Microfabricated structures for the *in situ* measurement of residual stress, Young's modulus, and ultimate strain of thin films

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Two microfabricated structures for the *in situ* measurement of mechanical properties of thin films, a suspended membrane, and an asymmetric "released structure," are reported. For a polyimide film on silicon dioxide, the membrane measurements yield a residual tensile stress of 30 MPa and a Young's modulus of 3 GPa. The released structures measure the ratio of residual stress to Young's modulus, and yield 0.011 at strains comparable to the suspended membranes, and 0.015 at larger strains. The ultimate strain as measured by both structures is approximately 4%.

The importance of accurate *in situ* determination of the mechanical properties and residual stress of thin films and coatings is well established.[1-6] The conventional method of residual stress measurement involves depositing the film of interest on a substrate (for example, a silicon wafer) and measuring the resulting substrate curvature.[5] Other *in situ* methods have been developed[2,7-9] which rely on the buckling of microfabricated structures, and are most readily applicable to the measurement of compressive stresses. This letter addresses the case of films under residual tensile stress.

We report here two types of microfabricated test structures and illustrate their use for the measurement of residual tensile stress, Young's modulus, and ultimate strain of a polymeric thin film. The first structure is a suspended thin-film membrane. The second, called a "released structure," is an asymmetric structure patterned into the thin-film membrane prior to removal of the support.

Square suspended membrane sites are fabricated using micromachining techniques.[10,11] A 5-μm-thick square diaphragm is etched in a (100) silicon wafer from the back using a silicon dioxide etch mask, a 5-μm p^+ diffusion of boron as the etch stop, and 50% hydrazine in water at 110 °C as the anisotropic etchant. A thin film of the polymer of interest (in our case, the polyamic acid precursor for a benzophenonetetracarboxylicdianhydride-oxydianiline/ metaphenylenediamine polyimide dissolved in *N*-methyl pyrrolidone) is spin cast on the wafer. Multiple coats, with a 14-min bake at 135°C between coats, are used to achieve the desired final thickness and to avoid single-layer pinholes. The polyimide is cured in nitrogen at 400 °C, and the silicon diaphragm is removed using a backside SF_6 plasma etch to form the free-standing polymer membrane. Membranes of size ranging from 2 to 10 mm on a side and film thickness ranging from 6 to 10 μm have been fabricated. A similar process has been reported by Bokov.[12]

The residual stress and Young's modulus of the film are determined from the load-deflection behavior of the membrane (see Fig. 1).[4,6,13] The wafer is mounted in a chuck which seals the cavity under the membrane and permits the application of differential pressure by use of a microliter syringe. A silicon pressure transducer mounted in the chuck measures the applied pressure. The entire assembly is placed on a microscope stage with a calibrated *z* axis and the deflection of the film at the center of the membrane is measured. Figure 2 shows load-deflection data obtained from suspended membranes of various sizes and thicknesses. The solid lines through the points represent the fit of the model to the data (see below). The total strain of these diaphragms (residual plus deformation) never exceeds 2%.

An analysis of the load-deflection behavior of the membrane using an energy minimization approach,[14] but modified to account for the presence of residual tensile stress,[13,15] leads to the following relation:

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

where p is the applied pressure, E is Young's modulus, σ_0 is the residual stress, $2a$ is the site size, t is the film thickness, and d is the membrane deflection. The residual stress appears only in the linear term, and Young's modulus only in the cubic term. Hence, independent determination of both quantities is readily accomplished. The model can be verified by varying the membrane geometry. The solid curves of Fig.

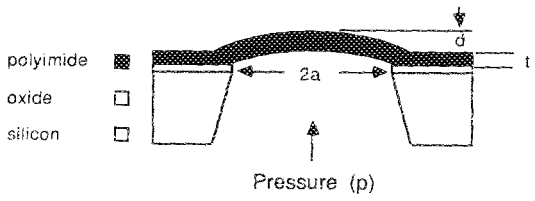

FIG. 1. Definition of membrane parameters.

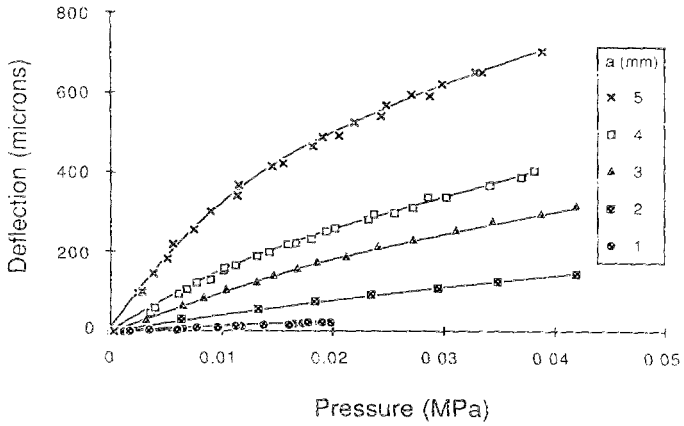

FIG. 2. Load-deflection data from square polyimide membranes of various sizes (in mm).

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TABLE I. Results from suspended membranes.

Batch	No. of samples	t (μm)	a (mm)	E (GPa)	σ_0 (MPa)	σ_0/E
1	2	10.5–10.6	2	...	30.6 ± 0.9	...
2	2	6.3–10.6	6	...	31.0 ± 2.3	...
3	3	8.3–8.5	8–10	3.0 ± 0.2	36.1 ± 3.7	0.012
4	3	7.0–7.5	8–10	2.9 ± 0.7	30.3 ± 0.2	0.010

2 were obtained using Eq. (1), and the extracted values of σ_0 and E are shown in Table I. Extraction of a value for E could be done only for the larger diaphragms where the cubic term is significant. Within each processing batch, there is good agreement between the σ_0 and E values. The batch-to-batch variation is not explained at present. The results are in reasonable agreement with published values for this polyimide[5,16] using wafer curvature measurements.

The "released structure" concept relies on the presence of residual tensile stress in the film. Fabrication is identical to the suspended membrane, except that an asymmetric structure is photolithographically patterned into the membrane prior to plasma-etch removal of the supporting silicon diaphragm. Removal of the diaphragm then "releases" the structure, which deforms due to the residual stress. Measurement of this deformation in conjunction with appropriate mechanical models yields the ratio of residual stress to Young's modulus.

The polyimide released structures are patterned in an oxygen plasma using an aluminum etch mask,[11] which is then removed in a phosphoric-acetic-nitric acid etch prior to plasma-etch removal of the silicon diaphragm. Finally, the deflection of the released structure relative to fiducial marks denoting the original unreleased position is measured using an optical microscope with a calibrated x-y stage.

We have investigated a wide variety of released structures having both T and H shapes (see Fig. 3).[11] For the T structures, the transverse beam is loaded in a bending mode by the residual tensile stress in the stem of the T, which can result in large deflections at small strains. For the H structures, residual tensile stress causes an axial shrinkage of the wide part of the beam and a corresponding axial extension of the narrower necks upon release. Figures 4 and 5 show SEM photos of the T and H structures, respectively. In Fig. 4, the cross beam of the T structure has deflected by 26 μm. Figure 5 shows a pair of H structures, one of which has deflected on release, the other of which has resulted in failure on release

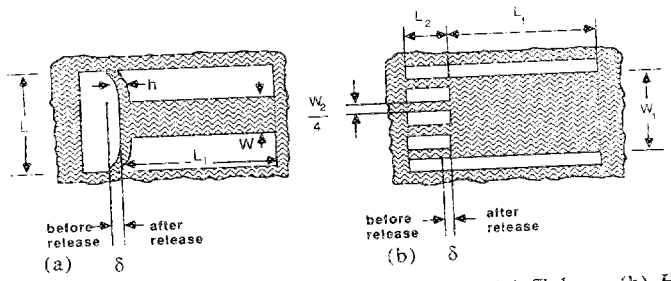

FIG. 3. Released structures—schematic plan view. (a) T shape; (b) H shape.

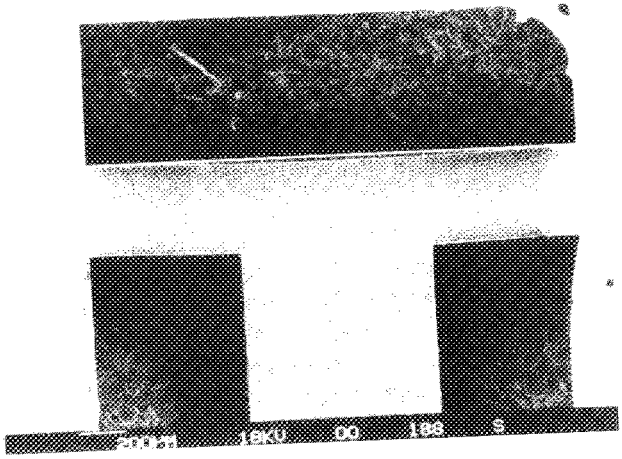

FIG. 4. T released structure (SEM photo).

by exceeding the ultimate strain in the thin necks. The unfailed H structure shown has deflected by 12 μm.

For the case where the ultimate strain is not exceeded, the in-plane stress-strain behavior of the film can be modeled using elementary beam-bending theory for the T and axial-beam theory for the H.[17] For the geometries shown in Fig. 3, the relations are

$$T \text{ structure: } \frac{\sigma_0}{E} = \delta\left(\frac{1}{L_1} + \frac{16}{W(L^3/h^3 - W^2L/h^3 + W^3/2h^3)}\right), \quad (2)$$

$$H \text{ structure: } \frac{\sigma_0}{E} = \delta\left(\frac{W_1/L_1 + W_2/L_2}{W_1 - W_2}\right), \quad (3)$$

where δ is the measured deflection of the structure once it has been released. Based on similar analyses, the total strain of the structures shown in Figs. 4 and 5 can be estimated as 1.1% for the T and 3.4% for the H.

σ_0/E data have been obtained from a variety of released structures. For the T structures, an average value of 0.011 ± 0.001 was obtained, in good agreement with the results from the membranes (which are also low-strain structures). Some H structure results are shown in Table II. The

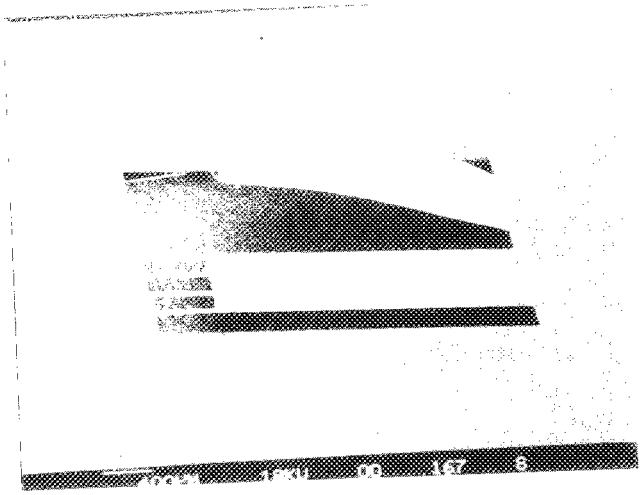

FIG. 5. H released structure (SEM photo).

TABLE II. Results from released structures (film thickness 5.5 μm).

Structure	L_1 (μm)	L_2 (μm)	W_1 (μm)	W_2 (μm)	σ_0/E
1	1535	505	690	2×45	0.0136
2	1535	505	690	4×20	0.0140
3	2510	505	690	2×95	0.0151
4	2510	505	690	4×45	0.0149

average value of σ_0/E is 0.015 ± 0.001, consistently higher than the other structures. Some of the discrepancy may be due to imprecision in the simple beam theory analysis. Another possibility is that the *H* structures are strained beyond their proportional limit.

In Fig. 5, the *H* structure with the thinner necks has fractured. Since the geometry of the structure before fracture is known, the ultimate strain at break can be determined (4.5% for the structure shown). In thicker films (10 μm), ultimate strains as high as 8% have been observed. It is possible that the thicker sample begins to crack during the final stages of etch removal of the support, providing some strain relief which, in turn, affects the observed ultimate strain value.

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