Experimental Quantification of the Residual Stress of Thin Membrane Materials in MEMS Devices Using Point Deflection

Sofiane Soulimane, Arnaud Pouydebasque*, Sébastien Bolis, Fabrice Jacquet, Claudine Bridoux, Florian Dupont, Christophe Poulain, Stéphane Moreau and Stéphane Fanget

CEA, LETI, MINATEC Campus, F-38054 Grenoble, France

Abstract

In this study, a novel methodology to assess the stiffness and evaluate the residual stress of thin, flexible polymer membranes is presented. This methodology is based on original test structures where the membrane is supported by a liquid in a circular cavity and uses stiffness measurement by point deflection with a nano-indenter. Knowing the Young’s modulus and Poisson’s ratio of the material and using the geometrical dimensions of the membrane (thickness and radius) the residual stress can be extracted from the stiffness values using an analytical model. Several polymers with different Young’s modulus, chemical composition, and thickness were evaluated using this method. Values of residual stress were extracted that are comparable to those obtained by the well-established Stoney method when this was applicable (i.e. relatively large residual stress) with a much lower uncertainty. Moreover, very low values of residual stress were measured with this method that could not be estimated reliably using the Stoney method.

Key Words: Polymer Membrane, Residual Stress, Nano-Indenter, Membrane Flexion, Stiffness

1. Introduction

There is a growing interest in the development of flexible polymer membrane materials for actuators and sensors applications. Polymer materials have been proposed to be good candidates for the development of new micro electro-mechanical systems (MEMS) devices featuring a very flexible membrane (i.e. micro-pumps, speaklets or variable focus lenses, etc. [1–3]). Due to their wide range of mechanical properties, they can be used as a flexible support with very large deflection amplitudes. Key parameters to optimize the deflection amplitude are the membrane thickness, the material Young’s modulus and residual stress. However, these parameters are not always known (especially the membrane residual stress) and it is not straightforward to predict accurately the best design for optimum device actuation. The purpose of this work is to establish a method to study the relationship between the materials mechanical and physical properties (elastic modulus, residual stress and thickness) and the actuation capacity. Generally, the polymer choice is driven by theoretical and practical concerns as well as its technological applications such as the range of material properties is usually limited.

Membrane materials are generally subjected to multiaxial loading during service applications. The behavior of a movable membrane depends on its mechanical properties and in particular on the material residual stress. Residual stress knowledge and control are mandatory for the development of technologies implying thin film membranes for microsystems applications. An example of technology requiring a polymeric membrane with large actuation range is the variable focus lens [3,4]. The membrane displacement is obtained by balancing the ac-
tuation force (electrostatic or piezoelectric for instance) with the mechanical restoring force that is mainly determined by the membrane Young’s modulus, residual stress, and thickness.

As underlined in [4] using multi-physics finite element method (FEM) simulations, the deflection of the membrane of a variable focus lens is dramatically reduced when a tensile residual stress is introduced in the membrane. A good knowledge and control of the residual stress is thus mandatory for the design and optimization of such a device.

2. Methods and Device Fabrication

2.1 Measurement Method

Usually, the residual stress is estimated by the Stoney method [5,6] by measuring the bow of a wafer before and after film process. However, this method does not take into account the effects of the subsequent process steps and the method becomes inaccurate to evaluate low stress levels (typically below 10 MPa) for thin membranes because of the very small difference in the wafer bow before and after membrane formation. In this contribution, we propose a method based on the point deflection method applied to a layer suspended on liquid using a nano-indenter.

A nano-indenter XP tool from MTS was used. The principle of the measurement consists in applying a load with a co-spherical tip (5 μm of radius) perpendicularly to the analyzed sample surface as shown in Figure 1. This type of measurement allows to access to the mechanical properties of the suspended layer such as the Young’s modulus [7–9].

2.2 Fabrication Process

The fabrication process of the test devices used in this work is described in Figure 2. Different polymeric materials were investigated: three siloxane derivatives with different thickness, Young’s modulus and stress, and parylene. Table 1 summarizes their main properties. Polymer membrane materials were spin-coated (siloxane derivatives) or vapor-phase deposited (parylene) on a silicon wafer (Figure 2(a)).

Circular, 40 μm high, cavities were then defined using a photosensitive adhesive polymer (defined as the resist wall in Figure 2(b)). A liquid was dispensed in the cavities and the silicon wafer was then assembled to a glass wafer by polymer bonding (Figure 2(c)). Finally, the silicon wafer was thinned down and the remaining silicon was removed by conventional deep reaction ion etching (DRIE) in the cavities areas (Figure 2(d)).

The presence of liquid below the membrane permits to achieve the release of very thin and flexible membranes without suffering from stiction at the bottom of the cavity or fracture that could have occurred otherwise.

In order to assess the validity of the method, devices having a mineral polycrystalline silicon membrane (4 μm thick) with different radii (from 450 to 2550 μm) were also fabricated. In this case, due to the rigidity of the membrane, the presence of liquid in the device was not necessary and the devices were obtained by a simple DRIE of the Si wafer in order to release the membrane.

![Figure 1. Schematic representation of point deflection method applied to a membrane released on liquid.](image1)

![Figure 2. Summary of the fabrication process flow of the tested devices.](image2)
2.3 Analytical Model

The method based on the point deflection applied to a suspended layer on liquid allows to access to the stiffness of the self-standing structure. The stiffness $S$ is expressed by the following relationship:

$$ S = \frac{F}{h} $$

where $F$ is the applied force and $h$ is the bending distance (out-of-plane displacement). $S$ is determined by the slope of the load vs. displacement curve at the contact of the indenter tip with the membrane, characterized by a positive slope change. Figure 3 shows a characteristic load-displacement curve measured with the nano-indenter on the structures defined previously. During the initial stage (between 520000 and 525000 nm) the nano-indenter head is in an approach mode where it is trying to detect the sample surface. In this regime, the load values reported by the nano-indenter are not meaningful. However, once the surface is detected, the variation of the load with the displacement changes and one finds the expected increase of the load (from -1500 µN to 0 µN) when the displacement increases (between 530000 nm and 560000 nm). The stiffness was calculated from the initial part of the curve (between 530000 nm and 535000 nm) to limit the non-linear effects related to large deformations.

In the case of a concentrated transverse load applied to the center of a circular membrane in the small deformation regime and for residual tensile stress in the membrane, the stiffness $S$ can be expressed as [10]:

$$ S = \frac{\sigma \cdot t}{8 \cdot \alpha \cdot \beta^2 \cdot g'(k)} $$

where $\sigma$ is the membrane residual stress, $t$ is the membrane thickness, and $\alpha$ and $\beta$ are geometric constants. In the case of a circular membrane $\alpha = 0.00497$ and $\beta = 2$ [6]. The function $g'(k)$ is related to the stress level and is defined as [10,11]

$$ g'(k) = \left[ \frac{K_i(k) - \frac{1}{K_i(k)} \cdot (I_0(k) + 1) + K_0(k) + \ln \left( \frac{k}{2} + \gamma \right)}{I_0(k)} \right] $$

and

$$ k^2 = \frac{12 \cdot (1 - \nu^2)}{\beta^2 \left( \frac{a}{l} \right)} \left( \frac{a}{l} \right)^2 \frac{\sigma}{E} $$

where $I_0$ ($I_1$) is the modified Bessel function of the first kind of order 0 (order 1), $K_0$ ($K_1$) is the modified Bessel function of the second kind of order 0 (order 1) and $\gamma$ is the Euler’s constant. $E$ and $\nu$ are the membrane material Young’s modulus and Poisson’s ratio and $a$ is the membrane radius.

Knowing the membrane Young’s modulus and Poisson’s ratio and the membrane thickness and radius, it is possible to extract numerically the residual stress from the stiffness measurement using (2), (3) and (4).

When $k > 300$ (which corresponds to $\frac{a}{l} = 10^4$), the residual stress can be well estimated according to the following relationship [10]:

$$ \sigma = \left( \frac{S}{A} \right) \cdot \left( \frac{1}{l^2} \right) $$

where $A$ is a constant depending on the physical and mechanical characteristics of the membrane:

$$ A = \left( \frac{\beta^2 \cdot E}{12 \cdot (1 - \nu^2) \cdot a^2} \right)^{\frac{1}{4}} \cdot \frac{1}{20.08 \cdot \alpha \cdot \beta^2} $$

Figure 3. Load vs. displacement curve measured using a nano-indenter head.
Note that the assumption \( \left( \frac{a}{r} \right)^2 \cdot \frac{\sigma}{E} \geq 10^4 \) corresponds to materials with a low Young’s modulus and a low thickness compared to the membrane radius as it is usually the case for the membranes investigated in this work.

3. Results and Discussion

As a first step, mineral polycrystalline silicon (Si poly) membranes were studied to verify the measurement feasibility. Different membrane radii were investigated (from 450 to 2550 μm), for a Si poly thickness of 4 μm. Figure 4 shows the measured stiffness as a function of the position of the tip with respect to the center of the membrane for different membrane radii \( a \).

When the co-spherical tip is close to the edges of the membrane (typically less than 500 μm), the measured stiffness is very strongly dependent on the position of the tip with respect to the membrane. On the other hand, for the largest membrane radius \( a = 2550 \) μm, the stiffness is found to be very stable in a range ~ \( -a/2 \) to \( +a/2 \). The stiffness results presented thereafter were measured on the largest radius membranes (2.55 mm).

Using the analytical model presented in section 2.3, a residual stress of the Si poly membrane of 14 MPa was extracted assuming a Young’s modulus of 130 GPa and a Poisson’s ratio of 0.28. Note that the Young’s modulus and Poisson’s ratio used in this work were obtained from the polymers manufacturers. To validate the measurement methodology, extractions of residual stress values using the well-known Stoney method \([5,6]\) on samples having the same membrane material were performed. The residual stress extracted with the Stoney method, 15 MPa, was found to be extremely close to the value obtained with the point-deflection method developed in this work (14 MPa), giving us a good level of confidence in the validity of the extraction procedure.

Furthermore, several polymeric materials were studied in this work: three siloxane derivatives with thicknesses ranging from 2.8 to 5 μm, a Young’s modulus between 150 and 800 MPa and low residual stress. Parylene was also studied as reference material because its relatively high residual stress allows stress extraction using the Stoney method. All tested devices were circular with a cavity diameter of 5 mm. A fabrication process involving liquid encapsulation was used for the polymeric membrane devices in order to avoid membrane stiffness at the bottom of the cavity or fracture (see Figure 2). To assess the effect of the liquid on the measured stiffness, devices with a Parylene membrane were fabricated with two liquids featuring different viscosities ranging from 2 cPo to more than 100 cPo. A slight difference in the measured stiffness (about 10%, a value in good agreement with the uncertainty of measurement) was obtained showing that the viscosity has little effect on the final result especially in the small deformation regime. For the results presented thereafter, the low viscosity liquid was used as encapsulated liquid.

Figure 5 shows a stiffness measurement performed on a siloxane derivative \( n^1 \) membrane as a function of the position on the cavity diameter. As noticed previ-
ously, it can be seen that the measured stiffness does not vary significantly along the diameter (less than 5%). This observation was repeated for all the samples tested.

Table 1 shows the measured stiffness and the residual stress extracted by the method presented in this work. As shown in Table 1, the residual stresses extracted on devices with a siloxane derivative n°1 membrane with two different thicknesses (2.8 and 5 μm) are almost identical (15.6 MPa vs. 15.4 MPa). This seems to show that the measurement is really material characteristic and is independent of the membrane thickness.

To assess these results, extractions of residual stress using the Stoney method on samples having the same membrane material were performed. The mean residual stress obtained with this method for the different polymers tested is listed in Table 1. Figure 6 shows the comparative values of residual stress obtained using the Stoney method and the method by point deflection developed in this work, with an estimation of the uncertainty in the extracted values.

For the Stoney method; the margin of error for the extracted residual stress was evaluated as follows. The so-called Stoney formula permitting the residual stress evaluation from wafer bow measurements is [5,6]:

\[
\sigma = \frac{E_s \cdot t_f^2}{6 \cdot (1 - v_s) \cdot t_f} \cdot \frac{1}{R}
\]  

(7)

where \(E_s\), \(t_s\), and \(v_s\) are the substrate Young’s modulus, thickness and Poisson’s ratio, \(t_f\) is the thickness of the stressed film and \(R\) is the wafer radius of curvature induced by the stress. In our case, \(R\) was evaluated from the measurement of wafer bows using a contact profilometer. For 200 mm wafers, we estimate the bow measurement uncertainty to be ±3 μm. Knowing the film thickness, the margin of error can then be deduced from (7). Moreover, for thin polymeric membranes where the residual stress is very low (i.e. siloxane derivatives n°2 and n°3), the variation of the wafer bow can be hardly measured precisely. Assuming that less than 6 μm bow variation is not significant (corresponding to the total margin of error range) a stress measurement precision of 10 MPa (6 MPa) for a 2.8 μm (5 μm) thick membrane can be obtained from (7). In other words, we could not estimate residual stresses below these values with the Stoney method.

Figure 6. Residual stress extracted using the Stoney method and the point deflection method developed in this work for different circular membrane materials (radius 2500 μm).

Table 1. Summary of the polymeric materials mechanical properties, of the measured stiffness of these polymers and comparison of the residual stress extracted with the Stoney method with the residual stress extracted by the methodology developed in this work.

<table>
<thead>
<tr>
<th>Membrane</th>
<th>Si poly</th>
<th>Parylene</th>
<th>Siloxane derivative n°1</th>
<th>Siloxane derivative n°1</th>
<th>Siloxane derivative n°2</th>
<th>Siloxane derivative n°3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (μm)</td>
<td>4</td>
<td>1.1</td>
<td>2.8</td>
<td>5</td>
<td>5</td>
<td>2.8</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>130</td>
<td>3</td>
<td>0.8</td>
<td>0.8</td>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>Average stiffness (N/m) on circular membrane</td>
<td>160</td>
<td>40</td>
<td>50</td>
<td>98</td>
<td>14</td>
<td>8</td>
</tr>
<tr>
<td>Average residual stress Stoney method (MPa)</td>
<td>15</td>
<td>30</td>
<td>17</td>
<td>18</td>
<td>&lt; 6</td>
<td>&lt; 10</td>
</tr>
<tr>
<td>Margin of error (MPa)</td>
<td>±4</td>
<td>±10</td>
<td>±5</td>
<td>±8</td>
<td>N.A.</td>
<td>N.A.</td>
</tr>
<tr>
<td>Average residual stress point deflection method (MPa)</td>
<td>14</td>
<td>34</td>
<td>15.6</td>
<td>15.4</td>
<td>2.2</td>
<td>2.3</td>
</tr>
<tr>
<td>Margin of error (MPa)</td>
<td>±2</td>
<td>±4</td>
<td>±2</td>
<td>±2</td>
<td>±0.5</td>
<td>±0.5</td>
</tr>
</tbody>
</table>
On the other hand, the uncertainty of the stiffness measurement can be assessed to be +/-10%. The margin of error of the extracted residual stress using point deflection can then easily be deduced from (5). Note that when the measured stiffness is very low (typically below 15 N/m), +/-20% margin of error was used because the measurement was found to be more sensitive to dispersion.

For the parylene membrane, the results given by the two techniques are very comparable, giving us a good level of confidence in the validity of the values extracted by the point deflection method. Moreover, we can also note that the values obtained with the siloxane derivative n°1 are comparable for the two methods with a larger margin of error for the Stoney method. This is due to the fact that the bow measurements performed for this material show a very low difference between and after processing the membrane due to the low stress that implies a large sensitivity to measurement dispersion. In return, the values of residual stress cannot be extracted with a satisfactory precision with the Stoney method.

In the case of the siloxane derivatives n°2 and 3, where the residual stress is extremely low, the Stoney method cannot give precise values because the bow variation before and after membrane processing is hardly measured (wafer bow variation below 6 μm). However, using the point deflection method, residual stress values of 2.2 MPa and 2.3 MPa for the siloxane-derivative n°2 and 3 were obtained in a reproducible manner. In result, this shows that it is possible to access to very low residual stress values using this type of devices and extraction method.

4. Conclusion

In this work, we have developed an alternative methodology to assess the stiffness and evaluate the residual stress of thin, flexible polymer membranes. This methodology is based on original test structures where the membrane is supported by a liquid in a circular cavity and uses stiffness measurement by point deflection with a nano-indenter. For sufficiently large cavities, stable and reproducible stiffness values, almost independent of the position of the nano-indenter tip when excluding an edge area of about 500 μm, were measured. Knowing the Young’s modulus and Poisson’s ratio of the material and using the geometrical dimensions of the membrane (thickness and radius) the residual stress can be extracted from the stiffness values. This combination of alternative test structure and measurement procedure permits to evaluate the stiffness and the stress of polymeric materials that would have been too thin and flexible for other kind of devices and measurement techniques.

Several polymers with different Young’s modulus, chemical composition, and thickness were evaluated. With the new method developed in this work, we obtained values of the residual stress that are comparable to those obtained by the Stoney method when this was applicable (i.e. relatively large residual stress) with a much lower uncertainty. Moreover, very low values of residual stress were measured with this method that could not be estimated reliably using the Stoney method. We believe this methodology can help to evaluate the stress of new polymeric materials in order to make the appropriate choices on the most adapted polymer according to the desired application.

References


*Manuscript Received: May 1, 2013
Accepted: Oct. 12, 2013*