

A new bulge test technique for the determination of Young's modulus and Poisson's ratio of thin films

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A new analysis of the deflection of square and rectangular membranes of varying aspect ratio under the influence of a uniform pressure is presented. The influence of residual stresses on the deflection of membranes is examined. Expressions have been developed that allow one to measure residual stresses and Young's moduli. By testing both square and rectangular membranes of the same film, it is possible to determine Poisson's ratio of the film. Using standard micromachining techniques, free-standing films of LPCVD silicon nitride were fabricated and tested as a model system. The deflection of the silicon nitride films as a function of film aspect ratio is very well predicted by the new analysis. Young's modulus of the silicon nitride films is 222 ± 3 GPa and Poisson's ratio is 0.28 ± 0.05 . The residual stress varies between 120 and 150 MPa. Young's modulus and hardness of the films were also measured by means of nanoindentation, yielding values of 216 ± 10 GPa and 21.0 ± 0.9 GPa, respectively.

I. INTRODUCTION

The mechanical properties of thin films and the residual stresses in them have long been recognized to be important in the fabrication of electronic devices and microsensors.¹ This has provided a motivation for the study of mechanical properties of thin films. Unfortunately, the techniques commonly used to measure these properties in bulk materials are not directly applicable to thin films. Thus, specialized mechanical testing methods have been sought.

The bulge test was one of the first techniques introduced for the study of thin film mechanical properties.² In its original form, a circular film or membrane is clamped over an orifice and a uniform pressure is applied to one side of the film. The deflection of the film is then measured as a function of pressure allowing a determination of the stress-strain curve and the residual stress of the film. The stress state in the film is biaxial so that only properties in the plane of the film are measured. Traditionally the test has been plagued by a number of problems. The results are rather sensitive to small variations of the dimensions of the film and may be affected by twisting of the sample when it is mounted. Sample preparation is therefore crucial and special steps need to be taken to minimize these effects. The residual stresses in the film also have to be tensile. Finite element studies^{3,4} have shown that for films in compression, the circumferential stress near the edge of the film remains compressive even at high applied pressures, causing the film to buckle. Wrinkles in such films disappear only gradually as the pressure on the film is increased, leading to erroneous results. Finally, failure to take

into account the initial height of the membrane in the analysis leads to apparent nonlinear elastic behavior of the film.⁵

Developments in micromachining techniques and better analysis methods have made it possible to overcome many of the problems associated with the bulge test. In this paper, a new analysis of the deflection of rectangular membranes is presented and it is demonstrated how Young's modulus, Poisson's ratio, and residual stress can be accurately measured by testing both square and rectangular films with large aspect ratios. We also describe a technique to fabricate free-standing films of silicon nitride on silicon substrates, using standard lithography and anisotropic etching techniques. The dimensions of the films can be controlled precisely if the membranes are made rectangular in shape and oriented with the crystal axes of the Si substrate. Silicon nitride is used as a model system, but the technique can be extended to a large number of films with only minor modifications. The results obtained from the bulge test are compared to results from nanoindentation experiments performed on the same material.

II. ANALYSIS OF THE DEFLECTION OF A MEMBRANE

A. Square films

Calculation of the deflection of a membrane under a uniform pressure is a difficult problem. For the large deflections that are typical in bulge tests, the membrane behaves nonlinearly. Let u , v , and w be the components of the displacement parallel to the x , y , and z directions (see

Fig. 1). The strains in the membrane are then given by⁶:

$$\begin{aligned} \epsilon_x &= \frac{\partial u}{\partial x} + \frac{1}{2} \left(\frac{\partial w}{\partial x} \right)^2 \\ \epsilon_y &= \frac{\partial v}{\partial y} + \frac{1}{2} \left(\frac{\partial w}{\partial y} \right)^2 \\ \gamma_{xy} &= \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} + \frac{\partial w}{\partial x} \frac{\partial w}{\partial y} \end{aligned} \quad (1)$$

The nonlinear terms in these expressions arise from the fact that the deflection of the membrane in the z direction is large. Using the equilibrium equations, Hooke's law, and the appropriate boundary conditions, the deflection can be calculated. The problem can be reduced to the simultaneous solution of two nonlinear partial differential equations.⁶ This, however, is a nontrivial task.

A number of researchers have derived approximate solutions using an energy minimization method.⁶⁻⁹ In this approach, one assumes a displacement field for the membrane that contains a number of unknown parameters and satisfies the boundary conditions. According to the principle of virtual displacements, the unknown parameters are then determined by the condition that the total potential energy of the system is minimum with respect to the parameters. The displacement field used most often is the first term in the Fourier expansion of the actual deflection. The same method with a different displacement field is used in the present study in order to derive a more accurate expression for the load-deflection behavior of a square membrane. The displacement field for a square film with side $2a$ can be approximated by

$$\begin{aligned} u &= A \frac{x}{a^5} (a^2 - x^2)(a^2 - y^2) \\ v &= A \frac{y}{a^5} (a^2 - x^2)(a^2 - y^2) \\ w &= w_0 \frac{1}{a^4} (a^2 - x^2)(a^2 - y^2) \left[1 + \frac{R}{a^2} (x^2 + y^2) \right] \end{aligned} \quad (2)$$

where A , w_0 , and R are the unknown parameters. The potential energy of the membrane in the case of an

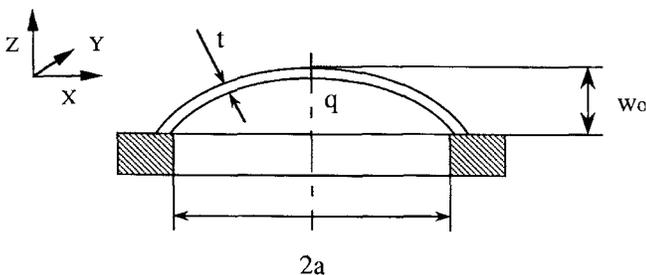


FIG. 1. Schematic diagram of a membrane with a uniform pressure applied to one side.

isotropic material is⁶

$$V = \frac{Et}{2(1 - \nu^2)} \iint (\epsilon_x^2 + \epsilon_y^2 + 2\nu\epsilon_x\epsilon_y + \frac{1}{2}(1 - \nu^2)\gamma_{xy}^2) dx dy - \iint qw dx dy \quad (3)$$

where t , E , and ν are the thickness, Young's modulus, and Poisson's ratio of the film, respectively, and q is the pressure applied to the membrane. The first term in this expression represents the strain energy of the membrane due to the stretching in the plane of the membrane. The contribution of bending to the strain energy has been neglected. This is valid because the deflection is much larger than the thickness of the membrane. The second term represents the potential energy of the pressure applied to the membrane. Minimization of Eq. (3) with respect to the undetermined parameters leads to a set of three simultaneous nonlinear equations in A , w_0 , and R , that can be readily solved. The deflection of the center of the membrane is then given by

$$w_0 = f(\nu) \left(\frac{qa^4(1 - \nu)}{Et} \right)^{1/3} \quad (4)$$

where $f(\nu)$ is a complicated function of Poisson's ratio which can be approximated by $f(\nu) \approx 0.800 + 0.062\nu$. The form of Eq. (4) is the same as that found by other researchers, except for the function $f(\nu)$. A few remarks about Eq. (4) are in order. First, for a given pressure and displacement, Young's modulus is proportional to the fourth power of a . Therefore, if one wants to measure Young's modulus by means of the bulge test, the dimensions of the film have to be measured very accurately. This is often impossible if the film is taken off the substrate and glued onto a sample holder. Second, the fact that $f(\nu)$ is a function of Poisson's ratio arises from the fact that the stress state in the membrane is not entirely equal-biaxial. The assumption of an equal-biaxial stress state has often been made in the derivation of the deflection of circular membranes.^{2,10,11} The strain state actually varies from equal-biaxial in the center of the membrane to plane strain at the edges, where the film is clamped. The biaxial modulus $E/(1 - \nu)$ alone is insufficient to characterize the load-deflection behavior of the membrane and the membrane is more compliant than one would expect based on the assumption of an equal-biaxial stress state. In Fig. 2 the variation of $f(\nu)$ with Poisson's ratio is compared to a finite element calculation of the same quantity⁷ and an energy minimization calculation using the first term of the Fourier expansion of the deflection.^{7,8} Agreement with the finite element calculation is excellent. The Fourier expansion, however, overestimates the compliance of the membrane significantly. The shape of the deflected mem-

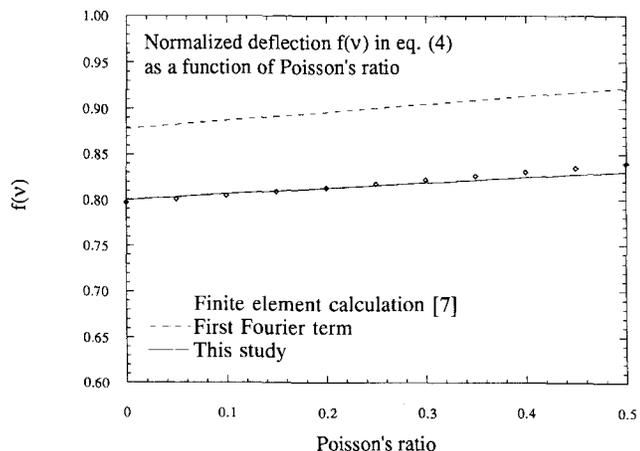


FIG. 2. Variation of the function $f(\nu)$ for square films with Poisson's ratio. The open diamonds correspond to results obtained from finite element calculations.⁷

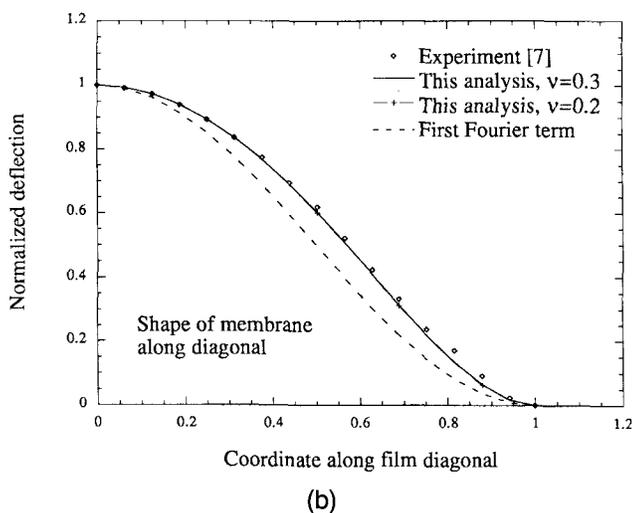
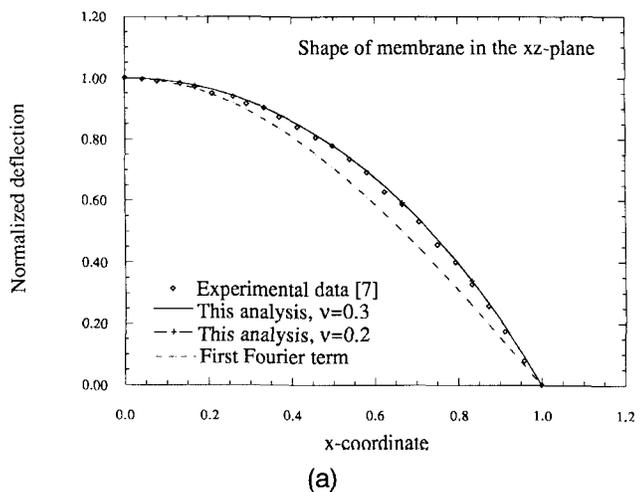


FIG. 3. The deflection of a thin film in the xz -plane (a) and along the diagonal (b). The shape of the membrane is virtually independent of Poisson's ratio of the film.

brane is depicted in Fig. 3. Both the deflection in the xz -plane and along the membrane diagonal are plotted and show very good agreement with experimental data.⁷ Even though the film is clamped along the edges, the slope of the deflection at the edges is not zero because for very thin films and large deflections the bending stiffness of the film can be neglected. According to finite element calculations⁷ the shape of a membrane for a given deflection is independent of Poisson's ratio. Although in this analysis the parameter R in Eq. (2) is a weak function of Poisson's ratio, the shape calculated varies only very slightly with Poisson's ratio.

B. Rectangular films

The load-deflection behavior of a rectangular film with sides $2a$ and $2b$ can be derived using the same energy minimization technique as for square films. The displacement field is very similar to that of square films in Eq. (2) but contains five unknowns instead of three. The resulting load-deflection relation is then

$$w_0 = g\left(\nu, \frac{b}{a}\right) \left(\frac{qa^4(1-\nu)}{Et}\right)^{1/3} \quad (5)$$

where $g(\nu, b/a)$ is a function of Poisson's ratio and the aspect ratio of the membrane. Figure 4 shows the change of $g(\nu, b/a)$ with membrane aspect ratio for three different values of Poisson's ratio. Apparently, a membrane shows a rapidly increasing deflection as its aspect ratio increases above unity, but once the aspect ratio exceeds 5, the deflection is independent of the aspect ratio. There are two limiting cases for which this calculation can be checked. First, for an aspect ratio of 1.0, the solution has to be the same as the one derived previously in this paper. Second, for an infinitely long membrane the

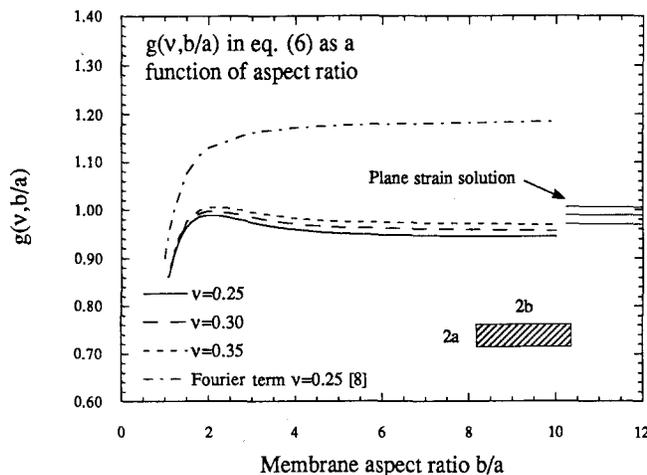


FIG. 4. Variation of the function $g(\nu, b/a)$ with membrane aspect ratio for three different values of Poisson's ratio. The solid lines at the right-hand side are the plane strain solutions (see appendix) for the same Poisson's ratios. For a given aspect ratio, $g(\nu, b/a)$ increases with increasing Poisson's ratio.

strain state must be one of plane strain since any plane perpendicular to the axis of the film is a mirror plane. In this case an exact solution for the membrane deflection can be readily derived (see appendix). The deflection in the center of the membrane is given by

$$w_0 = \left(\frac{6qa^4(1 - \nu^2)}{8Et} \right)^{1/3} \quad (6)$$

so that for large aspect ratios $g(\nu, b/a)$ must approach $[6(1 + \nu)/8]^{1/3}$. The virtual energy solution indeed approaches a limit value which is within 3.7% of the correct solution. The maximum in the plot at an aspect ratio of about two is most likely an artifact arising from the approximations used in the energy method. For small aspect ratios, one should use Eq. (5) for the membrane deflection, whereas Eq. (6) is better for large aspect ratios. In the plane strain case the load-deflection behavior of the membrane is fully determined by the ratio $E/(1 - \nu^2)$. As a result, the deflection does not depend as strongly on Poisson's ratio as for square membranes. Testing of long rectangular membranes therefore allows a more accurate determination of Young's modulus when Poisson's ratio is not exactly known. A similar observation has been made by Tabata *et al.*⁸

Comparing the results for square and rectangular membranes, an interesting observation can be made. If both a square and a much longer rectangular film are tested, the coefficients of q in Eqs. (4) and (6) can be determined. Elimination of Young's modulus from the two coefficients makes it possible to calculate Poisson's ratio of the film from the ratio $f(\nu)^3/(1 + \nu)$. This method will give at least a very good estimate of a quantity that is otherwise very difficult to measure. Since Poisson's ratio is rather sensitive to propagation of experimental errors in the calculations, a sufficient number of films should be tested.

C. The influence of residual stress on the deflection of a membrane

Until now, only membranes without residual stress were considered. The presence of such a stress, σ_0 , can alter the deflection behavior of a membrane considerably. The energy minimization technique used in the two previous sections fails to give a straightforward formula for the deflection in this case, since the nonlinear equations derived from the minimization of the total potential energy of the system have to be solved numerically for each value of the pressure q and stress σ_0 . However, if one assumes that the pressure can be resolved into two components q_1 and q_2 such that q_1 is balanced by the residual stress in the membrane and q_2 by the stretching of the membrane, a solution can be readily derived. An expression for q_1 as a function of the deflection of the

center of the membrane can be written as¹²

$$q_1 = \frac{\sigma_0 t \pi^3}{16a^2} w_0 \times \left[\sum_{n=1,3,5}^{\infty} \frac{(-1)(n-1)/2}{n^3} \left(1 - \frac{1}{\cosh\left(\frac{n\pi b}{2a}\right)} \right) \right]^{-1} = c_1 \frac{\sigma_0 t}{a^2} w_0 \quad (7)$$

whereas Eq. (5) or (6) can be used for q_2 , depending on the aspect ratio of the membrane. The load-deflection relationship for a stressed membrane is then given by

$$q = q_1 + q_2 = c_1 \frac{\sigma_0 t}{a^2} w_0 + c_2 \frac{Et}{a^4(1 - \nu)} w_0^3 \quad (8)$$

where c_2 is given by $g(\nu, b/a)^{-3}$ or $8/6(1 + \nu)$, depending on the aspect ratio. Figure 5 shows c_1 as a function of membrane aspect ratio. The constant is independent of material properties and has a value of 3.393 for square membranes, decreases rapidly as the aspect ratio increases, and reaches a value of 2 for infinitely long membranes. The conditions in which Eq. (8) holds are that c_1 does not change for large deflections and that c_2 is not a function of σ_0 . Again, Eq. (2) can be checked for two limiting cases. In the case of plane strain, the problem can be solved analytically and Eq. (8) gives the correct solution (see appendix). Finite element calculations have been done to study the influence of residual stress on the deflection of circular and square membranes.^{3,4,7} According to Lin,⁷ c_1 is constant and equal to 3.41. This is in very close agreement with Eq. (8). The same study also shows that at least for circular films, c_2 is independent of the residual stress in the film. More recent calculations for circular films,^{3,4} however, have shown that c_2 is a weak function of residual stress. One would expect Eq. (8) to

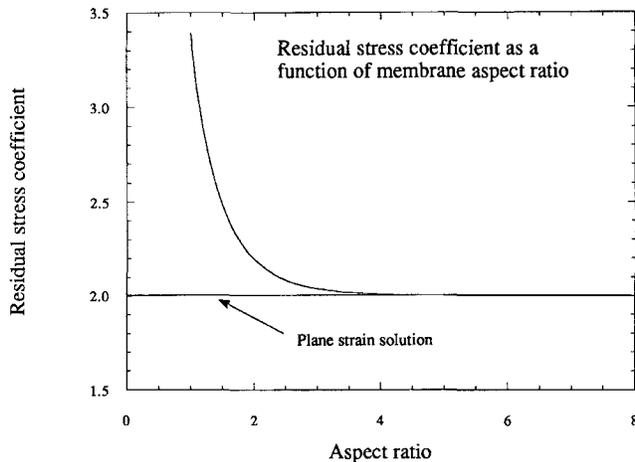


FIG. 5. The residual stress coefficient c_1 as a function of film as-

be quite accurate as long as the residual stress is not too high, i.e., smaller than 0.5 GPa.

According to Eq. (8) a plot of load versus deflection at the center of a membrane is a cubic parabola, the slope of which at zero deflection is determined by the residual stress in the film. The nonlinear term, on the other hand, yields information about Young's modulus. By fitting Eq. (8) to experimental data from the bulge test, both Young's modulus and residual stress can be calculated.

III. EXPERIMENTAL

A. Measuring apparatus

A schematic of the bulge tester used in this study is shown in Fig. 6. The sample to be tested is glued onto a sample holder and pressure is applied to one side of the film, by pumping water into the cavity under the film. The deflection of the film is measured by means of a laser interferometer with a He-Ne laser light source. The displacement resolution is half the wavelength of the light, i.e., $0.3164 \mu\text{m}$. The pressure is measured with a pressure transducer with a resolution of 70 Pa. A maximum pressure of 100 kPa can be applied. The experiment is controlled by computer via a data acquisition system.

B. Sample preparation

The nitride films examined in this study were deposited by means of LPCVD. The deposition temperature was 785°C and the gas pressure was 300 mTorr. The ratio of dichlorosilane to ammonia was 5.2:1. The deposition rate was $30 \text{ \AA}/\text{min}$ and the final thickness

of the films was approximately 2900 \AA . The substrates were (100) oriented *n*-type Si wafers, between 200 and $250 \mu\text{m}$ thick. The films were deposited on both sides of the wafers. Both square and rectangular windows with various aspect ratios were etched in the silicon nitride on one side of the wafers using standard lithographic techniques and plasma etching. In order to make free-standing films, the exposed silicon was etched using an anisotropic etchant containing potassium hydroxide and methanol at a temperature of 65°C . A typical film is shown in Fig. 7. The thickness of each membrane was measured by means of ellipsometry. Finally, a thin aluminum coating was deposited onto the silicon nitride to enhance the reflectivity of the films. Tests were performed on each of five square membranes and on eight rectangular films with aspect ratios varying from 1.2 to 4.9.

In order to have an independent check of the results of the bulge test, Young's modulus of the silicon nitride was also measured by means of continuous indentation testing. The indentations were performed on the film using a Nanoindenter, a high-resolution depth-sensing hardness tester, the description of which can be found elsewhere in the literature.^{13,14} Both applied load and displacement were continuously recorded during the experiments. A total of 36 indentations were made to plastic depths ranging from 20 to 60 nm. The depths of the indentations were small enough that only film properties were measured. The velocity of the indenter upon loading was between 3 and 6 nm/s. When the desired indentation depth was reached, the load was held constant for 15 s and then decreased at a rate equal to the last loading rate. Hardness and Young's modulus were calculated from the load-displacement curves using the analysis given by Doerner and Nix.¹³

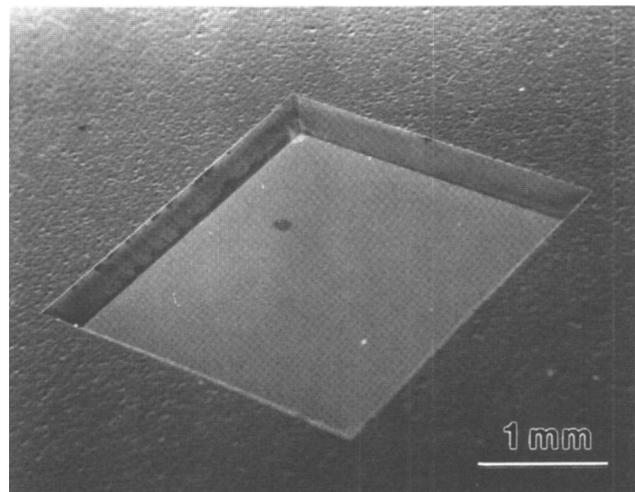
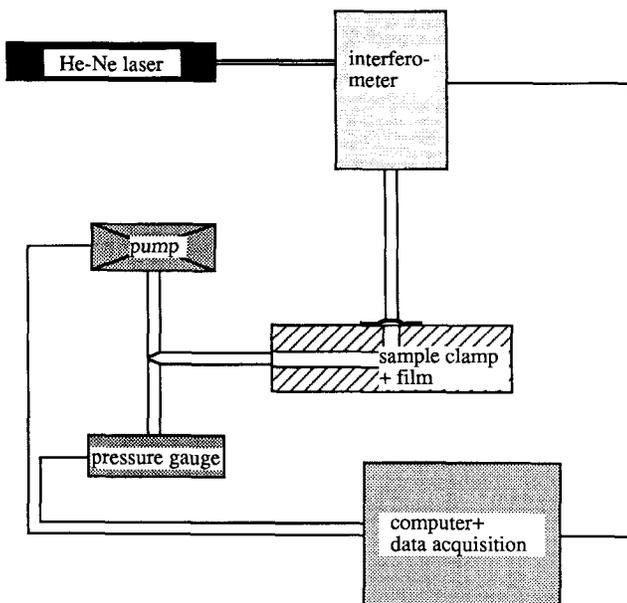


FIG. 7. Scanning electron micrograph of a square SiN_x window in a

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