CALIBRATED MEASUREMENTS OF ELASTIC LIMIT, MODULUS, AND THE RESIDUAL STRESS OF THIN FILMS USING MICROMACHINED SUSPENDED STRUCTURES

Fariborz Maseeh, Martin A. Schmidt, Mark G. Allen, and Stephen D. Senturia
MIT Microsystems Technology Laboratories, Cambridge, MA

Abstract

Quantitative determination of material properties such as the Young's modulus (E), Poisson's ratio (ν), residual stress (σ_0), and elastic limit of polymer films used in electronics and microsensors devices is essential. This paper reports on the calibration of the suspended square membrane method using both finite element methods (FEM) and comparison with conventional tensile tests. We also report on the use of suspended circular membranes to measure the elastic limit of thin films.

Introduction

Most microelectronic devices and sensors are fabricated using thin film deposition or diffusion. The films are often under residual stresses developed in the fabrication process. A number of different techniques have been employed for determining mechanical properties of thin films. Techniques such as beam buckling are used for measuring thin films under compressive stress [1], while released structures[2], and membrane deflection under applied pressure [3,4] have been employed to measure the properties of thin films under tensile residual stress. Wafer curvature methods can be used to measure average stress of both tensile and compressive thin films [6].

In this work we have used the membrane technique to measure the mechanical properties of polyimide thin films under residual tensile stress. A polyimide square membrane is microfabricated by spin depositing polyimide on a p^+ doped square silicon diaphragm. The p^+ diaphragm is fabricated by patterning the backside of the wafer and anisotropically etching the undoped silicon and stoping on the p^+ diffused layer. This layer is removed as the last step by dry etching, releasing a square polyimide membrane supported by the silicon wafer. The state of stress is maintained by the silicon support.

The residual stress and Young's modulus of thin films under tensile stress can be determined by fitting the pressure vs deflection data of the square membrane with a suitable model. The simplest approach is an approximate analytical model based on membrane mechanics. The center deflection d under applied pressure p of a square membrane of size 2a and thickness t with in-plane residual stress σ_0 can be shown from membrane mechanics to be of the form [4]:

$$p = c_1\,(t\,\sigma_0 / a^2)\,d + c_2\,(E\,t / a^4)\,d^3 \qquad (1)$$

where E is the Young's modulus, c_1 is a constant (3.0 in the membrane model) and c_2 depends weakly on Poisson's ratio (assumed to be 0.25 throughout the work, for which c_2 is 1.8 in the membrane model).

Equation (1) is used to extract σ_0 and E by fitting to the experimental pressure vs deflection results. In the original work, the constants c_1 and c_2 were evaluated by an energy minimization technique using an approximate functional form for the deflected shape of the membrane. This paper reports several approaches to confirm the results obtained from equation (1). Finite element methods (FEM) were used to fit the p vs d data independently, from which we both confirmed the functional form of Eqn.(1) and calibrated the constants c_1 and c_2. Young's modulus was independently measured by direct tensile testing of microfabricated thin polyimide strips from which the modulus values were compared against Eqn.(1) and FEM results. Circular membranes, for which superior analytical models exist, were microfabricated and from the p vs d of such membranes, σ_0 and E were evaluated. Finally, from the circular membranes we could detect the elastic limit properties such as the yield stress and strain.

Calibration of Membrane Equation

The finite element methods were utilized to fit the pressure vs center deflection data from the square polyimide membranes of different sizes. Fig.1 shows the FEM iterative procedure used to evaluate the stress and modulus. Three different membranes were chosen for which the p vs d data was available. Each membrane was modeled using nonlinear FEM. The input required specification of the geometry, boundary conditions, material properties, and applied loads. Each membrane was discretized with 64 4-noded shell elements. All boundaries were fixed. The residual stress was introduced artificially into the FEM model by using a thermoelastic material law and inducing a negative differential temperature. Nonlinear analysis based on large displacements and small strains were used in the solution. Convergence was achieved using full Newton iteration with line search in each run.

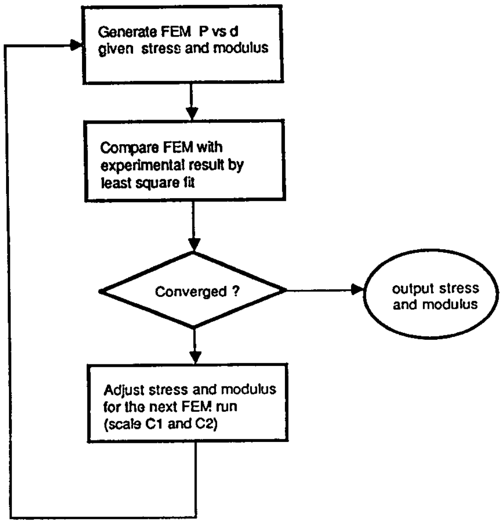

Fig.1

Iterative finite element approach to obtain residual stress and modulus

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The FEM output gives the deflection contour over the entire membrane as well as stresses over each element. The stresses were used for detecting the correctness in modeling. The center deflection vs the applied pressure from each run was of interest for comparison with the experimental data.

To investigate the integrity of Eqn.(1) as a tool to measure the residual stress and Young's modulus, we examined this equation both functionally and quantitatively. The results showed that the functional dependence of Eqn.(1) on geometry, on σ_0, and on E was correct. Furthermore, as shown by the results in Table 1, the FEM results agreed with Eq.1 to within 6% in residual stress and 10% in modulus, confirming the values of c_1 and c_2 to those levels of accuracy.

Side (mm)	Thick. (μ)	FEM Stress	Eq. (1) Stress	FEM/Eq (1) Stress ratio	FEM Modulus	Eq. (1) Modulus	FEM/Eq(1) Mod. ratio
4	10	28.25	30.0	1.067	2430	2600	1.070
8	8.5	37.60	39.8	1.058	2900	3200	1.100
10	7.0	28.71	30.0	1.045	2900	3200	1.100

Table 1

FEM vs Eq.(1) results for residual stress (MPa), and Young's modulus(MPa) using data from three square membranes of BTDA-MPDA/ODA. The two ratio columns indicate the agreement between c_1 and c_2 for the two models.

Direct Tensile Test

The goal of this measurement was to independently and directly measure the Young's modulus and to study the stress vs strain behavior of different polyimide chemistries. Two different polyimide types, namely, BTDA-MPDA/ODA and PMDA-ODA, were used in sample fabrication. Thin polyimide strips of 24 mm x 12 mm with different thicknesses were micromachined. The fabrication process follows the released structure fabrication procedure of Ref.[1] except that the diaphragms were anisotropically etched using KOH instead of hydrazine. Fig.2 shows the Instron specimen fabrication sequence. Polyimide was spin deposited on a 1 inch x 1 inch p^+ doped diaphragm, aluminum was evaporated on polyimide and patterned to the specimen shape as shown in Figs.2a,b. This forms an etch mask for the polyimide which is removed by dry etching. The aluminum layer is then removed by wet etching, and finally, the back side membrane is removed, releasing the specimen as shown in Fig.2d. Before loading this specimen in the Instron, two sections of the wafer are removed along four pre-etched scribe lines (in the back), as shown in Fig.2e, releasing the stress. The remaining silicon acts as supports for the grips of the Instron. Care was required during sample fabrication to avoid any edge cracks during handling. An Instron Model 1123 was utilized for the test with a crosshead speed of 0.5 mm/min.

Fig.3 shows stress vs strain curve for high viscosity BTDA-MPDA/ODA polyimide with thickness 14 μm. The specimen was loaded to 11 N (65 MPa) and unloaded. The unloading curve followed the loading curve closely except for a small offset which is believed to be due to backlash of reverse gears of the machine. The sample was reloaded and the loading curve followed the first one closely, as shown, up to 11 N, and then the sample was unloaded, and the unloading curve followed the loading curve again with the same offset. The sample was then loaded to the break point. The slope of the final loading curve was the same as the slope of the previous curves for this specimen. One would conclude that BTDA-MPDA/ODA has linear elastic brittle type behavior and the use of one E value in the analysis is sufficient. However, we will show below that, in fact, this type polymer does exhibit yield behavior prior to fracture. The Young's modulus and the strain at break for this specimen were measured to be 3040 MPa and 2.8% respectively. The E value agrees well with the membrane results for standard type BTDA-MPDA/ODA.

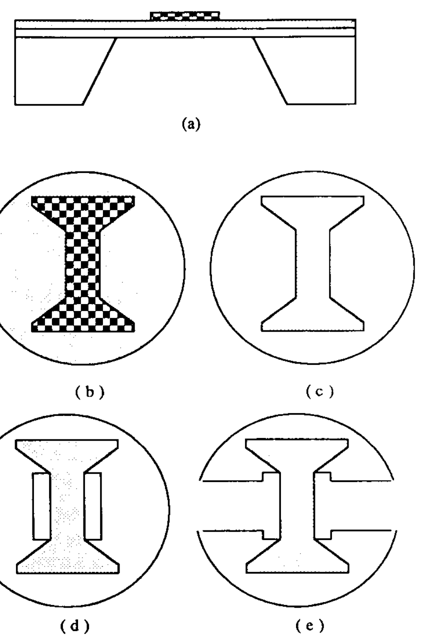
(a)
(b)
(c)
(d)
(e)

Fig.2

Fabrication of Instron specimen; a) p^+ diaphragm with polyimide and patterned aluminum; b) top view of patterned aluminum etch mask; c) polyimide specimen after dry etch and aluminum removal; d) backside silicon diaphragm removed, dashed lines are the etched scribe lines in backside; e) sections of silicon removed along scribe lines, yielding specimen ready for the Instron test

Fig.4 is the Instron test results for standard PMDA-ODA polyimide type sample 6.5 μm in thickness. The sample was loaded to 7 N and unloaded (solid line). The unloading curve was significantly different from the loading curve. Plastic deformation is evident as the unloading curve crossed the strain axis at an offset. The sample was reloaded and unloaded (dotted line), and again, an offset was observed on the strain axis. The loading and unloading was repeated one more time (not shown, but identical to dotted line), and finally the sample was loaded to the break point (dashed line) which occured at 8.7 N (112 MPa). The Young's modulus and strain at break were measured to be 2700 MPa and 15.6% for this sample.

The direct tensile test is a useful measurement technique for understanding the behavior of thin films. The information obtained from this test will help analytical modeling of structures involving polymer thin films in general. This is evident from the comparison of the two polyimide types where in one (BTDA-MPDA/ODA) a single value for E is sufficient in the analytical model since the material shows linear elastic behavior up to the break point, while in the other

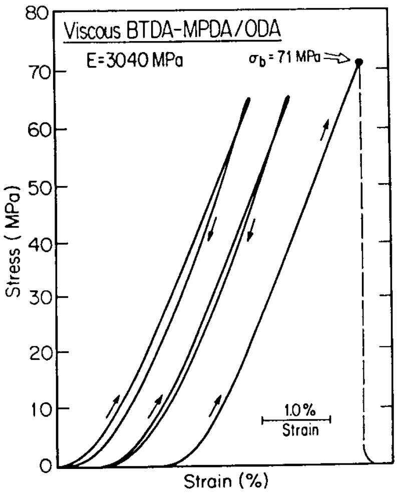

Fig.3

Stress vs strain results for BTDA-MPDA/ODA; two load cycles and final loading to break. Successive load cycles are displaced horizantally for clarity. The strain at break was 2.8%.

(PMDA-ODA) the stress-strain relation is nonlinear in which a single E cannot be assumed in the analysis after the proportional limit point.

On the other hand, because the Instron specimens are under uniform uniaxial stress, the yield and ultimate strength properties measured by this method can be dominated by edge cracks and other processing imperfections. In the following section, we present an alternative approach which is more effective for studying the yield properties of thin films.

Elastic Limit Properties

The elastic limit of thin polyimide films was measured by investigating plastic deformation of circular membranes under applied pressure. Circular membranes lack stress concentration points present in the square membranes. Furthermore, the fabrication procedure is free from sharp edges eliminating the presence of microscopic edge defects which are difficult to avoid in the Instron specimen fabrication. It was then possible to directly measure the yield point using the circular membranes in a controled experiment.

One inch diameter membranes of standard BTDA-MPDA/ODA type polyimide were fabricated and loaded to their elastic limit. The fabrication process consists of spin depositing polyimide on a silicon wafer, adhering a prefabricated circular ring to the polyimide side, and wet etching the silicon. The ring supports the released membrane, maintaining the state of stress in the polyimide film. The circular membrane fabricated as above is then mounted to another substrate for our standard pressure vs deflection measurements. From the p vs d results measured before the yield point, we obtained the stress and modulus to be 28 MPa and 3300 MPa respectively. These values were consistent with the results obtained from the previous methods. The load was then increased until plastic deformation was initiated and then the load was kept constant. The plastic deformation started at the center of the loaded sample, where the stresses are the highest and gradually moved outwards. Using the pressure at yield and the measured deformation, the yield stress and strain were determined.

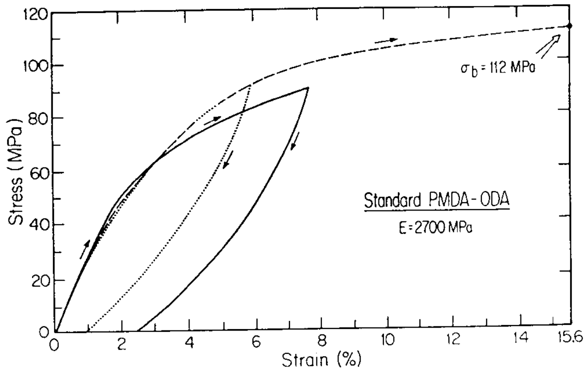

Fig.4

Stress vs strain results for PMDA-ODA; three load cycles and final loading to break :
solid line = first load/unload;
dotted line = second and third load/unload;
dashed line = loading to break

Fig.5 shows the measured p vs d results for a 1 inch circular membrane. The yield stress and strain was measured to be 130 MPa and 3% respectively. The measured yield strain of the circular membrane is higher than the break strain of the same chemistry polyimide from the Instron measurement (2.8%), implying the circular membrane technique's superiority (due to its symmetry) in studying the material behavior in the post-elastic range.

Fig. 6 shows the plastically deformed sample after the applied pressure was removed. The domed shape region is the plastically deformed region which initiated from the center of the membrane and propagated radially outward. The flat region is the unyielded region, which is still under residual stress.

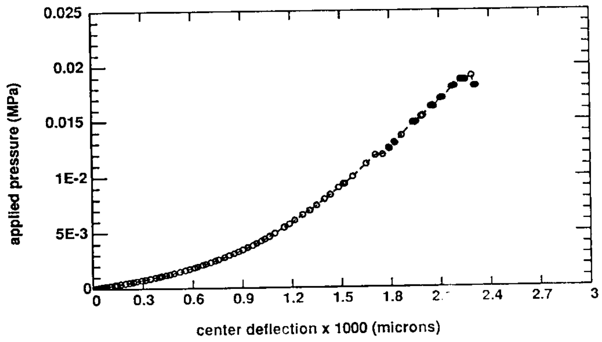

Fig.5

Pressure vs deflection results for a BTDA-MPDA/ODA circular membrane

Conclusion

A number of consistent measurement techniques have been employed to determine the mechanical propeties of thin polyimide films. The techniques can be adapted to other thin films from which microfabricated specimens can be made. The Instron test results provided useful information about the material behavior including Young's modulus whereas the membranes were used to determine quantitative values for residual stress and elastic modulus. Finally the axisymmetric state of circular membranes under pressure were utilized effectively in determining the elastic limit of thin polymer films.

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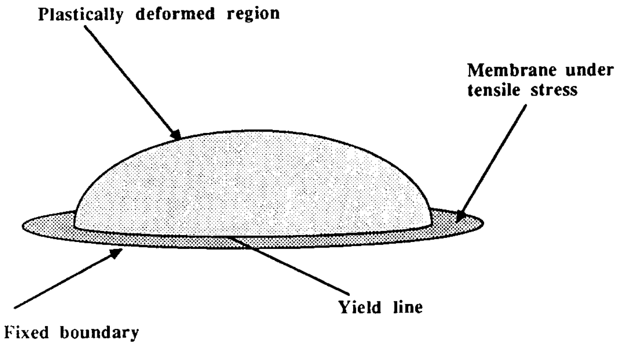

Fig.6

BTDA-MPDA/ODA circular membrane under plastic deformation; domed region is under permanent deformation, and the flat region under residual stress