

**Load Deflection Analysis for Determining  
Mechanical Properties of Thin Films with  
Tensile and Compressive Residual Stresses**

by

**Mayank T. Bulsara**


B.S., Ceramic Engineering, 1993  
Rutgers University, New Brunswick, NJ


Submitted to the Department of Materials  
Science and Engineering in Partial  
Fulfillment of the Requirements for the  
Degree of  
Master of Science

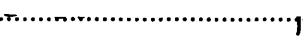
at the

Massachusetts Institute of Technology  
February 1995

© Massachusetts Institute of Technology 1995  
All Rights Reserved

Signature of Author .....  .....  
Department of Materials Science and Engineering  
January 20, 1995


Certified by .....  .....  
Stuart B. Brown  
Richard P. Simmons Associate Professor of Materials Manufacturing  
Thesis Advisor

Accepted by .....  .....  
Carl V. Thompson II  
Professor of Electronic Materials  
Chair, Departmental Committee on Graduate Students

MASSACHUSETTS INSTITUTE  
OF TECHNOLOGY

JUL 20 1995

LIBRARIES

Science  


Load Deflection Analysis For Determining Mechanical Properties of Thin Films  
with Tensile and Compressive Residual Stresses

by

Mayank T. Bulsara

Submitted to the Department of Materials  
Science and Engineering on January 20, 1995 in Partial  
Fulfillment of the Requirements for the  
Degree of Master of Science

## Abstract

An examination of load deflection (bulge) testing of coated and uncoated membranes by analytical and finite element methods was undertaken. The key parameters in the load-deflection behavior of a membrane were non-dimensionalized and examined to illustrate how to design test structures for materials systems of interest. Experimental and modeling procedures were examined to improve measurement of membrane response. An approach to test membranes with compressive residual stresses was proposed and used for determining the stress state for a  $p^{++}$  Si/SiN<sub>x</sub> system with and without a deposited coating.

**Thesis Supervisor:** Stuart B. Brown

**Title:** Richard P. Simmons Associate Professor of Materials Manufacturing

## Acknowledgments

I would like to thank Professor Martin Schmidt and Dr. Howard Goldberg for their guidance and expertise with membrane fabrication. My sincere thanks to Professor Stephen Senturia, Daniel Sobek, and Raj Gupta for their assistance with load deflection testing. I would also like to thank Dr. James Chen and the Gillette Company for their support of this work.

I wish to thank the fellow members of the Constitutive Modelling Group at M.I.T.: Patricio Mendez, Chris Rice, Will Van Arsdell, and John Wlassich for their encouragement and assistance. They have been the best of friends, as well as colleagues, through the past year.

Among the individuals I would like to thank for adding to my experience at M.I.T. are Jason Sprague, Kamala Crawley, Chris Wilson, and Laura Hatosy. I look forward to their continuing friendships.

I would like to thank Professor Stuart Brown for his patient tutoring and guidance during the course of this work. Professor Brown has been an excellent mentor and embodies the standards of professional excellence to which I will aspire for the rest of my career.

Finally, I want to thank my parents for their love and support throughout my life.

# Contents

Acknowledgments .....	3
1.0 Introduction.....	5
2.0 Procedure .....	5
3.0 Compressive Stress Computation .....	7
4.0 Analysis of Load Deflection Testing .....	12
5.0 Experimental and Modelling Considerations .....	16
6.0 Evaluation of p <sup>++</sup> Silicon/SiN <sub>x</sub> System .....	20
7.0 Conclusions and Future Directions.....	23
8.0 Bibliography .....	24
List of Figures.....	26

## 1.0 Introduction

An increasingly important aspect of thin film characterization is the ability to examine mechanical properties of thin film materials systems. The ability to characterize these properties is important for VLSI processing and materials systems used for micro-electromechanical structures (MEMS) [1,2]. The mechanical properties of thin film materials can be very different from the bulk properties of the same materials, so bulk properties cannot be assumed in all systems [3].

The idea of using a fluid to apply a pressure to one side of a membrane and examining material properties from the subsequent bulge was first introduced by Beams [4]. There have been many efforts to examine the behavior of membranes under pressure from one side to evaluate biaxial modulus, residual stress, and ultimate strain of thin films [2,5,6,7,8]. However, all the models are for thin films under a tensile state of stress [2,5]. In addition a proper analysis of how one would design and carry out experiments for a load deflection analysis has not been conducted.

## 2.0 Procedure

We used the load deflection model introduced by Allen and further developed by others [7,8,9,10,11]. Allen assumed the parameterized functions that represent of the membrane displacements of a pressurized membrane in the  $x$ ,  $y$ , and  $z$  direction and calculated the resulting strains. With the strains, he found the function parameters which minimized the stored elastic energy of the membrane. The profile of the membrane could then be determined. The functional form of the equation was verified with finite element analysis and more accurate values for the function parameters were derived. The final expression is as follows

$$p = C_1 \left( \frac{\sigma_o t}{a^2} \right) d + C_2 f(\nu) \left[ \frac{E}{(1-\nu)} \right] \left( \frac{t}{a^4} \right) d^3 \quad (1)$$

Here  $p$  is the pressure,  $d$  is the center deflection,  $t$  is the thickness,  $E$  is the Young's modulus,  $\sigma_o$  is the residual stress,  $\nu$  is the Poisson ratio,  $a$  is the radius/half-edge length,  $f(\nu)$  is a function of Poisson's ratio and geometry, and  $C_1$  and  $C_2$  are dimensionless constants which

are a function of geometry. For a square membrane  $C_1=3.41$ ,  $C_2=1.37$ , and  $f(v)$  is as follows

$$f(v) = 1.446 - 0.427v \quad (2)$$

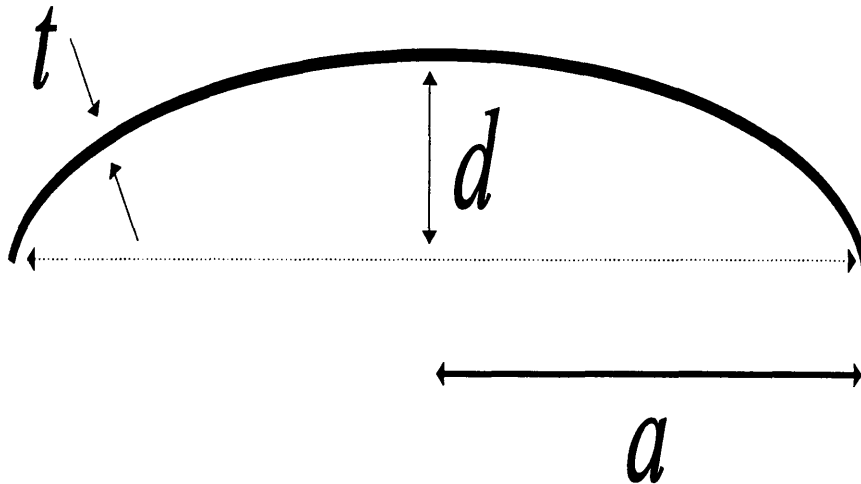
We chose to use square membranes for our testing structures because anisotropic etchants, which preferentially attack the {100} plane of silicon, allow easy fabrication of square structures whose sides are coincident with {011} planes [12,13]. We also assumed a Poisson ratio of 0.25, which would be a typical value for most covalently bonded materials [14]. With those parameters chosen equation 1 simplifies to

$$p = 3.41 \left( \frac{\sigma_o t}{a} \right) d + 2.44 \left( \frac{Et}{a} \right) d^3 \quad (3)$$

To analyze these quantities in the most general form we non-dimensionalized equation 3 and to arrive at

$$\frac{p}{E} = 3.41 \left( \frac{\sigma_o}{E} \right) \left( \frac{t}{a} \right) \left( \frac{d}{a} \right) + 2.44 \left( \frac{t}{a} \right) \left( \frac{d}{a} \right)^3 \quad (4)$$

Figure 1 is a schematic cross-section of a deflected membrane which identifies the key geometrical parameters in equation 4.



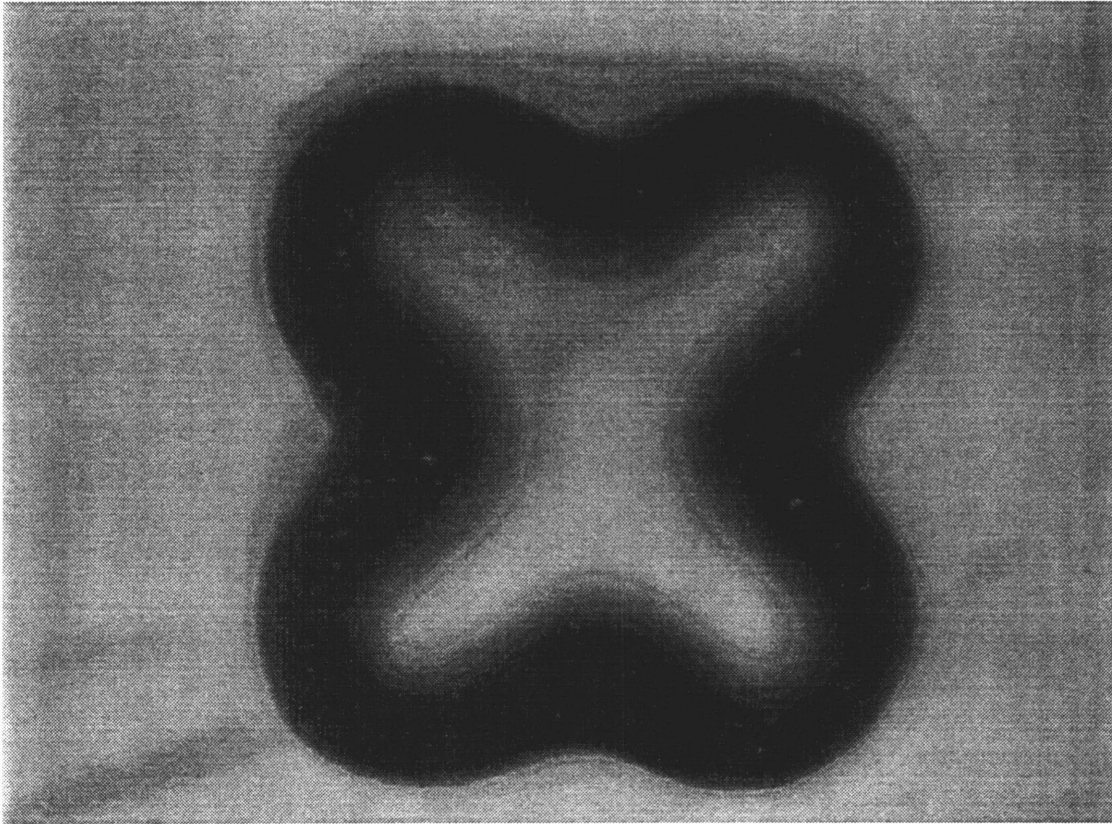
**Figure 1: Schematic of Deflected Membrane**

At this point it is worth noting that if an experimenter is interested in determining the optimum membrane for testing a thin film, the parameters which can be measured or controlled must be identified. The amount of pressure that can be applied to a membrane and the resolution to which one can detect its center deflection are frequent operational constraints, and must be determined first. If a measurement apparatus is designed to apply a limited pressure and cannot distinguish deflections with a resolution better than 10 microns, the extent to which the experiment can be engineered by other parameters is greatly limited. Secondly, the residual stress and the Young's modulus of the film to be measured, although not controllable variables, have a great effect on the pressure to deflection relationship. Since one is interested in measuring these properties, their exact values will not be known, but a rough estimate based on the type of material system involved (i.e. ceramic, metallic, polymeric) would improve the ability to select the proper testing system. The thickness and half-edge length are perhaps the most flexible parameters in an analysis, as they can be changed by orders of magnitude. A thin film coating can be on the order of a few hundred angstroms to a few microns in thickness. The half-edge length can be varied through a vast range due to the ability to pattern silicon to dimensions precise to the micron. In our analysis we looked at ranges which were feasible for most testing systems. Thickness/half-edge ratio ranged from  $10^{-2}$  to  $10^{-4}$ . Deflection/half-edge length ratio ranged from 0.00 to 0.07. Pressure/Young's modulus ratio ranged from  $10^{-11}$  to  $10^{-6}$ , and residual stress/modulus ratio varied from  $10^{-4}$  to  $-10^{-4}$ .

### **3.0 Compressive Stress Computation**

We propose that a film under a compressive state of membrane stress where the membrane is buckled, but not overly deformed, can have its residual stress measured. Figure 2 is a top view of a membrane which has buckled, but not fractured. The use of finite element modelling in coordination with a load deflection measurement can yield an approximate measurement of its stress state..

Most finite element codes are not capable of simulating a membrane with a compressive residual stress state directly. We propose that such a geometry and material state can be simulated via the imposition of a fictitious temperature change. A plate or mem-



**Figure 2: Top View of Buckled Membrane**

brane with its edges fixed, when experiencing a positive temperature change, will develop a compressive in-plane stress state. If the material could somehow keep its edges fixed and buckle, the stress could be accommodated, but at the expense of making the membrane more compliant. It can be reasoned that for a given applied pressure, a membrane under an initial compressive stress state would experience a greater deflection than if it were under a tensile state of stress. With this analysis a method for measuring a compressive stress state was formulated as follows:

- 1] Experimentally pressurize the membrane to a point where the membrane is bulged and the buckling is not apparent.
- 2] Use an FEM analysis to apply the same pressure to a simulated membrane of the same dimensions, and apply a sufficient temperature change to yield the exact deflection obtained experimentally.
- 3] An imposed stress state can be related to the simulated temperature change by

$$\sigma_{11} = \sigma_{22} = -E\alpha\Delta T \quad (5)$$

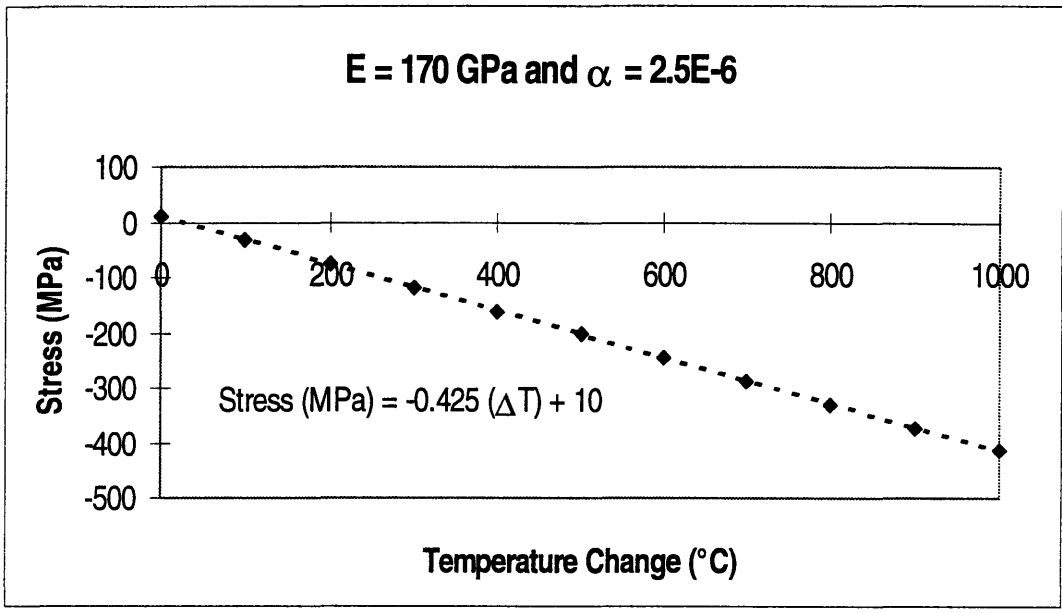


in the case where there are no strains (i.e. the edges are clamped). Numerical techniques may also be used to correlate simulated temperature changes to imposed stress states.

We implicitly assume that the residual stress acts linear elastically with the stresses introduced in the film upon pressure deflection. In other words, we assume it makes no difference if we apply pressure first, and then introduce a residual stress state. This contrasts to the actual situation where the stress state exists initially, and then we apply pressure.

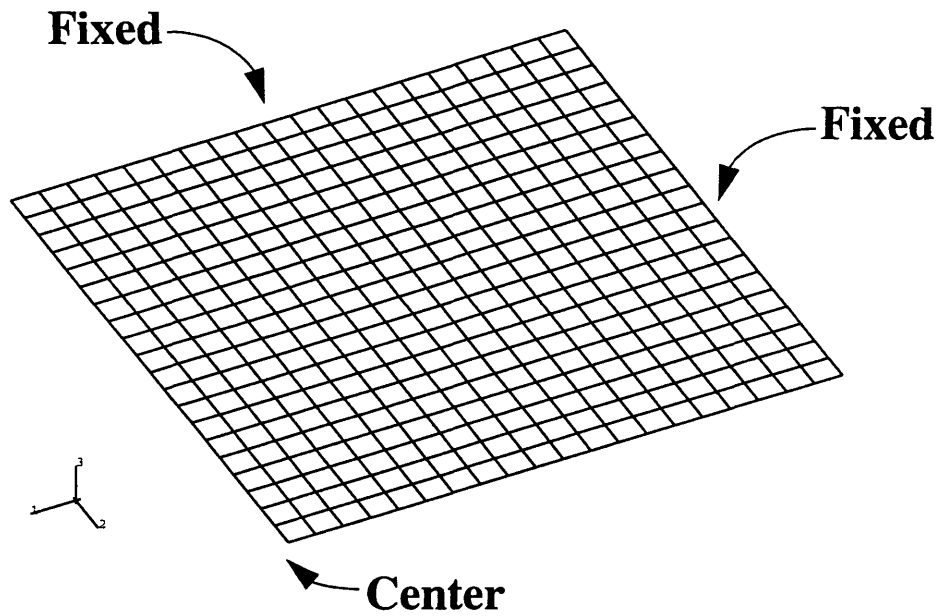
It should be emphasized from the beginning that in order to conduct the procedure above the moduli of the materials system must be determined independently of the load deflection technique. There are number of techniques which could be used to determine such elastic properties (nanoindentation, microbeam deflection, etc.) [15]. With the moduli known, a calibration curve of compressive stress as a function of temperature for the system must be developed. Using Abaqus 5.3 we implemented a single-shell element model fixed at all edges and applied temperature changes to obtain a stress versus temperature change curve. It is important to note that the simulated stresses are independent of thickness and edge length of the single shell element and that stresses in the membrane can be simulated by varying either the expansion coefficient or temperature change. We could keep a fixed temperature change and vary the expansion coefficient to obtain a calibration curve, but the method of varying temperature is more intuitive. Figure 3 is a plot of a calibration curve for a silicon membrane, assuming isotropic behavior and a Young's modulus of 170 GPa.

The single element used in our calibration had an initial tensile stress state of 10 MPa. This initial tensile stress state was not necessary for the calibration curve. However, in order for Abaqus 5.3 to simulate a membrane which undergoes pressurization, an initial tensile stress state is necessary. The initial tensile stress state in the calibration curve was introduced so the calibration curve had the same reference residual stress state as future simulations. The behavior was linear, as expected, and is depicted exactly by equation 5. For a single layer, isotropic system equation 5 is sufficient. We chose to use Abaqus 5.3 to simulate multi-layer membranes and anisotropic materials systems. If the system were a multi-layer thin film the individual layers could be examined with equation 5 or single-layer shell element models, but the composite stress state would have to be a weighted



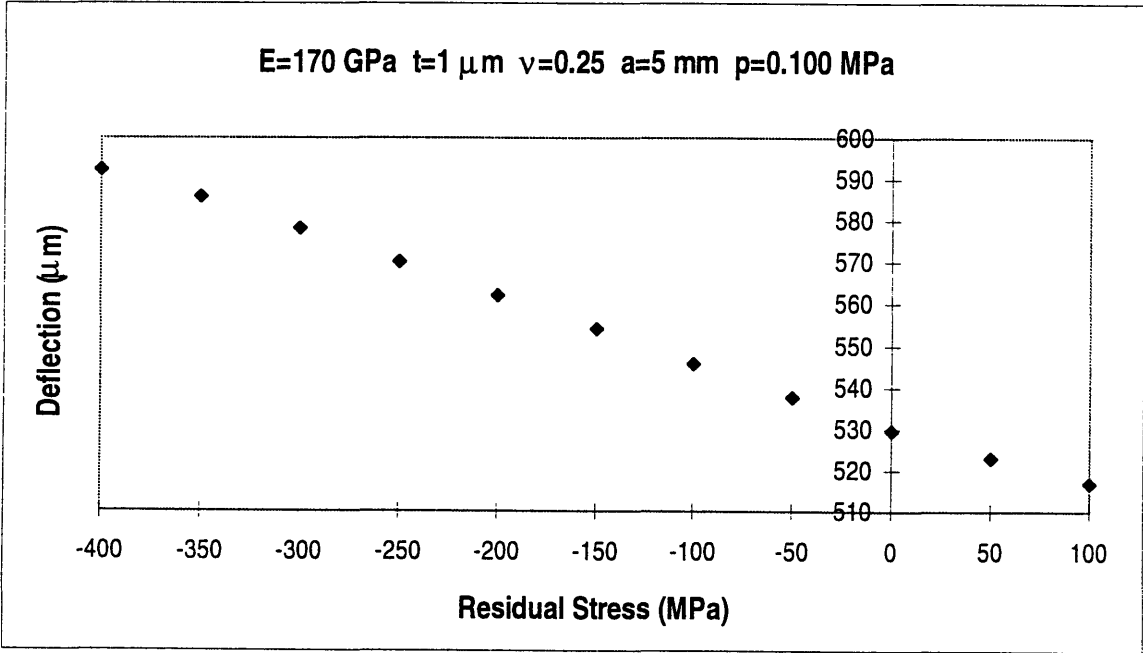
**Figure 3: Calibration Curve of Imposed Stress vs.  $\Delta T$**

**ABAQUS**

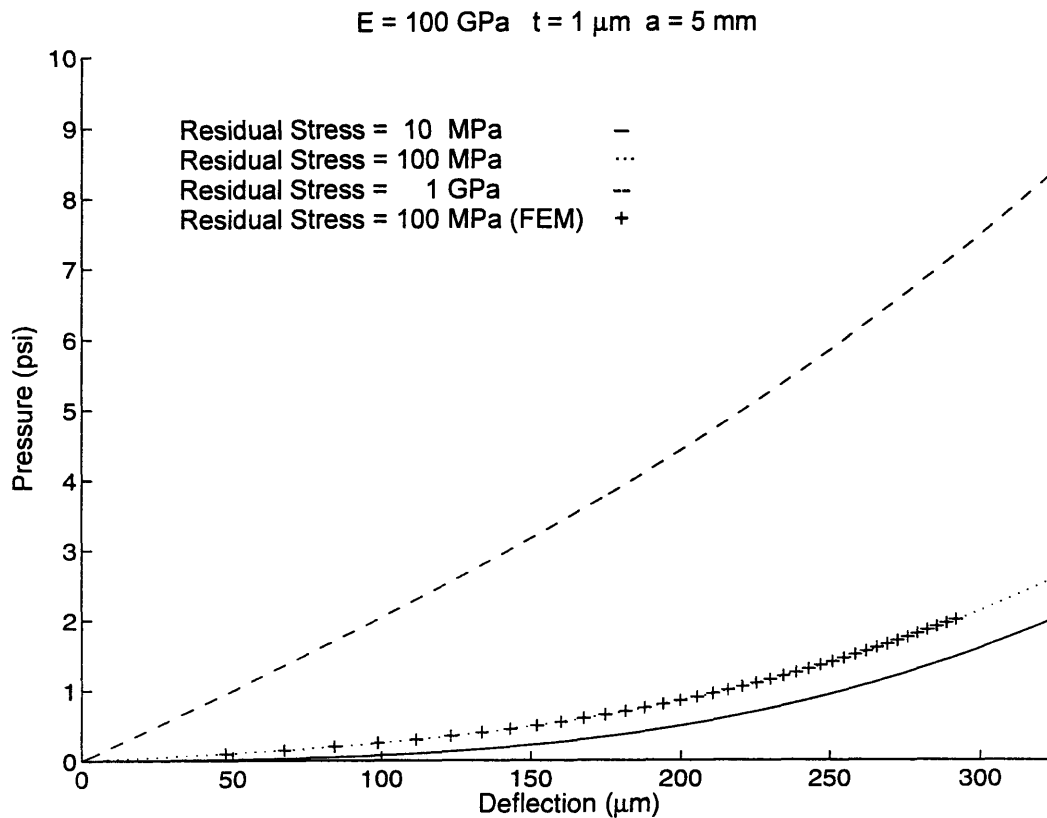


**Figure 4: 200 Element Mesh of 1/4 Membrane**

average of the stress states in the individual layers. We verified our model by imposing stresses, based on the calibration curve from figure 2, on a model of a typical silicon membrane (only one-quarter of the membrane need be modelled due to symmetry) under reasonable loading conditions. Figure 4 is a plot of our finite element mesh which shows one-quarter of a membrane with two edges fixed. Figure 5 is a plot of deflection versus residual stress state for a given half-edge length, thickness, Poisson ratio, and applied pressure. The stress states from 10 MPa in tension and up were imposed directly with the initial conditions parameter available through Abaqus. The values below 10 MPa were imposed via the calibration curve from figure 2. There are two important observations from this curve: 1) the deflections for the modelled membrane, experiencing a fixed pressure, increased as the residual stress state became progressively compressive; 2) the deflection behavior of the models with the stresses imposed via the calibration curve varied continuously from the deflection of the membranes with the tensile stress imposed directly.



**Figure 5: Maximum Deflection vs. Residual Stress State**

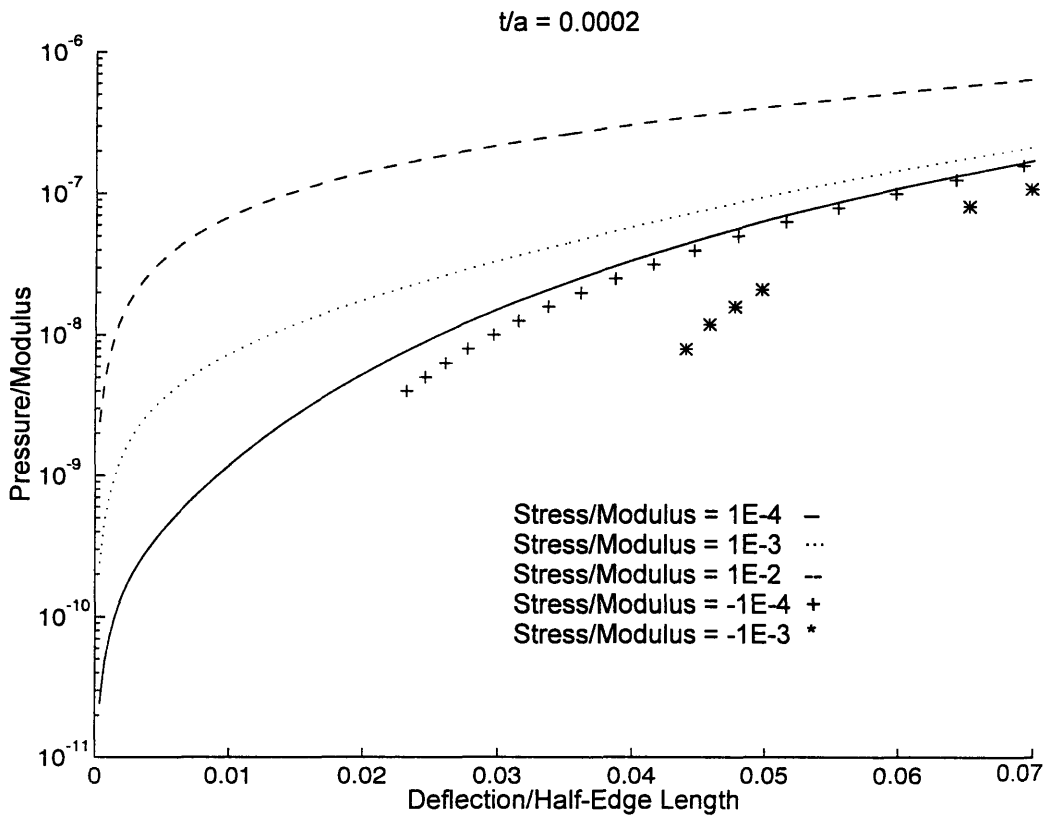


**Figure 6: Pressure - Deflection Plot for a Typical Membrane Sample**

## 4.0 Analysis of Load Deflection Testing

This part of our analysis incorporates equation 3 and our proposed system for determining compressive stress states to examine how one could properly design a load-deflection experiment.

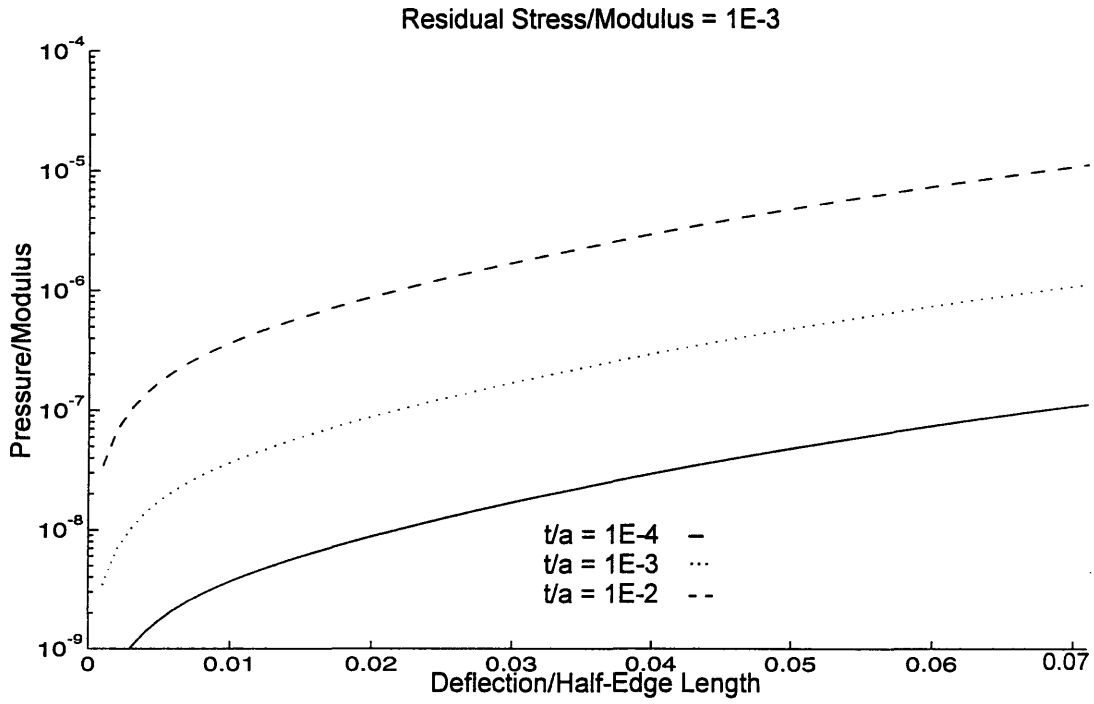
Figure 6 is a plot of equation 3 for three typical membranes. In addition we applied our finite element model to show that our analysis is consistent with the accepted energy minimization solution. Figure 7 is a plot of pressure/modulus (from this point we will use the term Modulus to implicitly refer to Young's modulus) plotted against different residual stress/modulus ratios with a fixed thickness/half-edge length ratio. The stress to modulus ratios vary from tensile to compressive regimes. For a fixed modulus and half-edge length the sensitivity to residual stress state is greatest at low pressures and small deflections. At higher pressures the behavior of the membranes starts to converge. An experimenter can use this plot a number of ways, but one experimental methodology will be hypothesized. With the knowledge of an approximate stress/modulus ratio and a



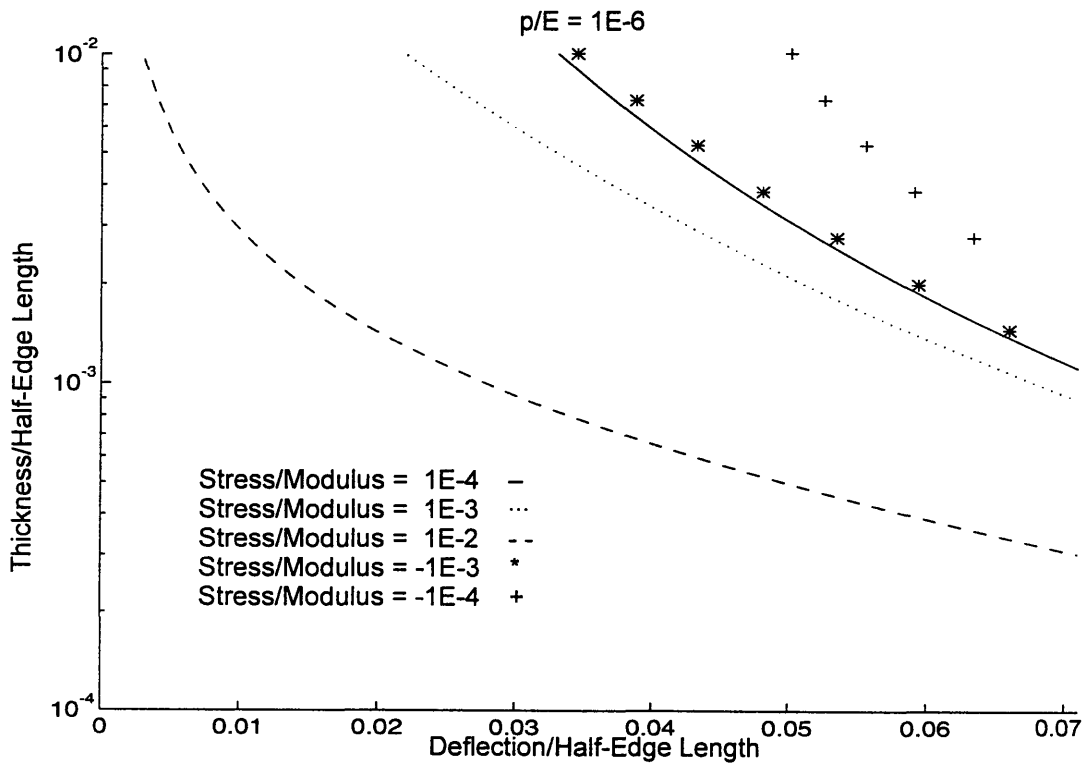
**Figure 7: Effect of Residual Stress/Modulus Ratio on Load Deflection**

defined deflection resolution, one can decide on a pressure system and membrane size which maximizes fabrication efficiency and reduces complexity in the procedure to mount the membranes. It will be discussed later that the method of mounting membranes is very important for accurate deflection measurements.

Figure 8 plots pressure/modulus versus deflection/half-edge length with variations in the thickness to half-edge length ratio. From equation 3 it can be seen that a change in thickness/half-edge length should have lead to a linear response in the  $p/E$  versus  $d/a$  relationship, and the plot shows such a response. An examination of this figure illustrates what was stated earlier in that the two most controllable parameters in a load deflection experiment are the thickness and half-edge length. For a system with a given modulus and half-edge length, an order of magnitude decrease in the  $t/a$  ratio leads to a greater range of deflections for a given pressure range. If an experimenter uses an approximate modulus and had a fixed deflection resolution and pressure range, an optimum thickness and membrane size could easily be determined. This allows the most efficient use of fabrication



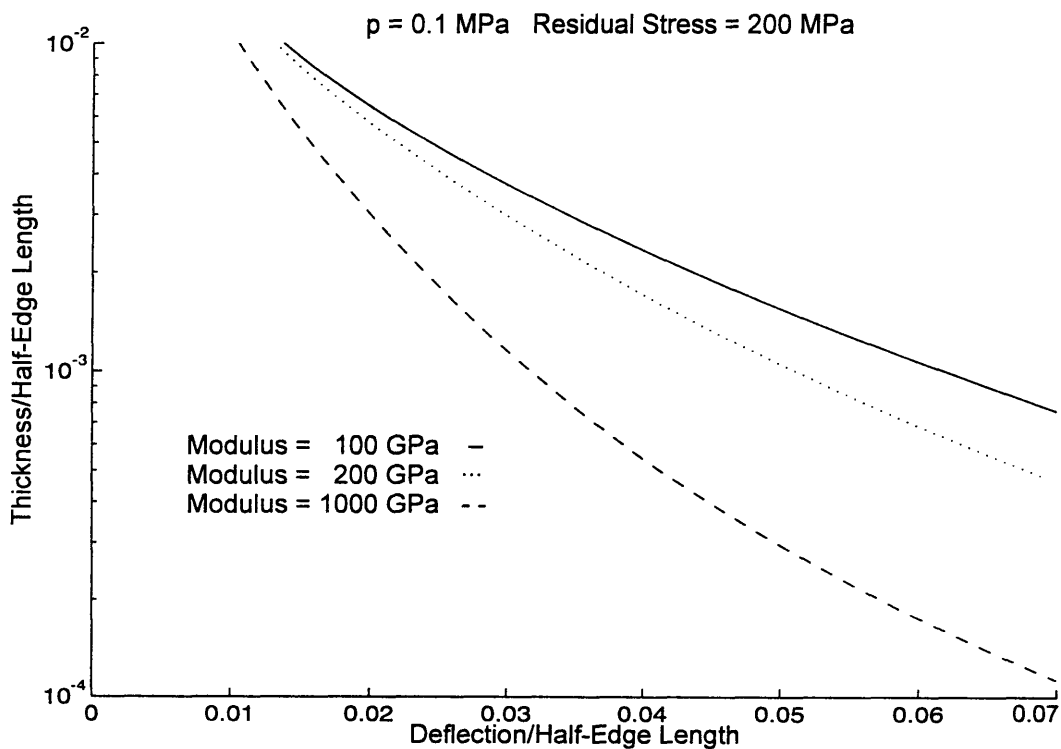
**Figure 8: Effect of Thickness/Half-Edge Length Ratio on Load Deflection**



**Figure 9: Effect of Residual Stress/Modulus Ratio on Load Deflection**

time, as fabrication of samples could easily take up to a number of weeks. Figure 9 is a plot of the effect of stress/modulus on  $t/a$  versus  $d/a$  with a fixed  $p/E$  ratio. Again the plot shows the trend that one would expect, in that membranes become increasingly compliant as the stress state becomes more compressive. The thickness of deposited thin film layers is a parameter that one can control, but in some systems it is limited by the stress state. For example, in systems which are under large tensile states of stress the thickness of deposited layers are limited by the ability to grow films which do not have critical flaws. If an experimenter was limited by this (i.e. the thickness is fixed), the edge length and maximum pressure applied could be modulated to determine the maximum deflection that one can obtain with a designed membrane. Thus, with the deflection resolution of a system determined, the total number of load deflection points which can be determined from an experiment is easily computable.

Figure 10 is a plot of  $t/a$  versus  $d/a$  with a fixed residual stress state and pressure.



**Figure 10: Effect of Modulus on Load Deflection for a Given Pressure**

This plot shows quantitatively that as the  $t/a$  ratios decrease the ability to distinguish Young's modulus increases. This is important if load deflection tests were being used to characterize membranes made from similar materials classes. The ability to detect a difference in modulus between two similar polymers would be much more difficult at high  $t/a$  or low  $d/a$  ratios. If an approximate modulus and residual stress state were known for a materials system, and the pressure was fixed, a good estimate for the maximum deflection for various membrane sizes could be extracted. A subsequent comparison of predicted maximum deflections can help the experimenter decide which testing system is most adequate for distinguishing between two similar materials systems.

Figures 6 through 10 do not encompass all materials systems, as there are quantities held constant in all simulations. However, in any load deflection system there are fixed quantities which are due to fabrication and materials constraints. This section was included to give insight to experimenters who do not have the experience to predict which test designs give the most information given simple test equipment and without excessive sample fabrication.

## 5.0 Experimental and Modelling Considerations

We earlier proposed a model for evaluation of membranes with compressive stress states, and established its merit with finite-element modelling. However, the model alone cannot be used to evaluate the stress state of a real system. An actual load-deflection measurement must be done in order for the model to be implemented.

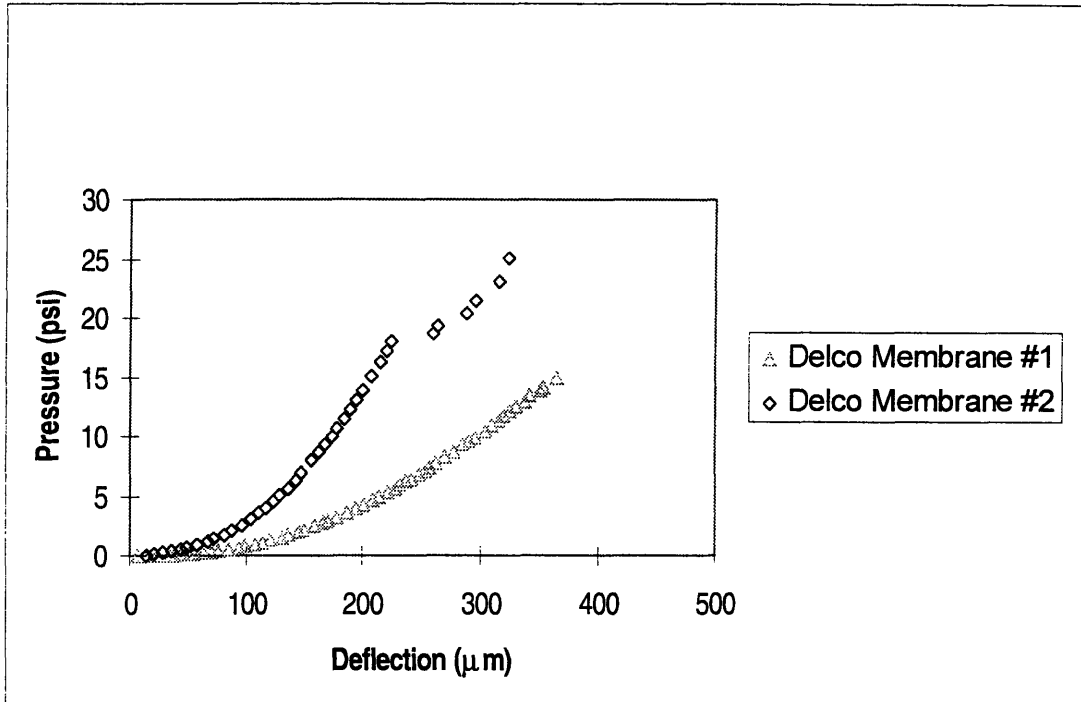
It is within the resolution of modern equipment to distinguish the deflection differences between membranes whose residual stress states are different by a few MPa. Even though measuring deflections as small as submicrons is feasible, the true difficulty is encountered in ensuring that the deflection that is measured is solely due to the membrane response. The handling and mounting of the membranes is extremely important, as we noticed in our experiments.

Our membranes were fabricated from two sets of wafers, one set being composed of in house  $p^{++}$  (100) silicon, and the other being  $p^{++}$  (100) silicon wafers fabricated at GM Delco. The in-house wafers were doped with boron with a solid source diffusion for 8 hours at 1125 °C. The Delco wafers were also doped with boron through a solid source



diffusion step. The in-house wafers had a doping profile which levelled off from  $10^{20}/\text{cm}^3$  at approximately  $6\ \mu\text{m}$  from the surface. The Delco wafers had a doping profile which levelled off from  $7 \times 10^{19}/\text{cm}^3$  at approximately  $8\ \mu\text{m}$  from the surface. All wafers had a  $1100\ \text{\AA}$  layer of LPCVD silicon nitride deposited on them. After the membrane patterns were placed on one side of the wafer with conventional photolithography techniques, the membranes were fabricated with a KOH etch at  $85\ ^\circ\text{C}$ . Although the etch stop and membrane thickness could be predicted from the doping profile, the actual thickness of the membrane was measured with an electronic wafer thickness measuring system. The membranes from Delco wafers were approximately  $6\ \mu\text{m}$  thick and the membranes from the in-house wafers were approximately  $7\ \mu\text{m}$  thick. The membranes from the Delco wafers were buckled. The source of this behavior was not investigated in this work, but the origin of buckling in other  $p^{++}$  silicon structures has been reported [16]. We used this system to test our proposed technique for evaluating the residual stress state of membranes under compression.

With our early experiments we found results which were not very reproducible. Figure 11 shows two load deflection plots for identically sized membranes from the same Delco wafer. The load deflection behavior is not only inconsistent, but sharp discontinuities occur at high pressures. This behavior is not consistent with plastic behavior, as the materials system we were evaluating is brittle and should fail catastrophically. We found similar behavior in other tests and believed that the epoxy which was used to mount the membranes may have lost adhesion at some point along the edge of the membrane. This type of behavior would lead to stray measurements at low pressures as well. If the substrate was not properly mounted, the deflection behavior of the area surrounding the membrane would also be measured and potentially misinterpreted as membrane behavior. In addition the clamping of the mounts in the vicinity of the membranes induces other stresses into the measurement. To alleviate some of these concerns we propose utilizing a mount which isolates the membrane from the clamped region, and a careful mounting of the membrane to ensure that the deflection response is due solely to the fabricated membrane and not from the surrounding substrate region. Figure 12 is a schematic representation of the type of mounting system we recommend. A mechanical method of securing the membrane to the mount would perhaps be the most reproducible and reliable. However,



**Figure 11: Comparison of Load Deflection Behavior of Identical Membranes**

the previous problem of introducing stresses near the membrane might be difficult to avoid with such a procedure.

The ability to anisotropically etch silicon was stated as one of the major reasons for favoring square membrane fabrication and analysis. Silicon is an orthotropic material where a 180° rotation about any one of three mutually orthogonal axes leads to an identical structure. The symmetry in an orthotropic material also requires that no interaction take place between the shear and normal components of stress and strain. The stiffness matrix values for silicon at 298 K compiled by Simmons and Wang are given by  $C_{11} = 165.8$  GPa,  $C_{12} = 63.9$  GPa, and  $C_{44} = 79.6$  GPa [17]. We examined the effect of treating silicon as an orthotropic material and changed the local orientation of our membrane simulation to make the edges to be coincident with {011} planes. Figure 13 displays the results of our analysis which depict a notable difference in load deflection behavior. At moderate pressures the deflections differed to an extent which would be easily distinguishable in an experiment. This effect would not be evident in amorphous or polycrystalline thin films, but should be considered when examining single crystal silicon or other epitaxial films.

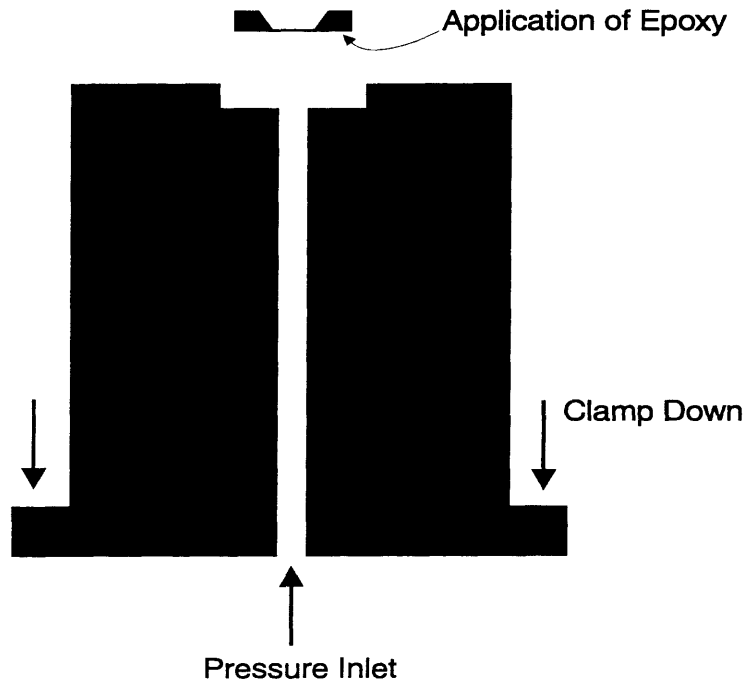


Figure 12: Schematic of Suggested Mounting Apparatus

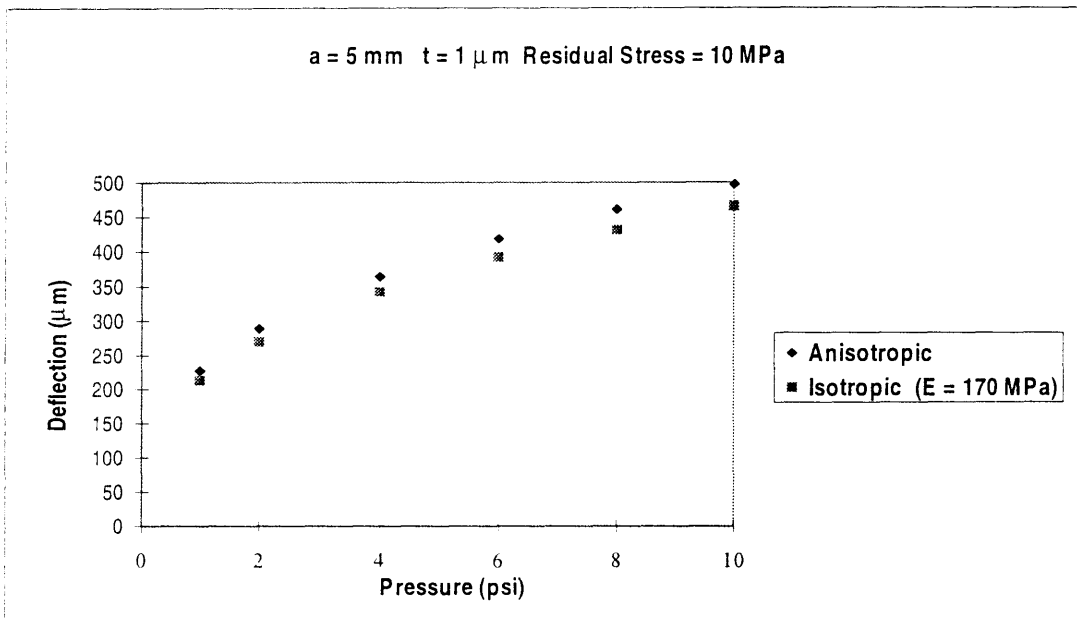


Figure 13: Simulation of Silicon Membrane as Isotropic and Orthotropic Material

## 6.0 Evaluation of $p^{++}$ Silicon/ $\text{SiN}_x$ System

After altering our load deflection testing mount and incorporating the orthotropic behavior of silicon in our finite element model, we were able to conduct our procedure to evaluate compressive stress states on two materials systems.

The load deflection measurement was carried out by mounting the membrane on a customized pressurizing apparatus on the stage of a Nikon UM-2 microscope. The stage was equipped with two Boeckler model 9598 digital micrometers which in turn were connected to a Metronics Quadra-Check II digital readout box. Standard routines within the Quadra-Check II were used to locate the center of the membrane and determine the membrane half-edge length. A 40X objective with a numerical aperture of 0.5 was used to focus on features of the membrane. A Microswitch 142PC30G pressure sensor was used to measure the pressure applied to the membrane. The pressure was applied with in-house nitrogen lines. A Mitutoyo digimatic indicator was used to track motion of the objective lenses in the  $z$  direction.

The shape of buckled membranes are not consistent and as a result, the initial deflection/position of the center of the membrane does not tend to a single value for every membrane. In order to compare load deflection tests of buckled membranes, we chose the zero deflection (flat membrane) point to serve as a reference point and normalized the load deflection curves to this point. For example, if the center of the membrane was initially below the flat position by  $5\ \mu\text{m}$  the load deflection curves were shifted a  $5\ \mu\text{m}$  deflection. Similarly, if the center of the membrane was initially deflected upward and was above the flat position, the deflection values were augmented by this amount and the load deflection curves were shifted to the right. The effect of mounting the membrane at an angle was also corrected by noting the height difference between two corners of the body diagonal of the membrane and extrapolating to where the center of the membrane would be if the membrane were perfectly flat. To illustrate, if we chose one corner of the membrane to be at  $z=0$  and measured the corner at the other end of the body diagonal to be at  $z=10$ , we would have chosen the flat membrane position to be at  $z=5$ . The deflection of the center of the membrane would be normalized relative to  $z=5$ .

Figure 14 shows the load deflection behavior of two membranes. Both membranes

were tested to failure and had their load deflection curves normalized to the flat position. The first membrane tested was Delco p<sup>++</sup> silicon membrane with 1100 Å of LPCVD silicon nitride on top ( $a = 5.115$  mm;  $t = 6.11\mu\text{m}$ ). To evaluate the compressive residual stress state we needed to examine one point on the curve with our finite element model. The point we chose is indicated on figure 14 and corresponds to  $p = 5.375$  psi and  $d = 226$   $\mu\text{m}$ . The materials parameters we specified in our two layer finite element model were the orthotropic constants for silicon and a Young's modulus of 300 GPa for SiN<sub>x</sub>. The Young's modulus value for SiN<sub>x</sub> was estimated with an accepted bulk value for Si<sub>3</sub>N<sub>4</sub>. The expansion coefficient of both layers was arbitrarily set to  $\alpha = 2.5 \times 10^{-6} / ^\circ\text{C}$ . We modelled one-quarter of the membrane and incorporated an initial residual stress state of 10 MPa. With the experimental conditions simulated in our model and  $\Delta T = 0$  °C, the deflection computed was 222  $\mu\text{m}$ . A  $\Delta T = 24$  °C was needed to match the 226  $\mu\text{m}$  deflection. Using a single shell element model this temperature change imposed the following in-plane stresses:

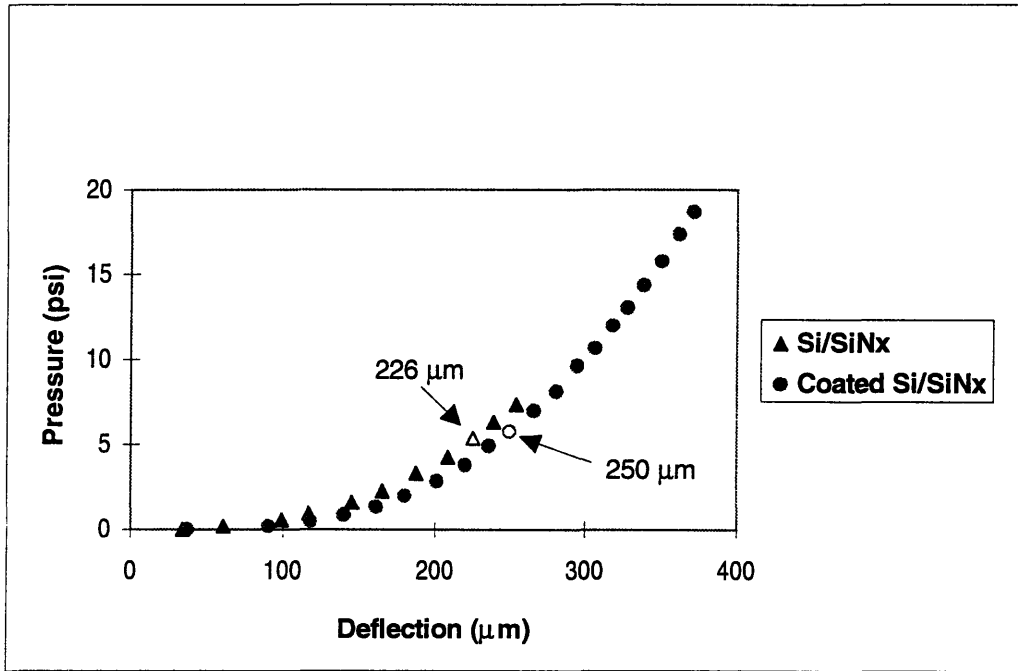
$$\begin{aligned} \text{Si:} \quad & \sigma_{11} = \sigma_{22} = -0.8 \text{ MPa} \\ \text{SiN}_x: \quad & \sigma_{11} = \sigma_{22} = -14 \text{ MPa} \end{aligned}$$

To obtain the composite stress state of the membrane the stresses were weighted with the following expression,

$$\sigma_{average} = \frac{\sum_i \sigma_i t_i}{\sum_i t_i} \quad (6)$$

where  $\sigma_i$  and  $t_i$  refer to the residual stress and thickness, respectively, of the  $i$ th layer of the membrane. The composite residual stress state for this system was calculated to be  $\sigma_{average} = -1.1$  MPa.

To study a system with a more distinguishable compressive residual stress state we sputtered a 200 Å coating on a Si/SiN<sub>x</sub> membrane. The coating increased the compressive residual stress state noticeably. For proprietary reasons the material deposited on the



**Figure 14: Load Deflection Measurements with Compressive Residual Stress States**

membrane cannot be disclosed. Referring to figure 14, the load deflection behavior of the membrane was changed significantly. We implemented the same procedure as before with the point on the curve denoted by the arrow ( $p = 5.752$  psi;  $d = 250$  μm). The material constants for the coating were  $E = 230$  GPa and  $\alpha = 2.5 \times 10^{-6}$ . The half-edge length was 5.114 mm and the thickness was 6.13 μm. The deflection of the simulated membrane with an original residual stress of 10 MPa and a  $\Delta T = 0$  °C was 228 μm. To match the 250 μm deflection a  $\Delta T$  of 141 °C was introduced. This temperature change corresponded to the following residual stress states:

$$\begin{array}{ll} \text{Si:} & \sigma_{11} = \sigma_{22} = -53 \text{ MPa} \\ \text{SiN}_x: & \sigma_{11} = \sigma_{22} = -131 \text{ MPa} \\ \text{Coating:} & \sigma_{11} = \sigma_{22} = -98 \text{ MPa} \end{array}$$

Using equation 6 the composite stress was  $\sigma_{\text{average}} = -55$  MPa. With the residual stress state in the Si/SiN<sub>x</sub> evaluated earlier, the residual stress state in the coating was calculated as  $\sigma_{\text{coating}} = -16$  GPa.

Since the stress states within the individual layers are weighted with equation 6, the accuracy to which the thickness of each layer is measured determines the largest error in the measurement.

## **7.0 Conclusions and Future Directions**

An approach to simulating membranes with compressive residual stress states was proposed and examined. Membranes with compressive residual stress states were simulated using this method and were consistent with predicted behavior. An analysis of the important load deflection parameters for square membranes was undertaken, and the importance of simulations of this type for experimental methods was emphasized. It was concluded that fabrication and equipment costs can be optimized with a knowledge of the materials system of interest and an examination of experimental limitations. In addition we proposed a testing apparatus which would improve the measured membrane behavior, and we emphasized the need to examine anisotropy in single crystal membrane systems when simulating. With the proposed model for examining compressive residual stress states and improved testing methods the residual stress state of Si/SiN<sub>x</sub> with and without an additional compressive coating was evaluated.

In all, the flexibility of load deflection testing and square membranes have not been fully explored. With proper experimental equipment and modelling methods a wide variety of materials phenomena may be investigated.

## 8.0 Bibliography

- [1] Jwo, J. and C. Chung, *Mechanical Characteristics of Aluminum Thin Films on Silicon and Gallium Arsenide*. Thin Solid Films, 1993. 235: p. 149-155.
- [2] Tabata, O., K. Kawahata, S. Sugiyama, and I. Igarashi, *Mechanical Property Measurements of Thin Films Using Load-Deflection of Composite Rectangular Membrane*. IEEE Transactions: 1989. p. 152-156.
- [3] Vinci, R.P. and J.C. Bravman, *Mechanical Testing of Thin Films*, IEEE Transactions: 1991, p. 943-948.
- [4] Beams, J.W., *Mechanical Properties of Thin Films of Gold and Silver*, in *Structure and Properties of Thin Films*, C.A. Neugebauer, J.B. Newkirk, and D.A. Vermilyea, Editor. 1959, Wiley: New York. p. 183-192.
- [5] Small, M.K. and W.D. Nix, *Analysis of the Accuracy of the Bulge Test in Determining the Mechanical Properties of Thin Films*. Journal of Materials Research, 1992. 7(6): p. 1553-1563.
- [6] Allen, M.G., M. Mehregany, R.T. Howe, and S.D. Senturia, *Microfabricated Structures for the in situ Measurement of Residual Stress, Young's Modulus, and Ultimate Strain of Thin Films*. Applied Physics Letters, 1987. 51(4): p. 241-243.
- [7] Pan, J.Y., *A Study of Suspended-Membrane and Acoustic Techniques for the Determination of the Mechanical Properties of Thin Polymer Films*. 1991, Ph.D. Thesis, Massachusetts Institute of Technology.
- [8] Sobek, D., *A Microfabricated Structure for Optical Measurements in Fluids*. 1992, M.S. Thesis, Massachusetts Institute of Technology.
- [9] Lin, P., *The in-situ Measurement of Mechanical Properties of Multi-layer Coatings*. 1990, Ph.D. Thesis, Massachusetts Institute of Technology.
- [10] Allen, M.G. *Measurement of Adhesion and Mechanical Properties of Thin Films Using Microfabricated Structures*. 1989, Ph.D. Thesis, Massachusetts Institute of Technology.
- [11] Allen, M.G. *Measurement of Mechanical Properties and Adhesion of Thin Polyimide Films*. 1986, M.S. Thesis, Massachusetts Institute of Technology.
- [12] Seidel, H., L. Csepregi, A. Heuberger, H. Baumgartel. *Anisotropic Etching of Crystalline Silicon in Alkaline Solutions: Orientation Dependence of Passivation Layers*. Journal of the Electrochemical Society, 1990. 137(11): p. 3612-3626.



- [13] Seidel, H., L. Csepregi, A. Heuberger, and H. Baumgartel. *Anisotropic Etching of Crystalline Silicon in Alkaline Solutions: Influence of Dopants*. Journal of the Electrochemical Society, 1990. 137(11): p. 3626-3632.
- [14] Courtney, T. H., *Mechanical Behavior of Materials*, 1990, McGraw-Hill: New York, NY.
- [15] Baker, S.P., M.K. Small, J.J. Vlassak, B.J. Daniels, and W.D. Nix. *The Search for the Supermodulus Effect in Mechanical Properties of Deformation Behavior of Materials Having Ultra-Fine Microstructures*, M. Nastasi, Editor. 1993, Kluwer Academic Publishers: Netherlands. p. 165-192.
- [16] Chu, W., and Mehregany. *A study of Residual Stress Distribution Through the Thickness of p<sup>+</sup> Silicon Films*. IEEE Transactions on Electron Devices, 1993. 40(7): p. 1245-1250.
- [17] Simmons, G. and H. Wang. *Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook. 2 ed.* 1971, M.I.T. Press: Cambridge, MA.

## List of Figures

Figure 1: Schematic of Deflected Membrane .....	6
Figure 2: Top View of Buckled Membrane .....	8
Figure 3: Calibration Curve of Imposed Stress vs. $\Delta T$ .....	10
Figure 4: 200 Element Mesh of 1/4 Membrane .....	10
Figure 5: Maximum Deflection vs. Residual Stress State .....	11
Figure 6: Pressure - Deflection Plot for a Typical Membrane Sample .....	12
Figure 7: Effect of Residual Stress/Modulus Ratio on Load Deflection .....	13
Figure 8: Effect of Thickness/Half-Edge Length Ratio on Load Deflection .....	14
Figure 9: Effect of Residual Stress/Modulus Ratio on Load Deflection .....	14
Figure 10: Effect of Modulus on Load Deflection for a Given Pressure .....	15
Figure 11: Comparison of Load Deflection Behavior of Identical Membranes .....	18
Figure 12: Schematic of Suggested Mounting Apparatus .....	19
Figure 13: Simulation of Silicon Membrane as Isotropic and Orthotropic Material .....	19
Figure 14: Load Deflection Measurements with Compressive Residual Stress States .....	22