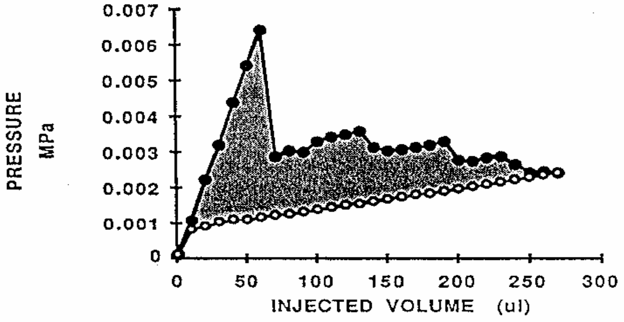

Figure 2. Adhesion PV data. Solid circles - during peel; open circles - after peel.

We have also carried out a theoretical derivation of the critical pressure necessary to initiate peeling of thin films of various geometries under lateral loading (pressure) and residual tensile stress [10]. This will be useful as a counterpart to the PV analysis described above, and is used to calculate a lower bound for γ_a should it be impossible to nucleate blisters without film failure. The approach utilizes a fracture energy balance and requires knowledge of the pre-peel load-deflection behavior of the suspended membrane. From an energy minimization analogous to the derivation of equation (1), the load-deflection behavior of films of several geometries with residual tensile stress can all be described by the equation:

$$p = k_1 d^3 + (k_2 + k_3)\, d \qquad (2)$$

where k_1, k_2, and k_3 are functions of the geometry of the test site and the type of film (plate or membrane). For thin films on infinitely rigid substrates, the peel criterion is:

$$\gamma_a = \left[\frac{5}{2}\frac{\Delta^4 c_1}{a^{11}} + \frac{3\Delta^2 c_2}{a^7} + \frac{2\Delta^2 c_3}{a^5}\right]\left[\, da/dA \,\right] \qquad (3)$$

where Δ is the generalized load-point displacement of the blister (in this case, blister volume), da/dA is the incremental dependence of blister size on blister area, and c_1, c_2, and c_3 are mechanical-property-dependent constants. Table I gives the values of the various parameters in equations (2) and (3) for three blister geometries: the clamped circular plate, the circular membrane, and the square membrane. By substitution of the appropriate constants into equation (3), and simultaneous solution of (3) with the corresponding load-deflection relation (2), a value for γ_a can be determined as a function of the critical debond pressure p_c. Thus, experimental measurement of the critical debond pressure can be related to γ_a once the mechanical properties of the film have been accurately determined.

Under certain conditions, equation (3) reduces to special cases which have been previously reported in the literature. For example, in the case of a clamped circular plate with zero residual stress undergoing small deflections, ($c_1 = c_3 = 0$) the peel criterion from equation (3) is:

$$\gamma_a = 0.5\, p_c d_c \qquad (4)$$

Table I. Geometric constants for adhesion model

	SQUARE MEMBRANE	CLAMPED CIRCULAR PLATE	CIRCULAR MEMBRANE
k_1	$1.83\,E\,t / a^4$	$2.77\,E\,t / a^4$	$3.56\,E\,t / a^4$
k_2	0	$5.68\,E\,t^3 / a^4$	0
k_3	$3.04\,t\,\sigma_o / a^2$	$4\,t\,\sigma_o / a^2$	$4\,t\,\sigma_o / a^2$
c_1	$0.429\,E\,t$	$2.42\,E\,t$	$0.917\,E\,t$
c_2	0	$5.44\,E\,t^3$	0
c_3	$1.88\,t\,\sigma_o$	$3.82\,t\,\sigma_o$	$2.55\,t\,\sigma_o$
Δ	$16a^2 d / \pi^2$	$a^2 d \pi /3$	$a^2 d \pi /2$
da/dA	$1/2a$	$1/2\pi a$	$1/2\pi a$

where d_c is the critical center deflection at which debond initiates; this relation has been obtained by Williams [11]. Alternatively, for the case of a circular membrane undergoing large deflections with zero residual stress, ($c_2 = c_3 = 0$) the peel criterion from equation (3) is:

$$\gamma_a = 0.625\, p_c d_c \tag{5}$$

Gent [12] has also analyzed this case assuming a slightly different load-deflection profile and has obtained a value of 0.65 for the premultiplying factor in equation (5).

For square test sites under residual stress such as the suspended membranes, the relation between γ_a and the critical center deflection d_c at which debond initiates is given by:

$$\gamma_a = 3.70Et\,(d_c/a)^4 + 4.94\sigma_o t\,(d_c/a)^2 \tag{6}$$

The relation between γ_a and p_c can be obtained by simultaneous solution of equations (1) and (6), allowing determination of γ_a from a measurement of either p_c or d_c during peel.

The upper limit of γ_a which can be measured using this technique is limited by the tensile strength of the film (this effect is common in many standard adhesion tests; for example, the 90° peel test is also tensile-strength limited). It was determined that membranes fabricated by the standard process and cure schedule could not be peeled; films always ruptured before blisters were formed. For these samples, a lower bound for γ_a was calculated by using the pressure at which the film ruptured as the p_c value for the square membrane.

In order to observe blister nucleation, it was necessary to degrade the adhesion of the PI/SiO_2 interface. This was done by immersing the test sites in 90°C H_2O for varying lengths of time. Table II gives a summary of the adhesion data obtained from the suspended membranes. Sample 1 was not subjected to any degredative processing and burst before blister nucleation; a lower bound for γ_a was calculated from equation (6) to be 360 J/m^2. As expected, adhesive strength generally decreased with increased immersion time (samples 2-6) although this effect was not investigated quantitatively. Upon drying, an increase in adhesive strength was observed (samples 2,3). Agreement between the PV method and equation (6) was observed to be within 50% except for sample 4, which showed significant plastic deformation, invalidating the PV analysis. Equation (6) uses only one data point to calculate γ_a, while the PV method is averaged over the entire wafer. This may account for differences in the two approaches. Furthermore, equation (6) implicitly assumes an incrementally symmetric peel, which is not strictly correct for the square membrane. However, as peel of the square membrane continues, a circular blister is formed, allowing application of the appropriate form of equation (3). These details are presently under study.

Adhesion Measurements - Island Structures

The suspended membrane blister test, like other peel tests, is limited by the tensile strength of the film. One way to overcome this problem is to use thicker films [13]. In the blister test, we have additional flexibility. Different geometries for the microfabricated site are possible which can facilitate peel of thinner films even in systems with very good adhesion.

Equation (3) suggests that if a geometry can be found in which da/dA can be increased, larger values of γ_a may be measured at the same load. For simple blisters, this derivative is inversely proportional to the membrane size (Table I). Decreasing the membrane size fails since the deflection

Table II. Adhesion data

#	Sample	Environmental Conditions	Analysis method	γ_a (J/m^2)
1	6x6 blister site	none	Equation (6)	>360
2	3x3 blister site	18 hr. H_2O 90°C	PV	1.5×10^{-4}
		18 hr. H_2O 90°C + 1 hr. dry 60°C	Equation (6)	4.4×10^{-3}
			PV	3.5×10^{-3}
3	6x6 blister site	20 hr. H_2O 90°C + 6 hr. dry 25°C	PV	2.0
			Equation (6)	3.1
4	10x10 blister site	8 hr. H_2O 90°C	PV	1200 (*)
			Equation (6)	320
5	10x10 blister site	14.5 hr. H_2O 90°C	Equation (6)	64
6	10x10 blister site	14.5 hr. H_2O 90°C	Equation (6)	43

(*) Extensive plastic deformation observed

Δ will also decrease. This problem is overcome in the island structure shown in Figure 3, where the polymer film will be peeled only off the center island. The deflection Δ is a function of the difference $a_2 - a_1$, where $2a_2$ is the characteristic size (edge length or diameter) of the entire suspended membrane, while $2a_1$ is the characteristic size of the island. However, the derivative da/dA is proportional only to $1/a_1$. Thus, a large geometric advantage can be obtained by decreasing a_1 while keeping $a_2 - a_1$ large.

Although the critical pressure analysis for the island structures is considerably more complicated than for the simple blisters, an approximate relation between p_c and γ_a based on a circular geometry can be developed. For a membrane whose load-deflection behavior is dominated by residual tensile stress and which is suspended over a circular annulus of inner radius a_1 and outer radius a_2, γ_a is related to p_c by:

$$\gamma_a = \frac{p_c^2 a_1^2}{32 \sigma_0 t}\left[\frac{\beta^2 - 1}{\ln \beta} - 2\right]^2 \qquad (7)$$

where β is defined as the annular ratio a_2 / a_1. Although approximate, it is instructive to examine the limiting behavior of equation (7). As β approaches unity, γ_a approaches zero (since no film is exposed, no adhesion can be measured even at inifinite pressure), while as β approaches infinity, γ_a gets large for any pressure p_c. Thus, it is theoretically possible to measure large γ_a values at pressures less than the ultimate tensile stress of the film by making the center island sufficiently small.

Concentric square island structures have been fabricated with an outer size ($2a_2$) of 10 mm and inner size ($2a_1$) of 1 and 2 mm. Smaller a_1 values can be obtained by underetching the film on a 1 mm island until only 0.25 mm or even 0.125 mm sections of the film remain adhered to the center island (Figure 3b). Peel has been achieved using these underetched structures. Although finite-element analysis of the square island structure will be required to generate accurate values of γ_a from observed debond pressures, order of magnitude values of γ_a can be obtained from equation (7). Preliminary experiments indicate that such values are in the range of 1000-3000 J/m^2, in fair agreement with values obtained from application of the peel test to thicker films [13].

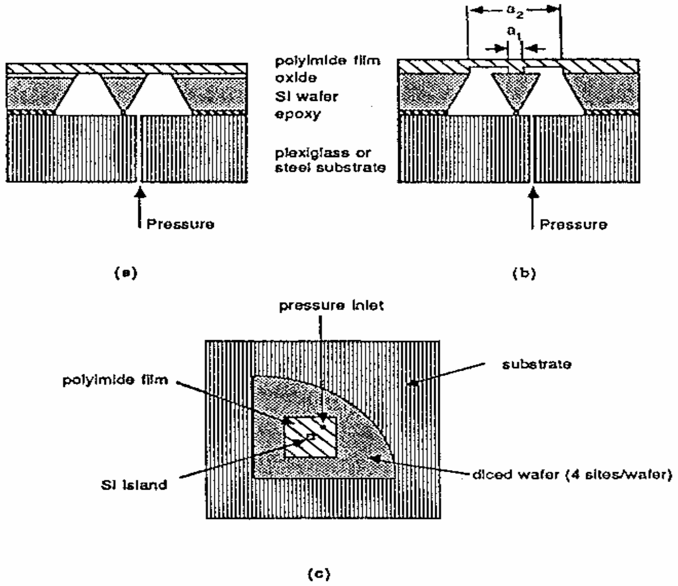

Figure 3. Island adhesion structures.
(a) side view; (b) side view - underetched film; (c) top view

Conclusions

Microfabricated test structures for the in-situ measurement of adhesion of thin films have been described. Young's modulus and residual tensile stress are determined from pre-peel measurement of the load-deflection behavior of suspended membranes. On systems of weak adhesion, a blister test using suspended membranes has been carried out. For systems of good adhesion, an island test structure has been developed allowing even thin films to be tested. Mechanical models for all three structures are described.

Acknowledgement

This work was supported in part by E.I. DuPont de Nemours & Co. and the Office of Naval Research. Microfabrication was carried out in the Microsystems Technology Laboratories, and in the Microelectronics Laboratory of the MIT Center for Materials Science and Engineering, which is supported in part by the National Science Foundation under Contract DMR-84-18718. The development of the fabrication process for suspended membranes was carried out by Mehran Mehregany. We also wish to acknowledge David Parks for discussions on fracture energetics and Alan Gent for providing a preprint of his work.

References

1. R.W. Hoffman, in Physics of Nonmetallic Thin Films, ed. Dupey and Cachard, NATO Advanced Study Institutes Series B, vol. 14 Plenum Press, New York, 1976
2. L.B. Rothman, J. Electrochem. Soc., **127**, 2216 (1981)
3. J.W. Beams, in Structure and Properties of Thin Films, ed. C.A. Neugebauer, J.B. Newkirk, and D.A. Vermilyea, Wiley, 1959
4. R.J. Jensen, J.P. Cummings and H. Vora, IEEE Transactions on Components, Hybrids, and Manufacturing Technology, **7**, 384-393 (1984)
5. P. Geldermans, C. Goldsmith and F. Bendetti, in Polyimides, Synthesis, Characterization, and Applications, ed. K.L. Mittal, Plenum Press, vol. 2, 695-711 (1984)
6. M. Mehregany, S.M. Thesis, Department of Electrical Engineering and Computer Science, Massachusetts Institute of Technology, May 1986
7. M. G. Allen, M. Mehregany, R. T. Howe, and S. D. Senturia, submitted to Appl. Phys. Lett.
8. M. Mehregany, M.G. Allen, and S.D. Senturia, Proceedings of the 1986 Solid-State Sensors Workshop, Hilton Head, S.C., June 1986
9. S. Timoshenko, Theory of Plates and Shells, Chapter 9, McGraw-Hill,1940
10. M.G. Allen, S.M. Thesis, Department of Chemical Engineering, Massachusetts Institute of Technology, May, 1986
11. M. L. Williams, J. Appl. Poly. Sci., **13**, 29 (1969)
12. A. N. Gent and L. Lewandowski, J. Appl. Poly. Sci., in press
13. D. Suryanarayana, K.L. Mittal, J. Appl. Poly. Sci., **29**, 2039 (1984)