THE MECHANICAL PROPERTIES OF SUBMICRON-THICK, LARGE-AREA 3C-SiC DIAPHRAGMS

By

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proprietary material contained therein.
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The Mechanical Properties of Submicron-Thick, Large-Area 3C-SiC Diaphragms

Abstract

by

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This thesis presents the results of an investigation to characterize the mechanical properties of submicron-thick, single crystalline cubic silicon carbide (3C-SiC) films. 3C-SiC films in this thickness range are of significant interest due to their potential utility in MEMS and NEMS applications, and information regarding the mechanical properties of these films is critically important. The load-deflection technique was used to determine the Young’s modulus and residual stress of epitaxially-grown (100) 3C-SiC films that were bulk micromachined into suspended diaphragms. Diaphragm thicknesses were nominally 500 nm and 250 nm. It was found that the films have residual stresses that are high and tensile (1.21GPa), which affected accurate determination of the Young’s modulus. The durability of these films, as well as 2 μm-thick polycrystalline 3C-SiC films, was investigated by subjecting the structures to cyclic loading. No distinguishable differences were observed in the residual stress and Young’s modulus after 30,000 load-deflection cycles.
Chapter 1

Introduction

1.1 Objective:

Microelectromechanical systems (MEMS) is a rapidly advancing technology area that seeks to combine micrometer-scale sensors and actuators with solid state electronics to enable perception and control of the local environment. Born from microelectromechanical systems (MEMS), NEMS is essentially the nanoscale counterpart of MEMS. Both technology areas currently leverage heavily on silicon and silicon based processing techniques, many of which are used in state-of-the-art integrated circuit fabrication. To date, a large class of MEMS and NEMS devices consist of mechanical structures such as cantilevers, bridges, and diaphragms that are used to realize mechanical resonators, mass sensors and related force transducers. Because the fabrication techniques for these devices are largely derived from the integrated circuit industry, the main materials for MEMS and NEMS include Si, Si$_3$N$_4$, GaAs and related III-V semiconductors and common metals. However, the desire to expand the use of MEMS and NEMS beyond what is currently capable has motivated the development of new structural materials.

SiC is a particularly promising material for MEMS and NEMS structures due to its chemical inertness, temperature stability (electrically and mechanically), strength and high acoustic velocity. The Young’s modulus of SiC films has been reported to be in the 150 to
520 GPa range [1-17], with a value as high as 710 GPa reported [17]. The Young’s modulus of SiC is considerably higher than other common Si-based NEMS materials, such as silicon (185 GPa) [18] and Si₃N₄ (200-330 GPa) [19]. The high Young’s modulus of 3C-SiC translates directly to a higher acoustic velocity. High acoustic velocity materials are particularly attractive for nanoscale resonators because high acoustic velocities translate directly to high resonant frequencies. In fact, the first mechanical resonator to exhibit a fundamental resonant frequency of over 1 GHz was a 3C-SiC NEMS bridge [20]. The preponderance of Young’s modulus data for 3C-SiC comes from thin film specimens that have thicknesses in the micron to tens of microns range. To the best of our knowledge the literature lacks any information regarding the Young’s modulus of submicron 3C-SiC films, especially as thicknesses approach 100 nm.

In addition to Young’s modulus, other mechanical properties of importance to MEMS and NEMS include residual stress and fatigue resistance. Epitaxial 3C-SiC films grown on Si in the micron thickness range generally exhibit residual stresses that are tensile in nature and range between 100 to 400 MPa range [3]. Like the Young’s modulus, little, if anything has been reported about the residual stress in submicron-thick epitaxial 3C-SiC films. The literature is equally absence of any information about the fatigue properties of submicron 3C-SiC films. The residual stress will affect the resonant frequency of vibrating nanostructures and the fatigue properties will speak to the long-term viability of such structures when under load.

The primary purpose of this thesis is to characterize the Young’s modulus, residual stress, and fatigue resistance of sub-micron thick 3C-SiC films using large-area, bulk micromachined, 1 x 1 mm² diaphragms as test structures. To the best of our knowledge, this
is the first time that such large-area diaphragms were used in conjunction with load-deflection testing to characterize the mechanical properties of submicron-thick 3C-SiC films.
2.1. Young’s Modulus

The Young’s modulus of a solid material is a proportionality between the stress and the strain of the material for a uniaxially applied force [21]. The Young’s modulus is independent of the bulk geometry of the solid and is expressed as:

\[
E = \frac{\sigma}{\varepsilon}
\]  
(Eq. 2.1)

where \( \sigma \) is the normal stress and \( \varepsilon \) the normal strain. Normal stress is defined as the ratio of force (F) applied through a cross sectional area (A):

\[
\sigma = \frac{F}{A}
\]  
(Eq. 2.2)

Normal strain is the ratio describing the change in length (\( \Delta L \)) of a solid compared to the unstressed length (\( L_0 \)):

\[
\varepsilon = \frac{\Delta L}{L_0}
\]  
(Eq. 2.3)
2.2 Poisson’s Ratio

The Poisson’s ratio ($\nu$) describes the change in cross sectional area when a material undergoes compressive or tensile stress. The Poisson’s ratio is the ratio of transverse strain ($\varepsilon_D$) compared with axial strain ($\varepsilon_A$). Here, $\varepsilon_D$ is given by the change in cross sectional area divided by the unstressed area, and $\varepsilon_A$ is the change in length divided by the unstressed length:

$$\nu = \frac{\varepsilon_D}{\varepsilon_A} = -\frac{\Delta D}{\frac{D_0}{L_0}} \frac{\Delta L}{L_0}$$  \hspace{1cm} \text{(Eq 2.4)}$$

2.3 Residual Stress

Residual stress describes the biaxial stress of a film which is often a result of various mismatches between the film and the underlying substrate. A leading source of residual stress in films grown by chemical vapor deposition (CVD) is the mismatch thermal expansion coefficients between film and substrate. Many CVD films, especially those grown by epitaxy, are deposited at relatively high temperatures (ie., 400 to 1350°C). As the reactor system is cooled after deposition, the film and substrate will contract by different amounts. This contraction (or expansion if the material is being heated) is described by the coefficient of thermal expansion. In the case of a thin film that is deposited onto a substrate, the two materials are chemically bonded together at the interface. If the contraction does not cause catastrophic delamination or cracking in the film, post deposition cooling will result in the build-up of stress in the film.
The residual stress can be categorized as tensile or compressive depending on whether the film contracts or expands, respectively. A film under compressive stress expands with relation to the substrate, and convention assigns a negative value to this stress. A film under tensile stress will contract relative to the substrate, and is characterized by a positive value.

2.4 Fatigue

Fatigue describes the development of structural damage when a material is subjected to cyclical loading and stress. Fatigue occurs when a material is loaded and unloaded repeatedly to a load level which is below the level at which the material will suffer catastrophic and irreversible damage. As the material is loaded and strained, microscopic cracks may begin to form and grow. This formation of microcracks will weaken the material and lessen its ability to withstand further cycling. If this continues, eventually the defect density of the material may reach a critical level and will lead to a catastrophic failure, causing fracture or burst in brittle materials.

Fatigue will likely start at a dislocation, a break or defect in crystal structure. Due to the already altered crystal structure of the bonds at these sites, the material will generally be weaker at these locations and allow for greater mobility within the lattice. The extra mobility may allow for further separation of the lattice which may permit the defect to spread and affect the surrounding lattice bonds. These dislocations will continue to propagate, and as the material is loaded, the stress may cause the lattice at these dislocations to slip or pull, allowing the cracks to grow.
Fatigue life is significantly affected by factors such as material geometry, material properties, defect density and surface quality. When applying loads to certain geometries, such as square or rectangular diaphragms, the stresses will not be uniform. These nonuniformities will cause locations of concentrated stress that, if extreme, could result in structural failure. Surface roughness also can lead to a higher defect density in the near surface region, also enhancing mechanical failure under extreme loading conditions.
Chapter 3
Experimental Methods

3.1 The Load-Deflection Testing Apparatus

In order to determine the Young’s modulus and residual stress of the submicron-thick 3C-SiC films, bulk micromachined diaphragms made from nominally 250 nm and 500 nm-thick films were subjected to an interferometric load deflection measurement technique. The load-deflection technique, also called the bulge test, is a method commonly used in the study of MEMS to determine the elastic properties of thin films and has been extensively used by MEMS researchers at CWRU [1-5]. This technique works particularly well when the film of interest can be rendered into a suspended diaphragm by silicon bulk micromachining. The process involves the application of force in the form of differential pressure to the suspended diaphragm and measuring the respective displacement of the diaphragm as a function of applied pressure. This data is compiled to give a stress vs. strain relationship which can be analyzed mathematically to determine the Young’s modulus of the material and the residual stress in the film.

The load deflection testing setup used in this thesis is illustrated in Figs. 3-1 and 3-2. The apparatus consist of a microscope on which a Moire interferometer is mounted on a 10X objective. The interferometer is illuminated through the microscope objective by a
monochromatic 540 ± 10 nm beam of light. A beam splitter inside interferometer divides the incoming light into two beams along perpendicular paths. One beam is reflected by a mirror positioned at a fixed distance from the beam splitter, and the other is reflected by the test sample, in this case a pressure-loaded SiC diaphragm, which is illuminated by the second beam which is allowed to exit the interferometer. The reflected beams recombine at the splitter and are detected by a CCD camera and imaged by a television monitor. The pressure-loaded diaphragm deflects toward the interferometer, inducing phase differences in the recombined beam resulting in interference patterns. Due to the hemispherical shape of the deflected diaphragm, the interference pattern takes the form of concentric rings. Essentially, for each increment of the wavelength that the light travels between the diaphragm and the parting mirror, the recombined beam will experience a phase shift of 0°, creating constructive interference, and for each half increment, the recombined beam will undergo a phase shift of 180°, which leads to destructive interference. In terms of the patterns seen on the monitor, constructive interference results in high intensity rings while destructive interference causes the appearance of low intensity rings. At maximum deflection, the rings of both diaphragm geometries appear smaller and tightly concentric due to the bulged profile of the diaphragm. As the pressure is released and the diaphragm begins to flatten, the interference pattern will remain concentric but appear to enlarge. The images in Figure 3-3 illustrate the interference rings observed while testing a square diaphragm.
Figure 3-1: Schematic diagram of the load deflection apparatus used in this thesis.
Figure 3-2: Photograph of load deflection apparatus used in this thesis.
Figure 3-3: Photographic images of interference rings on pressure-loaded square diaphragms: (a) near maximum deflection and (b) near minimum deflection.

3.2 Preparation of Samples for Load Deflection Testing

To prepare bulk micromachined SiC diaphragms for load deflection testing, the samples were mounted; film side down, to aluminum chucks using Stronghold 7036 Blanchard Wax. This particular wax is very strong and hard at room temperature, melts at 65°C and can be used to form air tight seals between surfaces. The samples were mounted film side down in order to prevent delamination of the film, as well as to prevent any effect on the test from the geometry of the cavity sidewall [3]. The typical chuck is two inches in diameter, with a 0.66mm diameter hole drilled in the center, which connects to a piece of ¼ inch stainless steel tubing. The tubing protrudes from the side with a ¼ inch Swagelok
plumbing attachment. Figure 3-4 contains a photograph and cross-sectional schematic of a diaphragm chip mounted to a typical chuck.

![Photograph of diaphragm chip mounted on aluminum chuck](image1)

![Schematic diagram of diaphragm chip](image2)

**Figure 3-4:** (a) Photograph and (b) schematic diagram of a diaphragm mounted on an aluminum chuck for load deflection and fatigue testing.
3.3 Measurement Method

After mounting a diaphragm chip, the chuck was attached to a manually-controlled pressure manifold and positioned properly under the microscope-mounted interferometer. The manifold enabled both pressurization and depressurization of the suspended diaphragm by way of a series of quarter-turn ball valves and precision needle valves. For load deflection testing, the preferred measurement technique involved first pressurizing the diaphragm to a predetermined maximum testing pressure and then slowly depressurizing the diaphragm through the precision needle valves. As stated previously, pressurization results in diaphragm deflection that is detectable by the interferometer. For the setup used in this thesis, illumination of the interferometer with a 540 nm light source results in an interference pattern for every 270 nm of deflection. After reaching maximum pressure and establishing an interference pattern, the diaphragm was slowly depressurized at a rate such that changes in the interference pattern were clearly observable. Each time an interference ring would disappear, a pressure reading was recorded by a LABView program on a data acquisition computer by way of a voltage output from an Omega 24PC100G electric pressure gauge. This procedure was performed until the diaphragm was completely depressurized. Load-deflection testing was performed at room temperature and atmospheric pressure. The LABView program would then generate plots of applied pressure versus deflection, from which the Young’s modulus and residual stress for the diaphragm could be extracted.

3.4 Diaphragm Dimensional Measurements

In order to ensure proper analysis of the Young’s modulus and residual stress values of the diaphragms, precise measurements of diaphragm thickness, side width, and area are
required. The thickness of each diaphragm was measured using a *Nanospec 4000 AFT* optical reflectometer. The spectrometer measurements were verified by performing a cross sectional scanning electron microscopy (SEM) measurement on one of the diaphragm chips. It was found that the optical measurements were within 1% of the SEM measurements. Table 3-1 summarizes the measurement results and Figure 3-5 shows representative micrographs from the SEM measurements.

<table>
<thead>
<tr>
<th>Thickness by SEM (nm)</th>
<th>Thickness by Nanospec (nm)</th>
<th>Difference (nm)</th>
<th>Difference %</th>
</tr>
</thead>
<tbody>
<tr>
<td>527.0</td>
<td>522.0</td>
<td>50</td>
<td>-0.95%</td>
</tr>
</tbody>
</table>

*Table 3-1: Comparison of thickness measurements as taken by SEM and optical reflectometry.*
Figure 3-5: High resolution, cross-sectional SEM images used to determine film thickness. a) 45° cross sectional SEM image of a single crystal 3C-SiC film on Si. The 3C-SiC film is the dark region in the middle of the image. b) Cross sectional image of a doped poly-SiC film. [from Ref. 1].
Based on the data in Table 3-1, it was determined that optical reflectometry would be a suitable method to measure diaphragm thickness. As such, the Nanospec was used to measure the film thickness for each chip being studied. Such measurements were important since film thickness can vary significantly across the wafer from which the diaphragm samples were fabricated. The thickness variation becomes more pronounced for thinner films. Table 3-2 summarized the findings of a study to determine the range of thicknesses among the diaphragm test specimens for each of the nominal thicknesses studied in this thesis.

<table>
<thead>
<tr>
<th>Diaphragm Sample Group</th>
<th>Maximum Thickness (nm)</th>
<th>Minimum Thickness (nm)</th>
<th>% Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>3C-SiC 500</td>
<td>527</td>
<td>486</td>
<td>7.5</td>
</tr>
<tr>
<td>3C-SiC 250</td>
<td>277</td>
<td>163</td>
<td>41</td>
</tr>
</tbody>
</table>

*Table 3-2: Thickness variation for the single crystal 3C-SiC films investigated in this thesis.*

The side width of a diaphragm is an essential parameter for extracting the Young’s modulus and residual stress values from the pressure versus deflection data. For the diaphragms studied in this thesis, the side widths of each diaphragm were determined using an optical microscope at 50x magnification with a Microde I XYZ micrometer displacement measuring stage. Probable variations in side widths from chip to chip as well as on an individual chip required the measurement of the each side width of each diaphragm subjected to load-deflection testing. The single crystalline samples were determined to have widths with a range from 1.02 to 1.09 mm.
3.5 Calibration of the Load Deflection Apparatus and Operator

The first step in the load-deflection process was to ensure that the apparatus was properly calibrated and that the operator was capable of gathering accurate data. In order to ensure this, diaphragms that were characterized in a previously study were subjected to load-deflection testing. In specific, 2 micron-thick, polycrystalline 3C-SiC diaphragms originally fabricated and characterized by Jacob Trevino and documented in his M.S. Thesis were subjected to testing. Table 3-3 compares the measurements made in this thesis to those made on similar diaphragms from the same films by J. Trevino.
<table>
<thead>
<tr>
<th>CALIBRATION DATA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measurements from Trevino’s Thesis</td>
</tr>
<tr>
<td>A</td>
</tr>
<tr>
<td>B</td>
</tr>
<tr>
<td>C</td>
</tr>
<tr>
<td>D</td>
</tr>
<tr>
<td>E</td>
</tr>
<tr>
<td>Average</td>
</tr>
<tr>
<td>Standard Deviation</td>
</tr>
</tbody>
</table>

| Measurements from this Thesis | $\sigma$ (MPa) | $E$ (GPa) |
| 1                | 128             | 344       |
| 2                | 110             | 326       |
| 3                | 101             | 361       |
| 4                | 114             | 346       |
| 5                | 112             | 341       |
| Average          | **113**         | **343.6** |
| Standard Deviation | **9.75**       | **12.50** |

**Table 3-3**: A comparison of load-deflection data for a poly-SiC diaphragm characterized by Trevino [1] with a poly-SiC diaphragm characterized in this thesis. The diaphragms were likely not the same but came from the same fabrication run and same wafer.
These data indicate that both the instrument and operator were adequately prepared for the proposed investigation. For diaphragms fabricated from polycrystalline SiC films, J. Trevino found an average Young’s modulus of $\sim\! 336 \pm 12$ MPa and an average residual stress of $129 \pm 12$ GPa. By comparison, diaphragms made from the same films evaluated in this thesis yielded an average Young’s modulus of nominally $344 \pm 13$ GPa and an average residual stress of $113 \pm 10$ MPa. The fact that the average Young’s modulus value for the diaphragms measured in this thesis lies with the uncertainty of that measured by J. Trevino, and likewise for the residual stress, indicated that both the experimental apparatus and the operator were performing properly and thus ready for load-deflection testing on samples with unknown mechanical properties.

3.6 Nanoindentation

Nanoindentation is a testing method used to determine Young’s modulus and hardness of materials. In this technique, a small sharp tip, usually made of diamond and fabricated in the shape of a three sided pyramid structure with a width of a few nanometers, is pressed into the surface of a sample with a precisely controlled loading force. The depth of penetration is measured, and the area of the indent is calculated using the known tip geometry. A plot of the load versus displacement is generated and can be used to extract the reduced modulus, which is the combined modulus of the tip and the material being tested. The reduced modulus can be converted to the Young’s modulus by a simple conversion formula. In this thesis, nanoindentation was used to acquire an independent measurement of Young’s modulus for select samples subjected to load-deflection testing. The instrument used for the nanoindentation measurements made in this thesis was a Dimension 3100 Atomic Force Microscope with Hysitron Triboscope.
3.7 Cyclic Load Testing

To investigate the room temperature fatigue characteristics of the SiC diaphragms, a pressure cycling system was custom designed and fabricated. This apparatus, shown in the photograph of Fig. 3-6 could automatically pressurize and depressurize the diaphragms in cyclic fashion for a preset number of cycles. Each pressure/depressure cycle varied the differential pressure on the diaphragms between 85% of the predetermined burst pressure and atmospheric pressure. This apparatus utilized an *Alicat Scientific PC100PSIA-D* digital electronic pressure controller commanded by a *LabVIEW* program and a needle valve, set to leak slowly and release the pressure. During each cycle, the diaphragms under test were pressurized for 10s, and depressurized for 10s in repeated manner. Samples for testing were mounted on the same chucks used for load-deflection testing using the same mounting procedures. As such, the diaphragms were subjected to load deflection testing at predetermined intervals to determine the effect of cyclic loading on the Young’s modulus and residual stress. The apparatus was designed to support the simultaneous testing of up to five diaphragms per testing session.
Figure 3-6: Photograph of the pressure system designed and constructed for cyclic testing of bulk micromachined diaphragms.
Chapter 4

Fabrication of Silicon Carbide Diaphragms

In this thesis, diaphragms made from 250 nm and 500 nm-thick single crystalline 3C-SiC films as well as diaphragms made from a 1790 nm thick polycrystalline 3C-SiC film were investigated by load-deflection testing and cyclic load testing. All of the diaphragms studied here were fabricated prior to this thesis. The following sections describe the methods used to deposit the SiC films and to fabricate the thin film diaphragms and are included for completeness.

4.1 Single Crystal 3C-SiC Growth

The single crystalline 3C-SiC films were grown on 100 mm-diameter, (100) Si wafers by atmospheric pressure chemical vapor deposition (APCVD) in a custom-built, rf-induction heated, cold wall reactor housed in the Microfabrication Laboratory (MFL) at CWRU. The epitaxial growth process involved three key steps performed sequentially and first described in [4]. The first step was an in-situ surface treatment designed to remove the native oxide, organics and other contaminants from the Si substrate surface by heating the wafer to 1000ºC in the presence of H₂. This surface treatment was performed for 5 min, after which the wafers were cooled to below 500ºC. After the cool down step, an ultra-thin (~ 10 nm) 3C-SiC film
was grown on the wafer by a process called carbonization. Carbonization involved the direct conversion of the Si surface by exposing a heated Si substrate to a gaseous hydrocarbon. The hydrocarbon was mixed with H₂, which serves to etch non-stoichiometric deposits on the surface. The carbonization step was performed by adding C₃H₈ (15% in H₂) to the H₂ carrier gas flow, heating the wafers to roughly 1300°C, and holding the temperature and gas flows constant for 90 s. This step resulted in the formation of a continuous 3C-SiC film on the wafer surface. The process was self-limiting due to the very low diffusion rates of Si and C in SiC and the continuity of the thin SiC film, so film growth was sustained by adding SiH₄ (5% in H₂) to the C₃H₈ and H₂ gas flows. The film growth process was terminated by ceasing the flow of precursor gases and cooling the substrate. The epitaxial 3C-SiC films were grown using a H₂ flow rate of 25 slm, a C₃H₈ flow rate of 84 sccm during the carbonization step, and C₃H₈ and SiH₄ flow rates of 26 and 102 sccm, respectively during the film growth step. Under these conditions, single-crystalline 3C-SiC films were grown at a rate of 1 micron/hr (16.67 nm/min) which translates to a growth period of roughly 15 minutes for the 250 nm-thick films and 30 minutes for the 500 nm-thick films. Previous studies have shown that these films are single crystalline (100) 3C-SiC [4].

4.2 Polycrystalline 3C-SiC Growth

The polycrystalline 3C-SiC samples were prepared in the MFL at Case Western Reserve University using a low pressure chemical vapor deposition system described elsewhere [1, 5]. The films were deposited on 100 mm-diameter (100) single crystal, p-type doped silicon wafers. Prior to deposition, the wafers were subject to a full RCA clean. The deposition process used SiH₂Cl₂ and C₂H₂ (5% each) precursor gases as the sources of Si and C, respectively, with H₂ as the carrier and NH₃ (5%) as the dopant. SiH₂Cl₂ and C₂H₂ (5% in
H₂) flow rates were held constant at 35 sccm and 180 sccm, respectively. The flow rate of the ammonia doping gas was 64 sccm. The deposition temperature was held constant 900°C and the pressure was fixed at 4 Torr. Previous studies showed that these films have a low residual stress of between 130 and 150 MPa [1]. This process yielded a film of with an average thickness of 1790 nm.

4.3 Diaphragm Fabrication

Fabrication of suspended diaphragms from the aforementioned films involved conventional silicon bulk micromachining. Following deposition, the wafers were subjected to full piranha and RCA cleans. Upon the clean wafers a 1.5μm-thick thermal oxide layer was grown by wet oxidation at 1100°C on the backside of the wafers. A nominal oxide layer was grown on the SiC surfaces. The wafers were then patterned using conventional photolithographic processing techniques to form a photoresist-based oxide etch mask on the newly grown oxide surface. The unprotected regions of the oxide layer were etched away using a buffered hydrofluoric acid solution. During this step, the thin oxide on the SiC surfaces are also etch away. The resulting structure was a patterned silicon dioxide film that served as an etch mask for the silicon bulk micromachining step. The pattern was a series of square windows. Roughly 276 windows were patterned on each 100 mm-diameter wafer.

Once the oxide layers were patterned, the wafers were anisotropically etched in a bath of potassium-hydroxide (KOH) and deionized water for 26 hours at 55°C. In the unmasked regions, silicon was selectively etched along the (111) planes, creating a trapezoidal cavity with sidewalls at angles of 54.7°. The etch rate of SiC in KOH is negligible, so it acts as an etch stop, allowing this process to create clean, square SiC diaphragms with nearly uniform
area. This process generated square diaphragms for each respective sample of approximately 1 mm in width, with some variation in area due to uneven thickness in the substrate as well as variance in photolithographic patterning due to wafer curvature. The films were then rinsed thoroughly and individual chips were taken from the wafer and prepared for testing. Figure 4-1 is a schematic representation of the diaphragm fabrication process, while Figure 4-2 shows optical photographs of wafers and an individual chip after diaphragm fabrication.

**Figure 4-1:** Cross sectional schematic of the bulk micromachining process used to fabricate SiC diaphragms.

*Step 1:* Grow SiC layer on the frontside and grow a 2μ-thick SiO₂ film on each side. Spin photo resist in top of SiO₂.

*Step 2:* The photoresist is patterned.

*Step 3:* Using BOE, the oxide is etched in the unmasked regions.
Figure 4-2: Optical photographs of bulk micromachined SiC diaphragms: (top) wafer-scale images of 250 and 500 nm-thick diaphragms, and (bottom) an individual SiC diaphragm.
Chapter 5
Data and Analysis

5.1 Burst Pressure Measurement

Accurate determination of the Young’s modulus using the load deflection technique requires that a sufficiently large pressure be applied to the diaphragm to induce large deflections on the membrane. As mentioned previously, the maximum pressure to be applied to a particular diaphragm thickness was determined by pressurizing select samples until they burst. The burst pressure was recorded, and used to determine the pressure to be used for each type of sample for the load deflection test, which was set to be 4 PSI less than the burst pressure. Table 5-1 summarizes the results of the burst pressure measurements.

<table>
<thead>
<tr>
<th>SAMPLE TYPE</th>
<th>BURST PRESSURE (psi)</th>
<th>TEST PRESSURE (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 nm thick 3C-SiC</td>
<td>28</td>
<td>24</td>
</tr>
<tr>
<td>500 nm thick 3C-SiC</td>
<td>37</td>
<td>33</td>
</tr>
<tr>
<td>1790 nm thick Poly-SiC</td>
<td>36</td>
<td>32</td>
</tr>
</tbody>
</table>

*Table 5-1: Burst pressure and maximum test pressure for the samples studied using the load-deflection method.*
5.2 Load deflection Data and Analysis

Using the load deflection apparatus discussed previously, pressure versus deflection data was acquired. The pressure data took the form of a voltage output from the pressure transducer. This voltage was converted to pressure in pounds per square inch (psi) using the following formula:

\[ P = \frac{(V-1)}{0.05} \]  \hspace{1cm} (Eq. 5.1)

This pressure was then converted to units of pressure in Pascals (Pa) by:

\[ P_{(Pa)} = P_{(psi)} \times 6896 \]  \hspace{1cm} (Eq. 5.2)

The pressure values were then plotted against their complimentary deflection values with each data point recorded signifying 270 nm of diaphragm deflection. Using the load deflection equation for square diaphragms:

\[ P = t \frac{W_o^2}{a^2} \left[ C_i \sigma_o + \frac{f(v)}{a^2} \frac{E}{1-v} W_o^2 \right] \]  \hspace{1cm} (Eq. 5.3)

and the linearized form of this load deflection equation:

\[ P/W_o = \frac{t}{a^2} \left[ C_i \sigma_o + \frac{E}{1-v} \frac{f(v)}{a^2} W_o^2 \right] \]  \hspace{1cm} (Eq. 5.4)
where the function $f(v)$ is $1.37(1.446-0.427v)$, and the known values for diaphragm thickness, side width, $C_1$, and an assumed value of 0.23 for $v$, the Young’s Modulus and residual stress were extracted from the data. While graphic plots of pressure vs. deflection give a relevant visual description of the elastic properties of the film, the linear model allows for a more accurate extraction of the Young’s modulus and stress values. Using the linear model and treating $W_0^2$ as the independent variable, the Young’s modulus is a function of the slope ($m$) of while the residual stress is a function of the intercept ($b$) of the resulting line:

\[
E = ma^4 \left(1 - v\right) / (t f(v)) \quad \text{(Eq. 5.5)}
\]

\[
\sigma = ba^2 / (C_1 t) \quad \text{(Eq. 5.6)}
\]

Figures 5-1 and 5-2 are representative plots of diaphragm center deflection versus applied pressure for a nominally 500 nm-thick, single crystalline 3C-SiC diaphragm and a 1790 nm-thick polycrystalline 3C-SiC, respectively. Likewise, Figs. 5-3 and 5-4 are representative plots of applied pressure/diaphragm center deflection versus the square of diaphragm center deflection (i.e., the linearized plots) for a nominally 500 nm-thick, single crystalline 3C-SiC diaphragm and a 1790 nm-thick polycrystalline 3C-SiC, respectively.
Figure 5-1: Load deflection curve for a 527 nm-thick single crystal 3C-SiC diaphragm. The maximum pressure was 31.43 psi resulting in a maximum deflection of 22.4 μm. This film was found to have a Young’s modulus of 798 GPa and residual stress of 1.73 GPa.
**Figure 5-2**: Load deflection curve for a 1790 nm-thick poly-SiC diaphragm characterized in this thesis. The maximum pressure was 31.4 psi resulting in a maximum deflection of 24 μm. This film was found to have a Young’s modulus of 361 GPa and residual stress of 101 MPa.
**Figure 5-3:** Linearized load deflection graph used for the 527 nm-thick single crystal 3C-SiC diaphragm of Fig. 5-1.
Figure 5-4: Linearized load deflection plot for the 1790 nm-thick poly-SiC diaphragm of Fig. 5-2.
In order to account for potential errors in the testing process, the load-deflection test was repeated at least 5 times for each sample. Average values were obtained for Young’s modulus and residual stress by summing the individual values and dividing by the count of data sets being investigated. The standard deviation for the distribution of individual Young’s modulus and residual stress values were calculated using Equation 5.7, which describes a reasonable expectation of uncertainty in the gathered values.

$$\left(\frac{n\sum(x^2) - \sum(x)^2}{n(n-1)}\right)^{1/2}$$  \hspace{1cm} \text{(Eq. 5.7)}$$

Tables 5-2, 5-3 and 5-4 summarize the average Young’s modulus and residual stress, as well as the standard deviation for each of these parameters (i.e., the uncertainties in the average values) for the 500 nm-thick 3C-SiC diaphragms, the 250 nm-thick 3C-SiC diaphragms and the poly-SiC diaphragms, respectively.

**Table 5-2: Average Young’s modulus and residual stress values for the ~ 500 nm-thick single crystal 3C-SiC diaphragms investigated in this thesis.**

<table>
<thead>
<tr>
<th>Thickness (nm)</th>
<th>E (GPa)</th>
<th>St. Dev.(GPa)</th>
<th>σ (GPa)</th>
<th>St. Dev. (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>527</td>
<td>798</td>
<td>29.6</td>
<td>1.73</td>
<td>0.026</td>
</tr>
<tr>
<td>487</td>
<td>704</td>
<td>12.2</td>
<td>1.21</td>
<td>0.081</td>
</tr>
<tr>
<td>494</td>
<td>733</td>
<td>8.23</td>
<td>1.15</td>
<td>0.074</td>
</tr>
<tr>
<td>496</td>
<td>740</td>
<td>32.9</td>
<td>1.64</td>
<td>0.033</td>
</tr>
<tr>
<td>525</td>
<td>886</td>
<td>15.4</td>
<td>1.68</td>
<td>0.044</td>
</tr>
</tbody>
</table>
Table 5-3: Average Young’s modulus and residual stress values for the ~ 250 nm-thick single crystal 3C-SiC diaphragms investigated in this thesis.

<table>
<thead>
<tr>
<th>Thickness (nm)</th>
<th>E (GPa)</th>
<th>St. Dev. (GPa)</th>
<th>σ (GPa)</th>
<th>St. Dev. (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>273</td>
<td>692</td>
<td>36.3</td>
<td>1.25</td>
<td>0.044</td>
</tr>
<tr>
<td>281</td>
<td>573</td>
<td>12.13</td>
<td>1.16</td>
<td>0.017</td>
</tr>
<tr>
<td>273</td>
<td>794</td>
<td>1.788</td>
<td>1.35</td>
<td>0.020</td>
</tr>
<tr>
<td>248</td>
<td>962</td>
<td>26.07</td>
<td>1.44</td>
<td>0.039</td>
</tr>
<tr>
<td>272</td>
<td>783</td>
<td>21.32</td>
<td>1.24</td>
<td>0.015</td>
</tr>
</tbody>
</table>

Table 5-4: Average Young’s modulus and residual stress values for the ~ 1790 nm-thick polycrystalline 3C-SiC diaphragms investigated in this thesis.

<table>
<thead>
<tr>
<th>Sample</th>
<th>E (GPa)</th>
<th>St. Dev. (GPa)</th>
<th>σ (MPa)</th>
<th>St. Dev. (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>344</td>
<td>10.6</td>
<td>128</td>
<td>11.1</td>
</tr>
<tr>
<td>B</td>
<td>326</td>
<td>11.9</td>
<td>110</td>
<td>3.7</td>
</tr>
<tr>
<td>C</td>
<td>361</td>
<td>4.4</td>
<td>101</td>
<td>2.1</td>
</tr>
<tr>
<td>D</td>
<td>346</td>
<td>9.7</td>
<td>114</td>
<td>3.5</td>
</tr>
<tr>
<td>E</td>
<td>341</td>
<td>11.3</td>
<td>112</td>
<td>4.9</td>
</tr>
</tbody>
</table>

The poly-SiC films were studied to ensure calibration of the equipment and proper execution of the measurement technique, as samples from the same wafer had already been measured by Trevino [1] and thus made for good calibration standards. Trevino’s results for Young’s modulus and residual stress for these diaphragms ranged between 328 and 356 GPa.
and 108 and 137 MPa, respectively. The data in Table 5-4 compare favorably with Trevino’s results and thus indicated that the experimental method and apparatus operator were suitable for the investigation of the submicron 3C-SiC diaphragms.

Examination of Tables 5-2 and 5-3 show that the submicron-thick 3C-SiC single crystal films exhibit a much higher modulus and stress values than was expected or has previously been reported for 3C-SiC films. The results reported herein were especially surprising because thicker films (~ 2 micron) grown by the same epitaxial growth recipe and characterized by the load-deflection technique yielded values of 350 GPa and 438 MPa for Young’s modulus, and residual stress, respectively [3]. The values for Young’s modulus reported in this thesis however, are as much as 2.75 times higher than those reported in Ref. 3. However, the high residual stress values, which are almost 4 times larger than the stress values reported in Ref. 3. These discrepancies suggest potential issues in using the load-deflection technique to large-area, ultrathin 3C-SiC diaphragms, especially if the films exhibit extremely high residual stress values.

5.3 Error Analysis

In order to determine the accuracy and usefulness of the data gathered in this thesis, all sources of error were considered, including sample-based, equipment-based, and operator-based errors. These sources of experimental error were previously characterized for the load-deflection technique by Mitchell, et al., [3, 16]. As such, the methods used in the execution of this thesis follow those outlined Refs. 3 and 16. The following text provides details regarding the potential uncertainties introduced in each of these categories.
The sample-based uncertainties include uncertainties in the measurement of the diaphragm geometry, specifically diaphragm thickness and side width. The thickness uncertainty was found by comparing a SEM measurement to optical refractometry measurements, and was found to be within 1%. The uncertainty associated with diaphragm side width is mainly connected to the bulk micromachining process used to fabricate the diaphragms. Since KOH etches the silicon substrate along the (111) Si crystal planes (which are oriented at 54.7° with respect to the (100) Si wafer surface), variations in the wafer thickness lead to varying diaphragm widths across the wafer. This can also result in the diaphragms not being perfectly square, and for some of the diaphragms measured in this thesis, a difference of 0.3% between sides of the diaphragm was observed.

As detailed in Michell’s thesis, the instrument-related uncertainty is minimal, and is a result of calibration errors in the pressure transducer and voltage meter. The pressure transducer and voltage meter were calibrated before each use, and was less than 1% of full scale output.

Another potential source of uncertainty is associated the value of Poisson’s ratio used to calculate the Young’s modulus. For this thesis, the value was not measured, but rather assumed to be 0.23 based on the work by Mitchell. To examine the sensitivity of the load-deflection analysis to different Poisson’s ratios, a series of deflection versus pressure curves were mathematically generated using Eq. 5.3, each curve using a different value for Poisson’s ratio. All other values were held constant. The Young’s modulus and residual stress values for these curves came from the load-deflection data from an actual sample. Figure 5-5 shows the results of these calculations. The simulation showed that Poisson’s ratios between 0.15 and 0.30 (a variance of up to 35%) yielded no more than a 4% variance.
in the deflection data. This analysis shows that the load-deflection data is relatively insensitive to the Poisson’s ratio for the high tensile stress and potentially high Young’s modulus values associated with the SiC diaphragms investigated in this thesis.

![Theoretical Load Deflection](image)

**Figure 5-5**: Simulated load deflection plots for various values of Poisson’s ratio (NU). For these curves, the following parameters were used: \( E = 705 \text{ GPa}, \sigma = 0.95 \text{ GPa}, \) thickness = 525 nm, and width = 1.05 mm for all curves.

The primary source of uncertainty in this experiment is operator-based error. This uncertainty is a result of issues related to the fact that the operator must determine when a pressure reading is made by observing the disappearance of the interference rings, as well as
the operator miscounting or misjudging ring position when refocusing the microscope. For diaphragms with a maximum center deflection of about 24 μm, the microscope focus must be adjusted 3 to 4 times throughout the experiment as the depressurized membrane moves out of the focal region.

To account for these sources of error, the determination of the Young’s modulus and residual stress for a particular diaphragm was not made from a single load-deflection measurement, but rather from 5 to 7 distinct load deflection measurements. The data in each load-deflection data set was closely analyzed graphically by looking for inconsistencies in the linearized plots, which magnify missing rings, to determine that the experiment was done properly and the data was accurate. The values gathered were then analyzed and averaged to give the most accurate assessment of Young’s modulus and residual stress possible.

5.5 Discussion of The Load Deflection Results

As shown in Tables 5-2 above, load-deflection data for the 500 nm-thick 3C-SiC diaphragms yielded an average Young’s modulus and residual stress values ranging from 705 to 886 GPa and 1.21 to 1.73 GPa, respectively. Similarly, the Young’s modulus and residual stress were determined for the single crystalline 250 nm films to be in the range of 573 to 962 GPa and 1.16 to 1.44 GPa, respectively, as shown in Table 5-3. These values are much higher than was expected, being 2 to 3 times larger than the Young’s modulus and 4 times the residual stress values reported by Mitchell [3] for single crystalline films of approximately 2 μm in thickness. To gain insight on this issue, the actual load deflection data for a 525 nm-thick diaphragm was plotted against a mathematically-generated load-
deflection curve for the same diaphragm using the average Young’s modulus and residual stress value from Mitchell’s work. Figure 5-6 shows these plots. The difference in the two curves is, to a large degree, a result of the distinct difference in the residual stress values in the two films. This analysis suggests that the load-deflection behavior is sensitive to high residual stresses such that an accurate extraction of Young’s modulus may be difficult in extremely high stress films.

![Load deflection with actual data represented for 525 nm sample, against theoretical deflection using Young’s Modulus and residual stress values reported by Mitchell (2001) with thickness of 525 nm](image)

**Figure 5-6:** Load deflection with actual data represented for 525 nm sample, against theoretical deflection using Young’s Modulus and residual stress values reported by Mitchell (2001) with thickness of 525 nm
Finite element modeling (FEM) of the submicron-thick diaphragms was performed to gain insight as to the behavior of high tensile stress diaphragms. Figure 5-7 is a FEM model of a 1 x 1 mm², ~ 500 nm-thick subjected to an applied pressure of 25.5 psi. The Young’s modulus and residual stress values were those determined experimentally for an actual diaphragm. The specific values for Young’s modulus and residual stress were 713 GPa and 1.13 GPa, respectively. The model predicted a center deflection under these conditions of 15.92 µm while the experimentally determined center deflection was 23.8 µm. This discrepancy suggests that the high residual stress may be affecting the ability to extract accurate values of Young’s modulus from the load-deflection data.

Figure 5-8 is a finite element model constructed so that the center deflection of the model diaphragm matched the experimentally measured center deflection for the appropriate applied pressure (25.15 psi) and a Young’s modulus of 350 GPa. This model predicts that the residual stress should be 0.53 GPa, which is about one-half of the values determined experimentally. The two FEM models suggest that residual stress does play a critical role in the accurate determination of Young’s modulus, and that such a determination may be difficult for high tensile stress films.
Figure 5-7: FEM model of a constructed using the Young’s modulus and stress values as determined by load deflection experimentation. The experimentally measured deflection for the applied pressure was at 23.8 µm
Figure 5-8: Finite element model constructed so that the center deflection of the model diaphragm matched the experimentally measured center deflection for the appropriate applied pressure of 25.15 psi.

5.4 Nanoindentation Results

To better understand the load-deflection results from the submicron, 3C-SiC films, in particular the unexpectedly high Young’s modulus values, nanoindentation was performed on a select set of samples. Each nanoindentation test yields a plot of load versus displacement which is used to calculate the reduced modulus. The reduced modulus is the
combined Young’s modulus of the indenter tip and the sample material and can be mathematically described as:

\[
E_r = \frac{1}{\beta} \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_p(h_c)}}
\]  
(Eq. 5.8)

In Eq. 5.8, \(E_r\) is the reduced modulus, \(A_p(h_c)\), is the projected area of the indentation at the contact depth \(h_c\), \(S\) is determined from \(dP/dh\), and \(\beta\) is a geometrical constant on the order of unity. The reduced modulus is related to the material under load by the following equation:

\[
\frac{1}{E_r} = \frac{(1 - \nu_s^2)}{E_s} + \frac{(1 - \nu_i^2)}{E_i}.
\]  
(Eq. 5.9)

where \(\nu_s\) and \(\nu_i\) are Poisson’s ratio of the sample and the indenter, respectively, and \(E_s\) and \(E_i\) indicate Young’s modulus of the sample and indenter, respectively. If these values are known for the intenter tip, and the Poisson’s ratio of the sample is known, \(E_s\) can be determined.

For this thesis, five distinct indentation tests were performed on the 500 nm, 3C-SiC films. The results of these tests are summarized in Table 5-5. The reduced modulus for the films ranged from ~ 166 GPa to 204 GPa, and averaging 174 GPa. These values translate to a Young’s modulus that ranges from 166 GPa to 228 GPa and averaging 188GPa.
Table 5-5: Reduced and actual Young’s modulus of the single crystal SiC 500 nm film from nanoindentation.

<table>
<thead>
<tr>
<th></th>
<th>Reduced Young’s Modulus (GPa)</th>
<th>Young’s Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>165.72</td>
<td>178.21</td>
</tr>
<tr>
<td>2</td>
<td>165.54</td>
<td>177.9</td>
</tr>
<tr>
<td>3</td>
<td>156.01</td>
<td>166.25</td>
</tr>
<tr>
<td>4</td>
<td>204.40</td>
<td>228.13</td>
</tr>
<tr>
<td>5</td>
<td>176.19</td>
<td>191.36</td>
</tr>
<tr>
<td>AVG</td>
<td><strong>173.57</strong></td>
<td><strong>188.05</strong></td>
</tr>
</tbody>
</table>

The Young’s modulus values in Table 5-5 are clearly much lower than those found by the load deflection technique. In fact, it can be seen that the Young’s modulus values for the single crystal 3C-SiC films using nanoindentation are closer to that of the silicon substrate (E=185 GPa) than those commonly reported for 3C-SiC (350 to 510 GPa). It is likely therefore, that the Si substrate is manifest in the nanoindentation data as a result of 3C-SiC films being so thin relative to the force applied to the indenter. Because nanoindentation was only being used to supplement the load-deflection data, no additional effort was performed to determine optimal conditions. This being said, the fact that the Young’s modulus values for the 3C-SiC films are much lower than what was expected indicates that the excessively high Young’s modulus values as determined by load-deflection are likely not to be representative of the films actual properties. This suggests that the high residual stress in the submicron 3C-
SiC films may be dominating the load-deflection behavior of the diaphragms and thus adversely affecting the ability to discern the true Young’s modulus.

5.6 Load Deflection of High-Stress Diaphragms

The data in this thesis suggests that the submicron 3C-SiC films have extremely high tensile stresses, and these stresses make it very difficult to use the load deflection method to determine the Young’s modulus. This can be seen in Fig. 5-9 which compares the load-deflection behavior for a ~ 500 nm-thick 3C-SiC film with a poly-SiC film. The load-deflection behavior of the single crystalline film is nearly linear over the entire pressure range whereas the load deflection behavior of the polycrystalline film follows the typical trend expected of thin diaphragms. The fact that the load-deflection behavior of the single crystalline film is linear suggests that it is stress dominated, and the slope of this particular curve indicates that the stress value is high ($\sigma = 1.68$ GPa). In contrast, the non linear load deflection curve of the poly-SiC film yields a stress of only ($\sigma = 112$ MPa). Because the pressure versus deflection curve of the single crystalline diaphragm is dominated by the linear stress-based term, the diaphragm cannot be driven to a deflection regime where the cubic Young’s modulus-based term has major influence. Moreover, the data in Fig. 5-9 show that, as expected, the film with the lower residual stress deflects more at a given pressure than the film with the higher residual stress. In specific, at 31.4 psi, the poly-SiC sample is deflected by 24 $\mu$m, while at the same pressure, the thinner single crystalline SiC sample is deflected only 22.4 $\mu$m. Therefore it must be that the 525 nm film has much higher values for residual stress than the thicker poly-SiC film.
Figure 5-9: Load deflection data for a single crystal, 525 nm-thick 3C-SiC diaphragm and a 1790 nm-thick poly-SiC diaphragm. Both diaphragms have a nominal area of 1 x 1 mm$^2$.

This effect was classified by Huston, et al. [22] where they discovered that, in order to attain accurate Young’s modulus values using the load deflection technique, certain conditions must be met regarding the ratio of Young’s modulus to residual stress. They give
the following criteria in order to obtain accurate Young’s modulus values using the load
deflection method:

\[
\frac{E}{\sigma_0} > 0.1 \frac{(0.801 + 0.061\nu)^3 a^2 (1-\nu)}{h^2}
\]  

(Eq. 5.10)

Where h is the diaphragm thickness. For the submicron-thick diaphragms studied in this
thesis, the criterion of Eq. 5.10 was not met. Using representative values from the 500 nm-
thick diaphragms studied in this thesis, the first term of Eq. 510 comes to 461.2 and the
second term is 166752.2, thus clearly violating the inequality. This equation suggests two
issues pertaining to the diaphragms in this study; namely, extremely high stresses that lead to
low values for the left side of the inequality and large diaphragm areas that lead to high
values on the right side of the inequality. It is possible that the combination of high tensile
stress and large diaphragm area that makes the determination of Young’s modulus from the
load deflection data not possible.

5.7 Cyclic Testing Results

Another goal of this research was to study the fatigue properties of the SiC films. In
order to do this, they had to be repeatedly subjected to controlled load deflection under a
large number of cycles, to a load approaching the yield strength of the material. Five
samples of the single crystalline ~500 nm films and five of the polycrystalline ~1790 nm
films were tested up to 30,000 cycles. The samples were stopped at controlled intervals
throughout the cycling and subjected to load deflection testing. The results of the load
deflection tests were compared throughout. These results can be seen in Tables 5-6, and 5-7 below.

**Table 5-6:** Young’s modulus and residual stress values for a 525 nm-thick single crystal 3C-SiC diaphragm subjected to cyclic loading.

<table>
<thead>
<tr>
<th>Number of cycles</th>
<th>E (GPa)</th>
<th>σ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>886</td>
<td>1.68</td>
</tr>
<tr>
<td>500</td>
<td>886</td>
<td>1.73</td>
</tr>
<tr>
<td>1,000</td>
<td>887</td>
<td>1.66</td>
</tr>
<tr>
<td>1,500</td>
<td>898</td>
<td>1.66</td>
</tr>
<tr>
<td>2,000</td>
<td>886</td>
<td>1.7</td>
</tr>
<tr>
<td>2,500</td>
<td>883</td>
<td>1.69</td>
</tr>
<tr>
<td>5,000</td>
<td>889</td>
<td>1.67</td>
</tr>
<tr>
<td>10,000</td>
<td>893</td>
<td>1.65</td>
</tr>
<tr>
<td>15,000</td>
<td>871</td>
<td>1.66</td>
</tr>
<tr>
<td>20,000</td>
<td>869</td>
<td>1.68</td>
</tr>
<tr>
<td>30,000</td>
<td>884</td>
<td>1.68</td>
</tr>
</tbody>
</table>
**Table 5-7:** Young’s modulus and residual stress values for a 1790 nm-thick poly-SiC diaphragm subjected to cyclic loading.

Based on the data presented in the tables above, it is clear to see that the Young’s modulus and residual stress did not vary significantly over the course of the cyclic testing. This data suggests that both the thick poly-SiC films and the submicron-thick single crystal films are stable upon significant mechanical loading.
6.1 Discussion

This thesis involved an investigation to characterize the mechanical properties of submicron-thick, single crystalline cubic silicon carbide (3C-SiC) films using the load-deflection technique on bulk micromachined diaphragms. The films were epitaxially-grown (100) 3C-SiC films and were fashioned into 250 nm and 500 nm-thick suspended diaphragms by silicon bulk micromachining. It was found that the films have residual stresses that are high and tensile (1.21GPa), which affected accurate determination of the Young’s modulus. The durability of these films, as well as 2 µm-thick polycrystalline 3C-SiC films, was investigated by subjecting the structures to cyclic loading. No distinguishable differences were observed in the residual stress and Young’s modulus after 30,000 load-deflection cycles.

The Young’s modulus (in the 700 GPa range) was unexpectedly higher than what was measured on epitaxially-grown, 2 µm-thick, single crystal 3C-SiC films characterized using the same experimental setup. Nanoindentation was used as a means to independently determine the Young’s modulus, but the results were inconclusive due in some measure to the thickness of the samples. As such, nanoindentation was not able to confirm the high Young’s modulus values from the load-deflection measurements.
Mathematical analysis in the form of analytical modeling and finite element modeling suggested that the Young’s modulus values from the load-deflection measurement were affected by the apparently high residual tensile stresses in the diaphragms. The strong linear behavior of the load-deflection data indicates that the residual stress values must be high. Because the residual stress component in the load deflection data come from the small load/small deflection region of the data while the Young’s modulus component is found in the high load/high deflection region, high residual stresses, if they dominate the load-deflection behavior, could make accurate extraction of Young’s modulus very difficult. Analytical modeling and finite element modeling suggest that this might indeed be the case.

The results of the load deflection data as well as the geometric parameters of the test specimens were analyzed using previously published criteria for load-deflection testing of high stress/high modulus diaphragms. The results of this thesis are consistent with this criteria in that the stress levels are too high and the diaphragm areas are too large for accurate extraction of Young’s modulus. This being said, it is believed that the load-deflection measurements for these samples yield an accurate assessment of residual stress.

This thesis also involved a preliminary assessment of diaphragm durability. Diaphragms were subjected to cyclic pressure loading using a custom-built setup. Periodically throughout the loading trials, the diaphragms were evaluated using the standard load-deflection procedure to determine the Young’s modulus and residual stress. No discernable shifts in Young’s modulus (or more correctly some form of diaphragm stiffness since the actual Young’s modulus could not be determined accurately) and residual stress were observed after 30,000 load cycles for ~ 500 nm-thick 3C-SiC and 1790 nm-thick poly-SiC diaphragms. For the single crystal diaphragms, this suggests that the films are extremely
durable given the high stresses found in the films. Likewise the interface bond between the 3C-SiC diaphragms and the underlying Si substrate are very strong. High tensile stress, high durability, submicron 3C-SiC films should find utility in a wide range of MEMS and NEMS applications.

6.2 Future Work

This project has opened the opportunity for significant future work. A few examples of this include repeating the fabrication process of the sub-micron 3C-SiC films to ensure repeatability in the film properties and fabrication methods.

The results gathered here should be verified by other testing methods, such as load deflection of circular [19] or rectangular [11] diaphragms, beam resonance, or electrostatic beam deflection [23], along with many other established measurement methods to determine Young’s modulus and residual stress.
References


