Chapter 3

Pressure Bulge Test for Thin Film Characterization

3.1 Introduction

The mechanical properties of thin films may have large difference in them due to variations in processing conditions. The temperature, humidity, method of etching, or the order of fabrication procedures may induce a great difference in the parameters governing properties. To increase the reliability and control the costs, chip level characterization of mechanical properties such as the modulus is often found to be necessary.

Residual stress is an important parameter that can affect the performance and reliability of thin film devices. This is the stress in the material/device after fabrication and can be either tensile or compressive. It can be beneficial, as in the case of tempered glass, but can often lead to premature failure of the device. In thin films the residual stress can lead to poor performance of the film because of the residual stress state, and in the worst case can lead to the destruction of a micro fabricated device when it is released from the substrate. It is therefore desirable to be able to measure the residual stresses in thin films.

In most measurement techniques, the deformation (deflection/strain) is applied and the stress is related to the strain through the modulus in the stress-free state. As a result, it is

desirable that the method for characterizing the residual stress also include technique to measure the modulus.

There are generally two categories of characterization methods for residual stress and modulus, according to the complexity of the sample preparation: generalized techniques, and device specific methods.

Generalized Techniques

The samples for this kind of methods are thin films with substrates, and usually in large planar geometry. Characterizations can be done without releasing the film and other particular preparations of the samples. These techniques include:

- Diffraction based techniques

- Hole drilling

- Raman spectroscopy

- Optical fluorescence

- Indentation and indentation fracture

- Deflection techniques (wafer level)

Device Specific

The samples of these measurements are more complex in their geometry and usually need device fabrication processes. The techniques of this type include

- Deflection Techniques (device level):

  Bulge test, micro-bending test, and micro-tension test

- Micro rotating structure indicator

Details of the above methods are listed in the Appendix.

Among the above-mentioned methods, the bulge test is most versatile. It has the advantage of being able to characterize the residual stress, elastic modulus, and other important parameters such as yield strength and fracture toughness, while at the same time, the sample preparation is relatively simple compared to the micro rotating structure indicator test, micro-bending and micro-tension test without having the problems at sample clamping or damage as in the micro-tension test. It doesn't involve the substrate effect as the methods in the "generalized techniques" listed above. The comprehensive developments in both the theoretical analysis and experiment techniques make the bulge test a reliable method for mechanical characterization of functional thin film and thin film structures.

The current work takes the above mentioned advantages of the pressure bulge test for thin film characterizations. Single layer free-standing thin films of Si_3N_4 are tested. Multilayered functional PBT/Si_3N_4 thin film structure is also tested.

3.2 Experimental

An experimental setup was fabricated to measure the mechanical response of free standing thin film specimen under pressurized loading. The mechanical property parameters can

then be obtained by analyzing the loading vs. deflection relation. The pressure loading is controlled and recorded during the pressure bulge test. The displacement response is measured using an interferometric method with high resolution.

3.2.1 Pressure bulge test

3.2.1.1 Gas system–source and pressure chamber

The easiest way of setting up a pressure source is a syringe connected to a buffer chamber. By moving the piston to different readings on the syringe tube, one can get variable pressure on the sample surface, during either loading or unloading.

By estimating the total volume change by the movement of the piston of the syringe, the pressure difference ΔP can be calculated:

$$\Delta P = P_2 - P_1 = P_1 V_1 \left(\frac{1}{V_2} - \frac{1}{V_1} \right) = \frac{P_1 V_1}{V_2} - P_1 \approx 5 \, psi \qquad (3.1)$$

A more accurate way of controlling the pressure loading can be obtained by lecture bottle with needle valve, since this provides a gentle source of pressure and precise control of loading and unloading. Considering for a versatile apparatus to be able to fit in a characterization equipment such as an optical microscope or an X-ray diffractometer, the size of the over-all aperture needs to be compact.

A picture of the gas system is shown in figure 3.1. The central component of this setup is a buffer chamber. The pressure in the buffer chamber can be increased by opening a loading valve. Pressurized air is provided by a lecture bottle at 115 atmospheres. Given the small pressures used during the experiments, a regulator is required to reduce this pressure

to approximately 50 kPa. To decrease the pressure, an unloading valve is opened to create a leak. A protection valve can be used to isolate the film from the pressure-handling module, if needed.

The pressure inside the buffer chamber is measured by a 4-20 mA pressure sensor (Omega Engineering, DP25B-E-AI) connected to a meter (Omega Engineering, PX01C-0-005GI). The pressure can either be read on the meter or recorded by an oscilloscope connected to the output of the meter.

Figure 3.1. Pressure-handling module (without the meter).

3.2.1.2 Sample holder and sample handling chamber

A critical point of the setup is the fixation of the sample on its holder. The films to be studied are fixed on silicon substrates, with a square window in the middle. Manipulating a thin film is difficult as it is very fragile. The silicon substrate was therefore glued on a

polymethyl methacrylate (PMMA) holder with a hole in the middle to transmit the pressure (PMMA is a transparent polymer). The PMMA holder can then be sealed on a chamber and removed easily without damaging the film. This system allows the use of strong adhesive bonding between the wafer and the holder, which avoids the clamping problems encountered in previous experiments [1]. A schematic of the assembly is shown in figure 3.2, and a schematic of the fixation of a thin film on its PMMA holder is shown in figure 3.3.
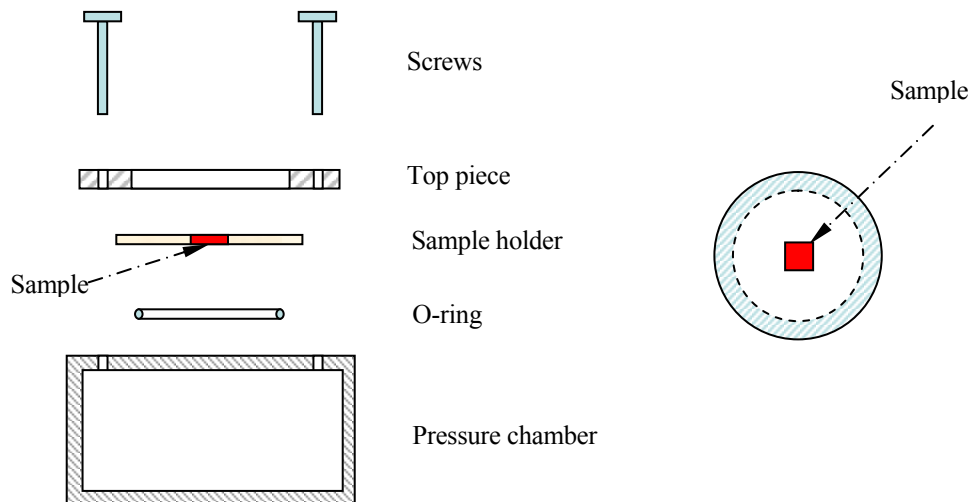

Figure 3.2. Assembly sketch and PMMA sample holder.

Before using strong adhesive (Araldite, model 2011), double-sided tape and silicone rubber were tried, but the strong adhesive was found to perform better. Double-sided adhesive tape can be used, but it requires the operator to push hard on the silicon substrate in order to seal it to the tape without any leak, which sometimes can damage the film. Silicone-rubber is too soft and hence it deforms when put under pressure.
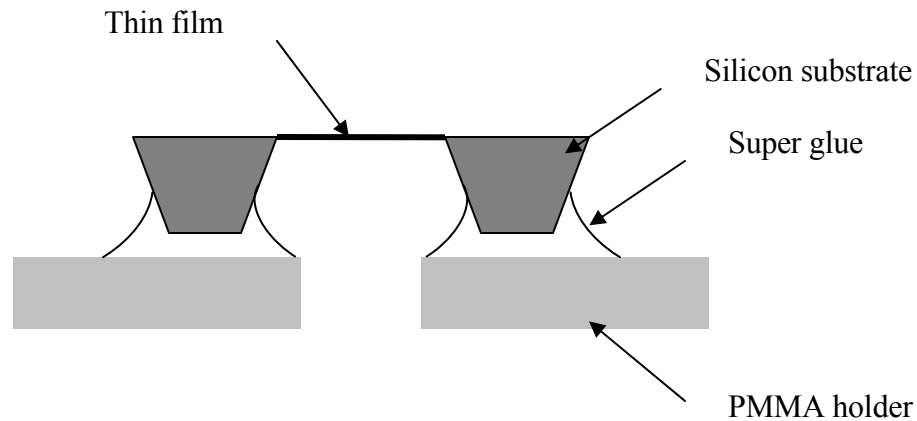
Thin film

Silicon substrate

Super glue

PMMA holder

Figure 3.3. Fixation of a thin film on its PMMA holder.

3.2.2 Real time in-situ full field displacement measurement

A Michelson interferometeric objective (Nikon, Plan 5x) was used to measure the deflection of thin films. The schematic of the measurement system is shown in figure 3.4. The mains elements of the displacement measurement system are a source of visible light at a fixed wavelength, $\lambda=546$ nm, a beam splitter, a reference mirror, a long working distance microscope (Nikon, 1x Tool Makers Objective Lens) and a CCD camera (Sony, XC-75). This setup is similar to the one used by Mitchell et al. [1]. The whole setup is mounted on a pressurized air table to isolate influence from vibrations. The pictures from the CCD camera are recorded on a computer using a frame grabber card (EPIX, PIXCI SV4). The computer is also used to send a signal to the oscilloscope (Nicolet, Model 40, bandwidth 100MHz) which records the pressure.
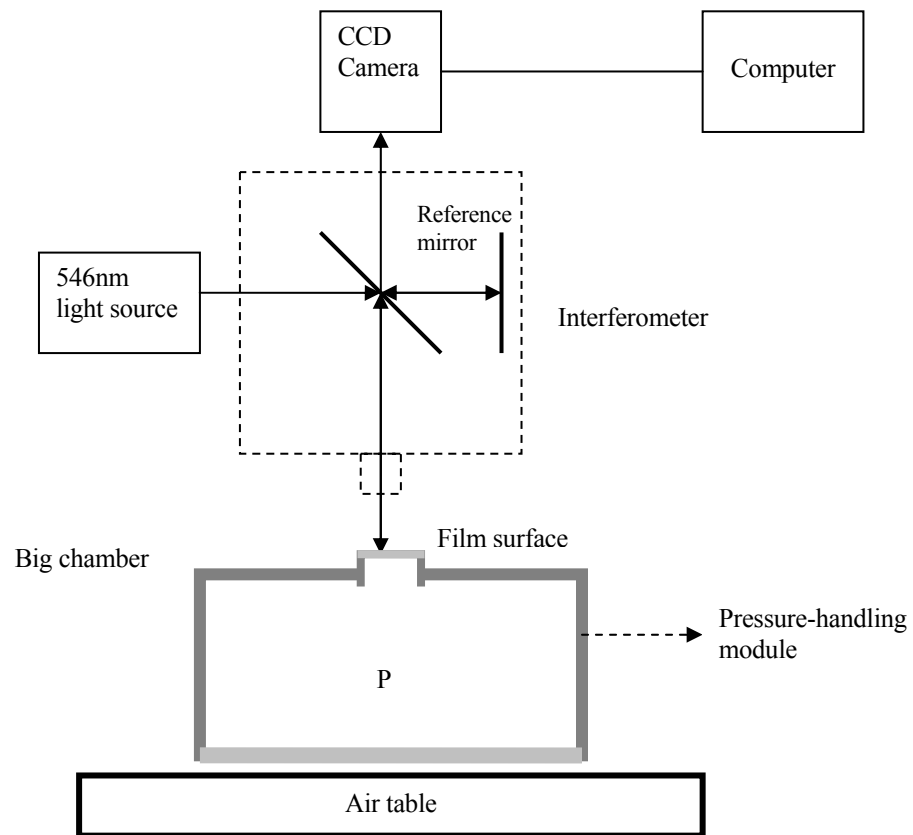
Figure 3.4. Schematic of the interferometric displacement measurement system.

The light beam is split into two components. One of these components reflects off on the surface of the thin film and re-enters the interferometer, where it combines again with the other component which has been reflected off reference mirror. Depending on the path length difference between the two beams, they will interfere either constructively or destructively with each other. The observer would see a series of dark fringes and bright fringes, as in figure 3.5. Each white (or black) fringe corresponds to a displacement of $\lambda/2$ (i.e., 273 nm). The center deflection can be obtained by counting the number of fringes from the center to one corner, and multiplied by $\lambda/2$ (i.e., 273 *nm*). The displacement

profile can be obtained with the same method: counting the number of fringes from the points of interest to the corner and multiplied by $\lambda/2$.
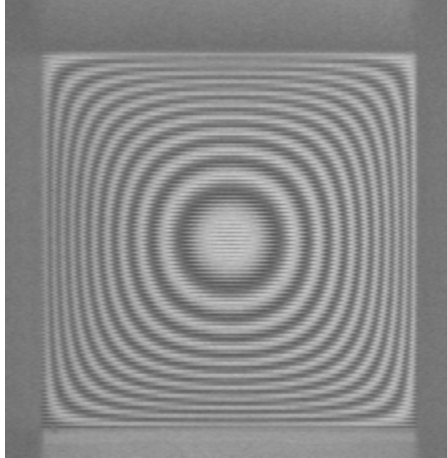

Figure 3.5. Typical interference patterns during the bulging of a thin film.

3.3 Materials

3.3.1 Properties of silicon nitride (Si$_3$N$_4$) measured by other methods

Silicon nitride (Si$_3$N$_4$) is a widely used functional material in MEMS devices due to its superior chemical and mechanical properties. The mechanical characterization of the residual stress and Young's modulus has been carried out for many years. However, due to different fabrication methods and process conditions, both parameters covered a very large range of values, as shown in table 3.1. The film density is believed to be the main parameter influencing the Young's modulus [2]. This large variability makes it difficult to predict *a priori* the behavior of Si$_3$N$_4$ thin films, which justifies the need for further experimental measurements.

Table 3.1. Measured residual stress and Young's modulus for silicon nitride thin films.

Author	Method	Production method	Residual stress σ_0	Young's modulus
Tabata et al., 1989	Bulge test	LPCVD	1GPa	290 GPa
Taylor, 1991	Nano-indentation	PECVD	-1.57 to 1.11 GPa*	178 to 271 GPa *
Vlassak & Nix, 1992	Bulge test	LPCVD	120-150 MPa	222±3 GPa
Levy et al., 1996	Nano-indentation	LPCVD	N/A	130 to 185 GPa*
Buchaillot et al, 1997	AFM and optical beam detection	(Commercially available)	N/A	192 GPa
Zhang et al., 2000	Microbidge test	LPCVD	291 ± 56 MPa	202 ±16 GPa
Lehmann et al., 2001	IR spectroscopy, XPS	Ion-beam sputtering	N/A	237 ±54 GPa
Khan et al., 2003	Scanning force microscopy	LPCVD	N/A	280-290 GPa
Vila et al, 2003	Nano-indentation	Sputtering	N/A	100 to 210 GPa*
Edwards et al., 2004	Bulge test	LPCVD	114-130 GPa	258 GPa
Edwards et al., 2004	Tensile test	LPCVD	N/A	257 GPa

* depending on film production conditions

3.3.2 Si$_3$N$_4$ TEM windows

The thin film that was chosen for characterization is a commercially available silicon-nitride membrane window sold by SPI (model 4123SN-BA, www.2spi.com). It is usually used as a support material for TEM microscopy. The SPI silicon nitride membrane windows are grown by low pressure chemical vapor deposition (LPCVD) at about 700 °C and at reduced pressure. It produces very high quality and stoichiometrically pure silicon nitride. It is highly chemically resistant, fatigue tolerant and creep resistant. A picture of the thin film is shown in figure 3.6.
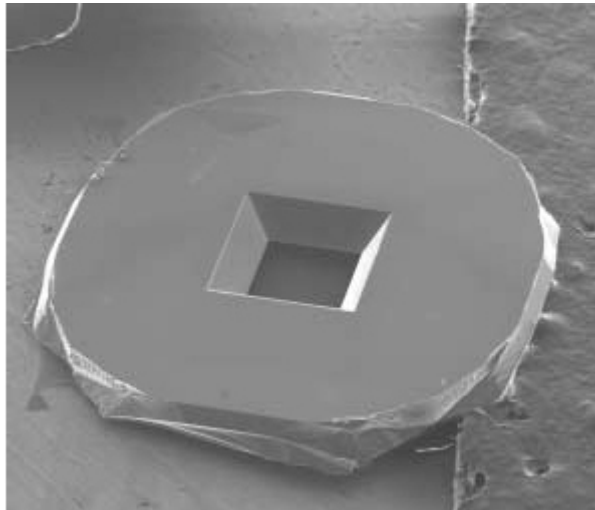

Figure 3.6. The silicon-nitride thin films used are located inside a square silicon window (total dimension diagonally in figure is 3 mm) by www.2spi.com

The characteristics of the thin films used are:

- thickness: 75 nm

- size: 500 μm × 500 μm (square)

The thin films are located inside a square window in a silicon substrate (see figure 3.6). The characteristics of the substrate are:

- thickness: 200 μm

- window size: 500 μm × 500 μm

- substrate size: 2 mm × 2 mm

3.4 Procedure

During each pressure-bulge experiment, real-time pressure loading signal was recorded by a digital oscilloscope (Nikolet, model 40, bandwidth 100 MHz), and full field interferometric patterns acquired by a CCD were recorded by computer via digital frame grabber. Each test followed the procedure listed below.

- Close the loading valve and the protection valve

- Open the unloading valve and record the output from the pressure meter at 0 pressure

- Fix the sample handling chamber on the stage under the interferometer

- Adjust the microscopic interferometer to get clear fringes

- Turn on the air-cushion to prevent the vibration

- Open the gas source

- Open the data capture software and set frame capture parameters

- Close the unloading valve

- Open the sample protection valve

- Start the external trigger for data capture software and at the same time open the loading valve gradually to the desired pressure then close the loading valve

- Save the data

- Open the software and setup parameters for unloading process

- Click the external trigger and at the same time open the unloading valve slowly and adjust the valve to have constant rate in dropping of pressure until ambient pressure

- Close the sample protection valve

- Save the data

The maximum number of frames that can be recorded by the frame grabber software is 189. It was hence parameterized to record one frame per second, during three minutes. This time was chosen because it represented a good compromise between two aspects that are described below.

On the one hand, the experiment should not last too long because the offset of the pressure sensor increases due to heating, introducing some error. For a three minutes experiment, the error coming from this change in the offset is less than 0.1%. Moreover, a

long experiment increases the risk of creating an undesired air leak. On the other hand, the experiment should not be too fast in order to be approximated by quasi-static conditions. If the pressure increases too fast, it is also difficult for the operator to control the pressure rate. Moreover, a pressure difference can appear between the film-handling chamber and the buffer-chamber, due to the viscosity of the air in the pipes. This pressure difference was calculated to be less than 1 Pa in our experiments, which was negligible compared to the pressures used in the experiment.

3.5 Model

3.5.1 Characterization of mechanical properties

The material properties can be obtained from analyzing the load deflection relation in a pressure bulge experiment. It's a classical mechanics problem to solve this relation for a thin plate under uniform pressure. The analytical solution for the problem of a circular plate under uniform pressure loading can be traced back to more than 100 years ago. However, considering the residual stress effect and the square or rectangular shape thin plates under large deformation is a much more challenging work than the stress-free or circular plate case. This type of analytical work was carried out only more recently.

A least-square fitting between the pressure (p)-displacement (d) curves with the analytical formula

$$P(d) = C_1 \frac{t\sigma_0}{a^2} d + C_2(\upsilon) \frac{tE}{a^4} d^3$$

(3.2)

gives the estimated values for the Young's modulus E and the residual stress σ_0 for a square film of side, a and thickness, t, where d is the maximum displacement at the center of the plate, and υ is the Poisson's ratio of the material.

At small deflection, the linear term is dominant as the residual stress makes significant influence on the thin film behavior. However, at large deflection, the cubic term dominant and Young's modulus control the mechanical behavior.

The general form of the formula (3.2) has been confirmed by many authors, with some variation of the numerical values of C_1 and $C_2(\upsilon)$ to better fit with the experiments. Finite elements analysis [3] and a new analytical solution proposed by Maier-Schneider, Mailbach and Obermeier [4] lead to a similar expression, with modified values for C_1 and $C_2(\upsilon)$. Table 3.2 shows the parameters C_1 and $C_2(\upsilon)$ for thin square films.

Table 3.2. Parameters C_1 and $C_2(\upsilon)$ for thin square films.

	C_1	$C_2 (\upsilon)$	$C_2(0.25)$
Allen et al (1987)	3.04	$1.473 (1\text{-}0.272\ \upsilon)/(1\text{-}\upsilon)$	1.37
Tabata et al. (1989)	3.04	$1.473 (1\text{-}0.272\ \upsilon)/(1\text{-}\upsilon)$	1.37
Pan et al. (1990)	3.41	$1.37(1.075\text{-}0.292\ \upsilon)/(1\text{-}\upsilon)$	1.83
Vlassak & Nix (1992)	3.393	$(0.8+0.062\ \upsilon)^3/(1\text{-}\upsilon)$	1.84
Maier-Schneider et al. (1995)	3.45	$1.994(1\text{-}0.271\ \upsilon)/(1\text{-}\upsilon)$	1.86

The agreement between the shape of the membrane predicted by the analytical solution and the shape measured experimentally validates the analysis. Moreover, Xu et al. showed

that using the previous analytical solution, bulge test led to values for Young's modulus and residual stress in close agreement with the values from nano-indentation and substrate bending methods [5].

Finally, Mitchell et al. have conducted a detailed error analysis of the bulge test [1]. The method was shown to be highly sensitive to the film's geometry and to the accurate measurement of the deflection. An uncertainty of 6.5 to 8.5% was calculated for the residual stress, and 7 to 9% for Young's modulus, using a setup similar to the one used here.

3.5.2 Full field displacement analysis

A full field fringe pattern was analyzed in order to draw the diagonal profile of a deformed thin film. Along the diagonal, the distance between a corner and each fringe was measured (figure 3.7).
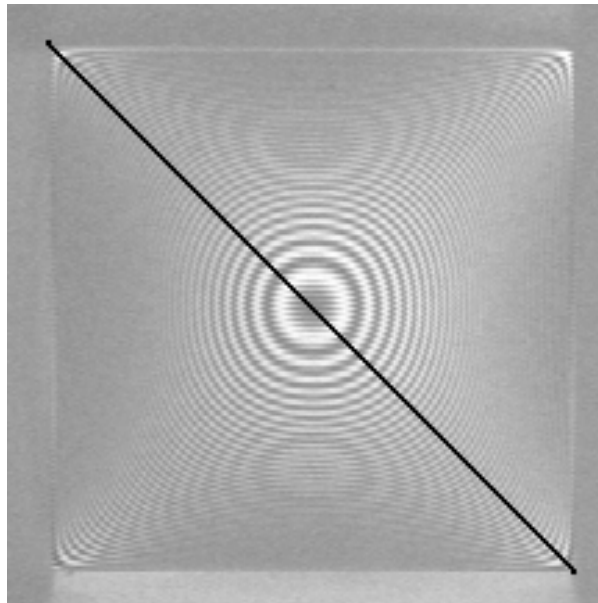

Figure 3.7. Image used to draw the diagonal profile.

The displacement profile was normalized with the maximum deflection at the center of the film in order to be compared with the analytical formula provided by Maier-Schneider et al. [4]:

$$W(x=y)=W_0(1+0.401\frac{2x^2}{a^2}+1.1611\frac{x^4}{a^4})\cos^2\frac{\pi x}{2a} \quad . \tag{3.3}$$

The results are shown in figure 3.8. There is a very close agreement between this theory used and the result from current experiment. The correct prediction of the film shape validates the theory, and in particular equation 3.3.
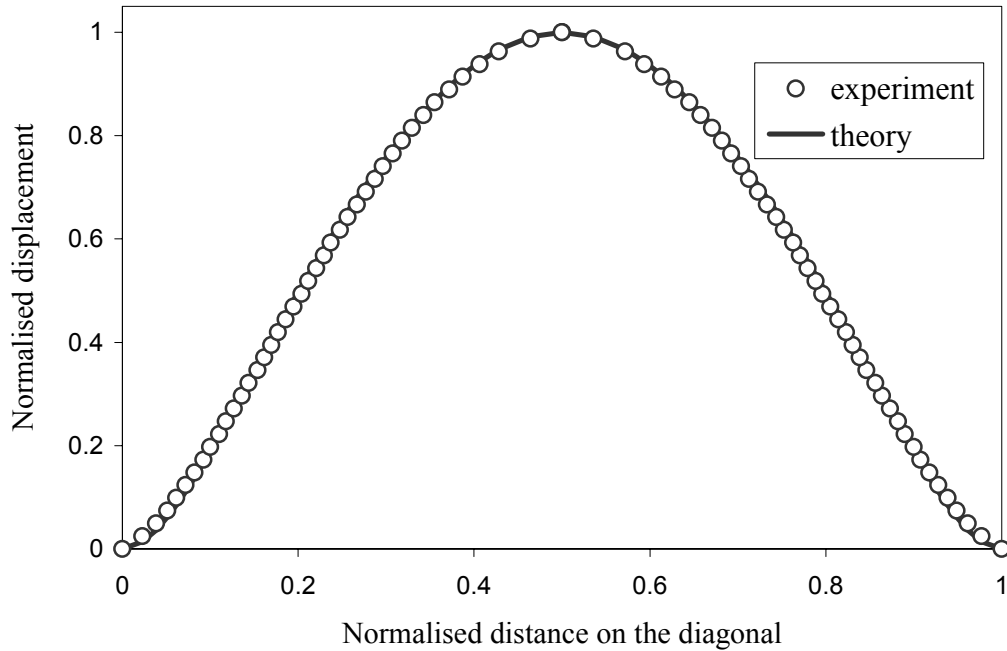

Figure 3.8. Comparison between the experimental diagonal profile and the profile predicted from the analytical solution.

3.6 Analysis of Si₃N₄ free standing thin films

3.6.1 Pressure-displacement curve

The pressure-displacement curves obtained for loading and unloading of a silicon nitride membrane (geometrical parameters) are shown in figure 3.9. The loading curve and the unloading curve are very similar, with very little hysteresis between the two.
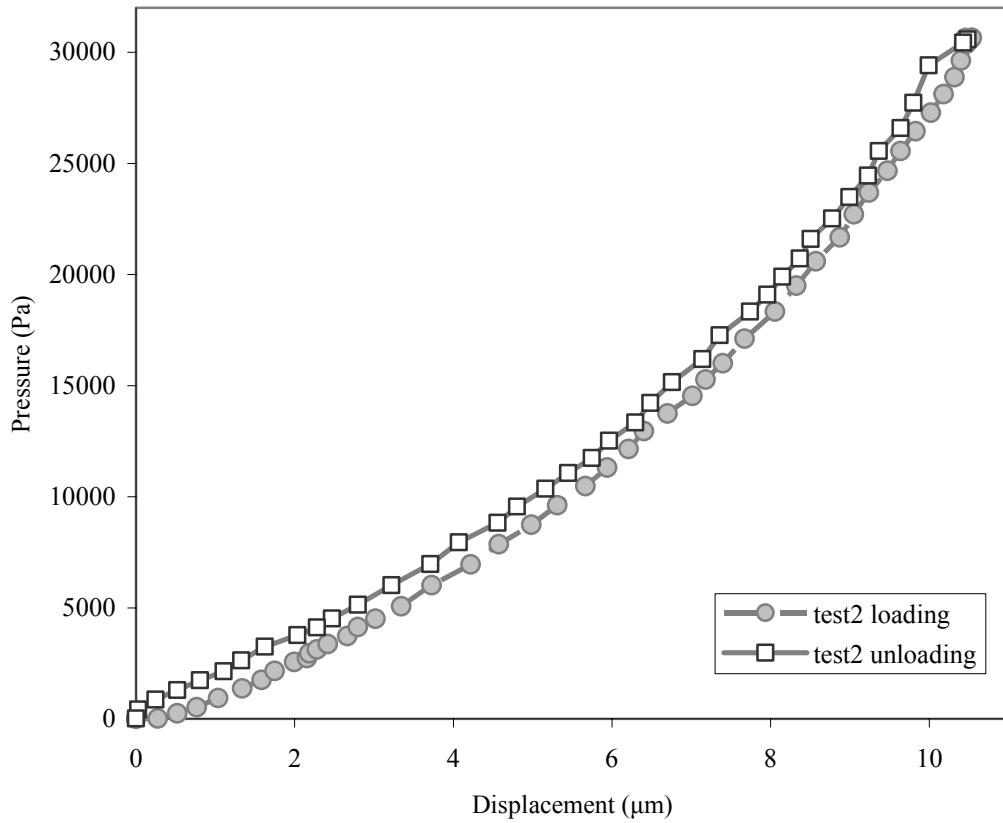

Figure 3.9. Pressure-displacement curve of a silicon-nitride thin film.
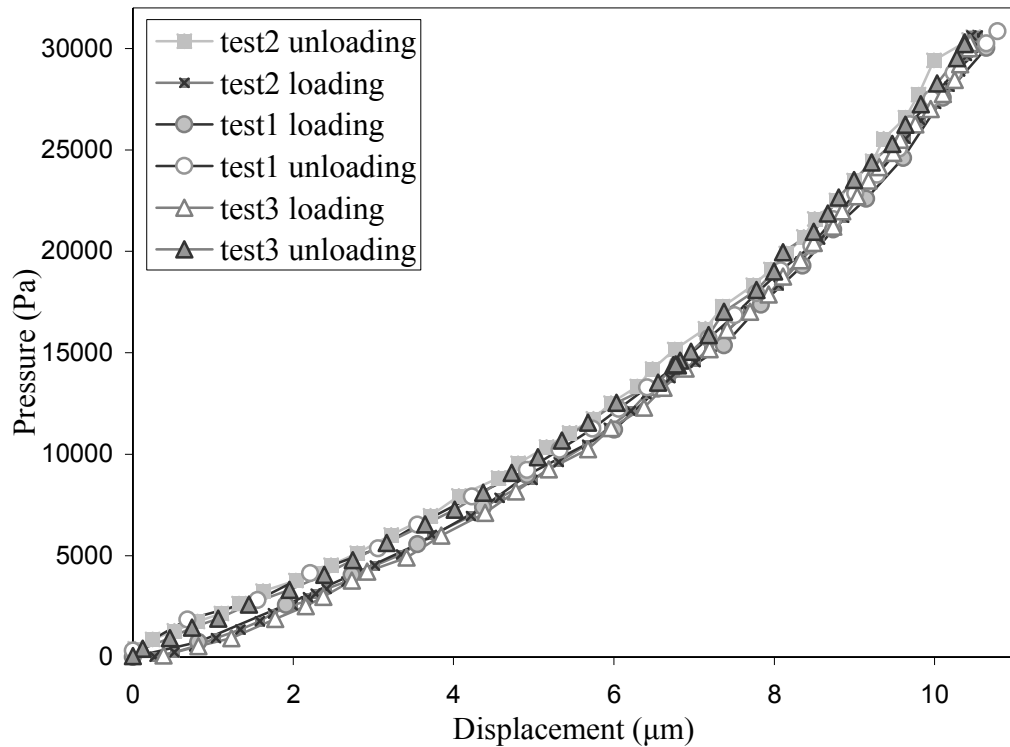
3.6.2 Repeatability of the experiment

Figure 3.10. Repeatability of the test on a single silicon-nitride thin film.

The same film was tested three times in order to evaluate the repeatability of the results and to check the accuracy of the method. The results obtained for one of the films are shown in figure 3.10. The three sets of curves are very similar. The loading curve is always below the unloading curve. This gap comes mainly from a delay between the pressure sensor and the video capture software, which can be corrected. The three loading curves and three unloading curves are closely repeatable within each group. This indicates the reliability of the current characterization method. The good repeatability also indicates that

there is no permanent displacement after each loading. Therefore, the material presents linear elastic behavior throughout all these tests.

3.6.3 Determination of Young's modulus and residual stress

In order to obtain the material parameters (modulus, E and residual stress, σ_0), a value has to be assumed for Poisson's ratio, ν. The range of values measured for silicon-nitride in the literature ranges from 0.22 to 0.25. The value most commonly assumed is 0.25 [1] and this value is used throughout for comparison purposes.
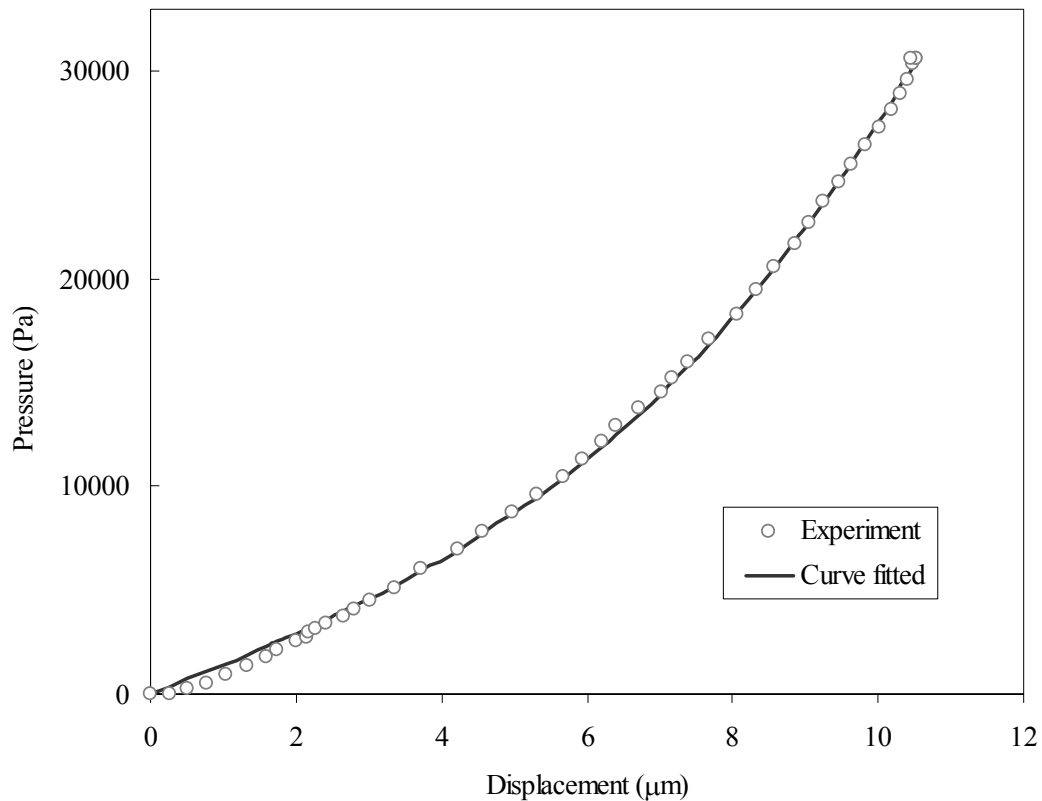

Figure 3.11. Fitting of a curve, where $E = 262$ GPa, $\sigma_0 = 248$ MPa, $R^2 = 0.99945$).

A least-squares fitting of the pressure-displacement curves with the analytical formula (3.2) gives the estimated values for the Young's modulus E and the residual stress σ_0 (figure 3.11). The values found for the two films tested are given in the following tables.

Table 3.3. Young's modulus measured on film 1.

E (GPa)	Loading	Unloading	Average
Test 1	257	227	242
Test 2	262	248	255
Test 3	291	252	271.5

Table 3.4. Residual stress measured on film 1.

σ_0 (MPa)	Loading	Unloading	Average
Test 1	351	397	374
Test 2	369	392	380.5
Test 3	332	391	361.5

Table 3.5. Young's modulus measured on film 2.

E (GPa)	Loading	Unloading	Average
Test 1	337	278	307.5
Test 2	358	273	315.5

Table 3.6. Residual stress measured on film 2.

Σ_0 (MPa)	Loading	Unloading	Average
Test 1	455	539	497
Test 2	414	529	471.5

The residual stresses found are in the range expected from the literature (table 3.1). The Young's modulus found for the first film is in the range expected, from 100 GPa to 300 GPa. The Young's modulus found for the second film is slightly higher than expected.

3.7 Characterization of PBT thin film

3.7.1 Material

The PBT thin film to be characterized was grown by the Ferroelectric MURI group at Caltech (www.femuri.caltech.edu), by metal organic chemical vapor deposition (MOCVD) [6, 7]. PBT stands for $(Pb_xBa_{1-x})TiO_3$. It is a solid solution of lead titanate and barium titnate. It has similar ferroelectric behavior of lead titanate and barium titnate, and can perform 90° domain switching under external stress state or electric field. This perovskite structure was chosen for its tetragonal geometry at usual temperatures and the linear variation of its strain (c/a) with its composition. Therefore, the expected strain would be in the range of 1.1% to 6.5%.

The PBT thin film is composed of three layers:

- PBT layer (thickness: 190 nm)

- IBAD MgO layer (thickness: 20 to 50 nm)

- Si_3N_4 layer (thickness: 75 nm)

In most of the ferroelectric thin films, the ferroelectric layer is usually grown on a thick substrate. The mechanical behavior of the film is therefore dominated by the behavior of the substrate, and the interface between the substrate and the ferroelectric layer imposes

geometrical constraint to the latter. In contrast, the PBT thin film investigated here is relatively free-standing, since it is dominated by the ferroelectric layer.

3.7.2 Analysis

Multi-layered thin film structure of PBT/MgO/Si$_3$N$_4$ was characterized by pressure bulge test. The maximum pressure used for the experiments was 13.2 kPa. Indeed, this pressure is enough to induce an in-plane stress that is expected to be magnitudes larger than the domain-switching threshold (1.1 MPa).

The pressure-displacement curves obtained are given in figure 3.12. The curves are nearly linear. Indeed, for low pressures, the linear term in eq. (3.2) is proportional to the residual stress and is dominant over the cubic term which is proportional to the Young's modulus. As a consequence, the influence of the Young's modulus on the pressure-displacement curve is expected to be small. The total thickness of the film is assumed to be 300 nm. The actual thickness is between 285 nm and 315 nm. Residual stress measured using the linear part of the curves is in the range 221–255 MPa (table 3.7) and the contribution due to the cubic part is small compared to the experimental noise. As a consequence, the Young's modulus can not be determined accurately. The loading and the unloading curves lead to an order of magnitude difference in the Young's modulus. Hence, experiments with higher pressure are needed to measure the effective modulus of the layered PBT films.
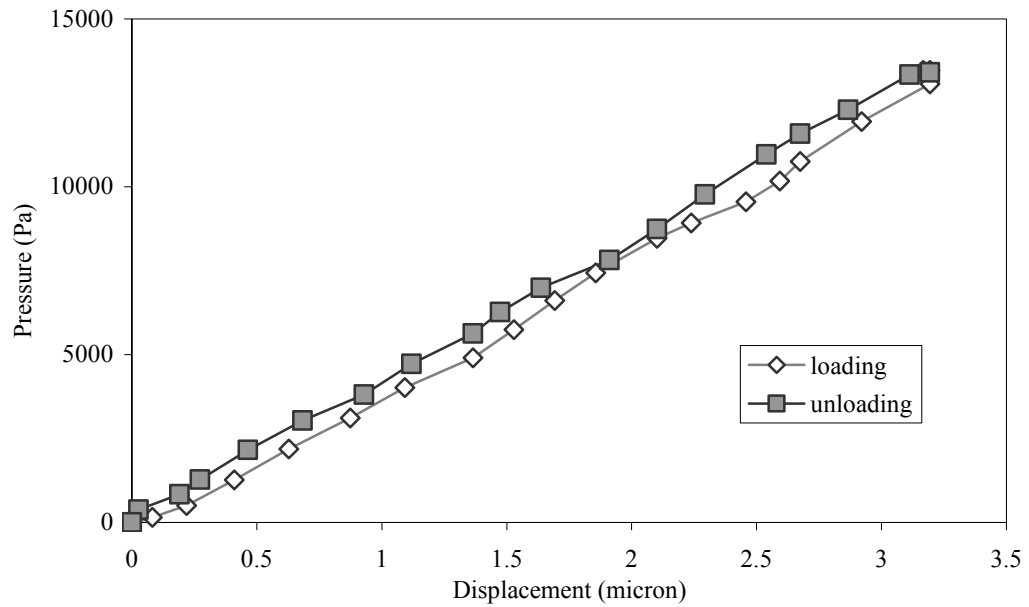
Figure 3.12. Bulge test result of the PBT thin film.

Table 3.7. Residual stress and Young's modulus measured on the PBT thin film (for *E*, *υ* = 0.25 was assumed).

	σ_0 (MPa)	E (GPa)
Loading	221	267
Unloading	255	28.5

3.8 Discussion and conclusion

3.8.1 Error analysis

3.8.1.1 Systematic error due to the assumption on Poisson's ratio

Systematic errors are errors that are identical in all experiments. They will therefore result in a biased average result. The theory used and imperfections in the experimental setup are the main factors that can result in a systematic error.

The Young's modulus measured is affected by the choice of Poisson's ratio, through the variation of the coefficient $C_2(v)$ in eq. (3.1). For silicon-nitride thin films, the range of values that Poisson's ratio can take is 0.22–0.25 and the corresponding error for E is 5%.

3.8.1.2 Random error

Random errors are the non-repeatable experimental errors that result in differences between two identical experiments. This source of uncertainty can be reduced by averaging the results over a large number of experiments.

The values given by the loading and the unloading curves for the silicon-nitride films are averaged for each experiment. The difference between different experiments made on the same film then gives an estimation of the random error (table 3.8).

Table 3.8. Evaluation of the bulge test random error.

Film	$E \pm$ random error	Random error (%)	$\sigma_0 \pm$ random error	Random error (%)
1	256 ± 15.5	6 %	372 ± 12	3.2 %
2	311 ± 4	1.3 %	484 ± 13	2.7%

The values measured by the bulge test are therefore very repeatable. There is slightly more uncertainty in the value of E ($\sim$ 6%) than in the value of σ_0 ($\sim$ 3%). Using a higher pressure would probably reduce the uncertainty on E. A more rigorous evaluation of the random errors would require repeating the experiments large number of times.

3.8.2 Limitations of bulge test

The residual stress can be either in tension or in compression. The compressive stress will cause ripples on the edges of the released thin films. These ripples change the mechanical behavior of the whole structure and in this case, the bulge test result can not be used to quantify the residual stress and the modulus correctly. For square or rectangular shape thin films, the four corners will have stress concentration during loading, which could result in yielding (in the case of ductile films) or damage (in the case of brittle films) and could affect the overall applicability of the bulge test.

3.9 Conclusions

For the functional thin films for MEMS devices, the mechanical properties are highly dependent on the processing conditions. On-site, chip level measurements are required for the evaluating the reliability of the devices. Among the available methods, bulge test is versatile and poses no problem for sample holding, edge damage or substrate effect. It has the advantage of being able to characterize the residual stress and the elastic modulus at the same time. The comprehensive developments in both the theoretical analysis and experiment techniques make bulge test a reliable method for mechanical characterization for functional thin film and thin film structures. Free standing silicon-nitride thin films are tested and the results presented above focus on the square membrane case. The residual

stress and elastic modulus for thin films of amorphous silicon nitride are presented and associated errors are analyzed. Limitations of the bulge test are discussed. Preliminary results of PBT/Si_3N_4 thin film structure are also reported.

3.10 References

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3.11 Appendix: techniques for mechanical characterization of functional thin films

The size scale over which residual stresses act are closely linked to the microstructure of the sample and also to the measurement method. For instance individual grains in a film might be in a stressed state, i.e. non-equilibrium lattice spacing, however if these individual grains are randomly oriented then the overall stress state could be zero. In this case

measurement techniques that probe at the grain size level would detect a residual stress, whereas broader sampling methods would not. Therefore, residual stresses can be categorized as macro, for stresses at scales larger than the grain size, and micro, for scales at or smaller than the grain size.

3.11.1 Diffraction techniques

Diffraction techniques are a common tool for residual stress measurements. A number of different diffraction probe beams exist, ranging from electrons, low energy X-ray sources, synchrotron X ray sources, to high energy neutron sources. The basis of all these methods is the same, in that the technique measures the lattice spacing of the sample, and based on the deviation from the "stress free" lattice spacing the residual stress can be determined. The simplest and commonly available method is X-ray diffraction. The sampled area is usually of the order of 1 cm^2, and depending on the X-ray energy and the geometry of the system the beam can sample different depths to give the stress state through the thickness. The higher energy techniques such as synchrotron and neutron are popular for bulk measurements because of their higher penetration depths, and greater resolution, however the increased penetration depth makes them unsuitable for thin film characterization.

Electron back scattered diffraction (EBSD) uses the electron beam in a scanning electron microscope to measure the lattice parameter of volumes as small as the electron beam itself, and so can determine the lattice parameters of individual grains. It is most often used to determine texture of samples, but can also be used to characterize residual stress on a

small scale. The advantage of this is that it can be used not only on large area films, but also features in patterned devices.

3.11.2 Deflection techniques

The change in shape brought about by the coating of a sample or device is the basis for many residual stress measurements performed in the semiconductor industry. By careful measurements of the change radius of large silicon wafers the residual stress can be determined by the Stoney formula given by

$$\sigma = \frac{Eh^2}{(1-v)6Rt},$$

where R is the radius of curvature, E is the modulus of the substrate, h is the film thickness, t is the substrate thickness, and n the Poisons ratio of the substrate. The measurement analysis is based on the film being much thinner than the substrate, the film is uniformly stressed, and the bending of the disc does not introduce an extensional strain. The elastic modulus for the coating does not appear in the equation, so for this method these constants are not needed, which could be an advantage when new compositions are examined where these are not known *a priori*.

There are many variations on this type of tests. In general it is used to determine growth of thin films by the sequential addition of coating layers. Many of the commercial test systems include an oven so that samples can be thermally treated to investigate the effect of these thermal treatments on the residual stress. The converse of this technique can also be used, that is where a coated sample has the substrate removed chemically and the change in

shape is again a measure of the residual stress. This method is popular because it is nondestructive.

3.11.3 Hole drilling

Hole drilling is a simple 'bench top' portable technique for residual stress measurement. By the controlled drilling of a hole in a test sample there is a redistribution of stresses brought about by the removal of this material. The strains induced are measured, usually with a specially constructed strain gauge, and there is a simple formula that relates the strains measured by the strain gauge rosette to the residual stress. These calculations are relatively simple for an isotropic uniform material, however for the case of thin film measurements there are many unknowns, including the thickness of the film. The technique is destructive, and scaling it down for thin functional films may be difficult.

3.11.4 Indentation techniques

Indentation techniques using depth sensing hardness measurement systems can be used to measure the elastic modulus and residual stress of thin films. Depth sensing hardness systems essentially measure the stiffness of the sample under an indenter, and based on knowledge of the indenter shape the stiffness of the sample can be derived. For homogeneous samples the analysis is reasonably well developed, but for thin films, both of which could be anisotropic, there are many approximations made to enable the determination of the film modulus. Residual stresses in thin films can be determined by introducing cracks into the film with the indentor. Based on the measurement of the crack lengths, and the indentation load, the residual stress can be determined through the fracture mechanics. There are several similar methods that differ in the treatment of the fracture

mechanics. In its simplest form cracks are induced on the brittle uncoated substrate by indentation, and the difference in crack lengths on a similar but coated sample is an indication of the stress brought about by the coating. This method obviously only works for brittle substrates and thin transparent coatings. One advantage of this technique is that it is a localized measurement, so it can be used on almost any feature in a MEMS device, providing the area sampled is well supported so that the indent can be made.

3.11.5 Raman spectroscopy

Raman spectroscopy, sometimes known as piezospectroscopy, makes use of the Raman effect to measure vibrational energy levels in matter. Raman scattering is a result of inelastic collisions between photons and molecules, and the energy level changes in the scattered light corresponds to vibrational transitions within the scattering medium. The Raman spectrum produced is characteristic of the constituent atoms, the spatial arrangement and bond strength. The stress state of the sample is also seen in the spectrum, thus it can be used to determine residual stress. The Raman probe is usually an Ar+ laser, and when this is coupled with a microscope area as small as 1 micrometer can be investigated. Compared to diffraction the technique is new and so interpretation and quantification are not as advanced, however since many of the materials used in the semiconductor industry do exhibit Raman scattering it is becoming a popular technique to investigate these material systems. The technique of optical fluorescence is similar to Raman spectroscopy, however here the shift in the luminescence lines can be linked to changes in the applied stress. The use of luminescence lines obviously further limits the range of materials. However it has been used to successfully map the residual compressive stress in alumina films grown on NiAl.

3.11.6 Bulge test

The bulge test is really a specialized form of deflection technique, where the substrate is removed to leave a thin membrane of the film, surrounded by the film still supported on the substrate. Although the manufacture of this device is obviously more involved than simply producing a film on substrate, the device is well understood, and is the basis for many pressure sensor devices. In its simplest form the bow of the film brought about by the removal of the substrate can be related to the residual stress. This method is essentially a miniaturization of the layer removal method where the bow of free standing film is measured to determine the residual test. However if the membrane is pressurized differentially and the bow is measured using interferometric techniques, a stress strain curve can be obtained, the slope of which gives the modulus of the film, and the intercept gives the initial residual stress.

3.11.7 Micro rotating structure indicator

This is one of several similar tests that use the deformations brought about when a stressed film is released to advantage. When during device manufacture a stressed film is released by removal of sub layers this brings about large strains, which can impair the device performance. However by careful design, a device can be constructed which not only turns this strain into a rotational motion but with suitable markers on the substrate and amplification of the strain by a lever, it can also point to the level of stress in the system. The disadvantage of this method is that the complexity of producing the test structure, which may be even more complex than the final device.