MECHANICAL CHARACTERISATION OF AMORPHOUS SI-BASED THIN FILMS FOR LARGE AREA MEMS

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Abstract

This thesis investigates the mechanical and chemical properties of inductively-coupled plasma enhanced chemical vapour deposited (ICPCVD) a-Si:H and biased target ion-beam deposited (BTIBD) a-Si$_x$Ge$_{1-x}$ thin films and mechanical performance of large area Fabry-Pérot type filters with large tuning range constructed from these films. The thin film properties are studied by means of a variety of thin film testing techniques. The mechanical performances of the designed Fabry-Pérot type filter structures are analysed by means of finite element modeling (FEM) utilising the material properties determined. Based on the outcome of these studies, a tuneable filter with height-suspended membrane of unprecedented large area is realised; compatible with Si(100) substrates and IR imaging arrays operating at cryo temperature, and is actuatatable across a wide temperature range.

For micromechanical design of MEMS devices, a range of mechanical properties of thin film materials are required, including Young’s modulus ($E$), shear modulus ($G$), Poisson ratio ($\nu$), density ($\rho$), pre-release thin film stress ($\sigma$), stress gradient ($\Delta \sigma$), and coefficient of thermal expansion (CTE, $\alpha$). These parameters are determined through a wide range of methods. Thin-film induced substrate bending with varying temperature is used to determine $\alpha$, $\sigma$, and $E$ for the thin film materials as a function of temperature. Specially designed microstructures, such as Guckel rings and microcantilevers, are used to determine $\Delta \sigma$ and to validate the $\alpha$ and $\sigma$ values determined through thin film induced substrate bending for temperatures ranging from 85 K to 300 K. Nanoindentation is another means of determining $E$ for the thin films. This technique is also used to investigate the long-term stability of the thin films undergoing various heat treatments.
Cantilever resonance, which involves the excitation of the bending and torsional vibration modes of a thin film microcantilever, gives a third means for determining $E$ as well as $G$ and $\nu$. Thin film deposition on a quartz crystal microbalance (QCM) is used to determine $\rho$ for the thin films.

The ultimate tensile strength $\sigma_T$ of the deposit films is determined through microbridge rupturing. Specially designed double-necked thin film microbridges are fabricated to rupture within 5 $\mu$m of vertical displacement by a nanoindenter. The tensile strength of a-Si:H and a-Si$_x$Ge$_{1-x}$ shows a slight dependence on composition. However, the dominant factor contributing to the strength of the thin films reported in this work is the nature of the deposition process. The BTIBD films, owing to increased defects compared to the ICPCVD films, show a wide variability in ultimate strength, but a potential for overall improved film strength.

FEM is used in conjunction with thin film induced substrate bending and microstructure deformation to confirm $\alpha$, $\sigma$ and $\Delta\sigma$. FEM input parameters for the deposited films are taken from nanoindentation, thin film induced substrate bending, QCM, and cantilever resonance. Deformation behavior of fabricated microstructures is compared to FEM simulation results confirming a temperature independent $\Delta\sigma$ in cantilever beams and a temperature dependent deformation behavior related to $\sigma$ and $\alpha$ in the Guckel rings. FEM is also used in conjunction with the tensile rupturing experimentation to deduce the ultimate tensile strength of the deposited films. It is found that the microbridge rupturing test gives a conservative estimate of the ultimate tensile strength of the films. Matching the experimental load-displacement curves to those predicted by FEM also bolsters the confidence in the experimentally determined inputs of $E$, $\nu$, $\sigma$, and $\rho$.

For a MEMS device to behave uniformly over a wide range of temperatures, the $\alpha$ of the substrate must closely match with that of the device material. The investigation of material properties reveals that $\alpha$ of the thin films is strongly correlated to deposition method. It is demonstrated that thermal expansion of ICPCVD a-Si is best matched to that of a Si(100) substrate, whereas the reported thermal expansion of BTIBD a-Si$_x$Ge$_{1-x}$ means such films are better suited to
depositions on a Ge or GaAs substrate, regardless of the composition.

The design and analysis of different support and actuation structures of the filter through FEM are also presented in this work. Computational time and accuracy are critical for use of FEM as part of an prefabrication design refinement tool. In this analysis, it is shown that round or curved features, which are computational time-expensive, can be modified to square or rectangular features to significantly reduce run time and improve modeling accuracy. It is also demonstrated that a departure from the "post-and-beam" support and actuation construction for the filter is not only required for proper adhesion of the large area MEMS device to the substrate, but also results in extended actuation range and improved membrane stability and flatness during actuation and cooling. The optimised support and actuation structure allows for the suspended membrane to travel the full span of the initial gap of over 1 \( \mu \text{m} \) without snap-down of the actuating electrodes.

A full scale Fabry-Pérot style filter was fabricated from ICPCVD a-Si:H film on a Si wafer utilising the design optimised through FEM. The structure exceeds 10 mm in dimension. The lateral dimensions of the fully released structure is unprecedented and maintains a flatness of \( \pm 30 \) nm over. The realised device is theoretically actuable across the complete gap, with experimental results demonstrating greater than 50 % actuation.
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Last, but not least, I would like to thank my family–my parents, sisters, dear friends, my fiancé James, and his family–for their amazing love and support.
List of acronyms

MEMS - microelectromechanical systems
MOEMS - micro-opto-electro-mechanical systems
FWHM - full width at half maximum
UV - ultra-violet
NIR - near infrared
SWIR - short-wavelength infrared
LWIR - long-wavelength infrared
FIR - far infrared
CTE - coefficient of thermal expansion
BTIBD - biased-target ion-beam sputter deposition
ICPCVD - inductively-coupled plasma-enhanced chemical vapour deposition
PECVD - plasma-enhanced chemical vapour deposition
PVD - physical vapour deposition
CVD - chemical vapour deposition
RF - radio frequency, high frequency
RIE - reactive ion etching
ICPRIE - inductively-coupled plasma reactive ion etch
FEM - finite element modeling
QCM - quartz crystal microbalance
TC - thermocouple
PID - proportional-integral-derivative
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<tr>
<td>( \sigma )</td>
<td>stress</td>
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<tr>
<td>( \sigma_T )</td>
<td>ultimate tensile strength</td>
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<td>( \varepsilon )</td>
<td>strain</td>
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<td>( S )</td>
<td>compliance</td>
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<td>( C )</td>
<td>stiffness</td>
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<td>nanoindentation stiffness</td>
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<td>( E )</td>
<td>Young’s modulus</td>
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<td>( E_r )</td>
<td>reduced modulus</td>
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<td>( M )</td>
<td>biaxial modulus</td>
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<td>( \kappa )</td>
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<td>length factor for Euler buckling</td>
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<td>( A )</td>
<td>area</td>
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<td>( h )</td>
<td>height, depth</td>
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<td>( f_b )</td>
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<td>( f_t )</td>
<td>torsional resonant frequency</td>
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<td>( f_o )</td>
<td>pre-deposition resonant frequency of QCM</td>
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<tr>
<td>( \rho )</td>
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<td>( V )</td>
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Chapter 1

Introduction

This research is concerned with the development of a microelectromechanical system (MEMS) technology for Fabry-Pérot filters for infrared imaging applications. The study is focused on the development of a novel structure for the actuation of large-area (millimeter scale) MEMS devices capable of operating at low temperatures so as to be compatible with imaging arrays which are often cooled to cryogenic temperatures for maximum performance. The targeted specifications are technologically unprecedented. The accomplishment of the research is expected to make a substantial and original contribution to the scholarship and technology in this field.

1.1 Micro-Electro-Mechanical Systems (MEMS)

A broad historical discussion of MEMS cannot start without mention of the most auspicious discovery and arguably the most important technological achievement to date— the 1947 invention of the transistor at AT&T Bell Laboratories by John Bardeen and Walter Brattain [1, 2]. In the years following, attempts were made at integrating electronic circuits onto single devices with Werner Jacoby patenting the first integrated transistor-amplifier in 1950 [3]. Jack Kilby [4], Kurt Lehovec [5], and Robert Noyce [6] from three different companies are all credited with key advancements during the 1950s and 1960s that enabled commercialisation of the integrated circuit (IC). From this point on, the advancement of the technology has snowballed. Gordon Moore noted in 1965 that the number
of components per IC was increasing 2-fold every 18 months with a concurrent reduction in cost [7]. This trend went on to be famously known as Moore’s Law.

In the wake of the IC boom was the development of various technologies that integrated electronics with mechanics at the micron scale. These devices, now called MEMS, finally entered mainstream consciousness in the late 1980s with a micro-motor developed by researchers at UC Berkeley [8, 9]. At present, MEMS describe a vast and growing body of devices, which include pressure sensors, accelerometers, micro-mirrors, micro-motors, fluid pumps and more.

When MEMS are paired with optical components one often refers to the resulting devices as micro opto-electro-mechanical systems (MOEMS). MOEMS have a wide range of practical applications including sensors that employ optics to detect vibration [10, 11] or acceleration [12–14], optical switches [15–17], and tunable optical filters [18–23]. This work focuses on the actuation and support components of a tunable optical filter.

1.2 Tunable micro-spectrometer

1.2.1 Principles of Fabry-Pérot microspectrometer

The tunable optical filter design employed in this work has a history that extends much further back than MOEMS themselves. In 1899 Pérot and Fabry reported on the phenomenon observed when light waves are reflected between two parallel silvered surfaces of a glass plate, also called an etalon [24]. If, instead of a single transparent plate with two reflecting surfaces, two parallel highly reflecting mirrors are used this structure is termed an "interferometer". In either case, Fabry and Pérot noted that only certain wavelengths of light will resonate within the cavity as illustrated by Figure 1.1.

In a Fabry-Pérot filter, the resonance wavelength is determined by the spacing between the two reflectors. Essentially, wavelengths that resonated in a cavity of length $d$ will be transmitted, while those that do not resonate will not transmit. As illustrated in Figure 1.1, for wavelengths $\lambda = d/n$, where $d$ is the cavity...
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Figure 1.1: Constructive interference of resonant wavelength of light for $n = 1$ and $n = 2$, and destructive interference of non-resonance frequencies in a partially silvered plate etalon. Figure redrawn from Ref. [25, 26]

spacing and $n$ is the wavelength order, constructive interference from repeated reflections results in increased intensity upon transmission, where as destructive interference of non-resonant wavelengths results in dissipation of these wavelengths.

In early spectroscopic methods, the filtered light was recombined through a focusing lens where constructive and destructive interference resulted in the dispersion of different wavelengths/frequencies producing characteristic light fringes and the subsequent measurement of a spectrum as depicted in Figure 1.2 (a) [27]. Today, the light waves are detected by a photo detector, which gathers information on the intensity of the various wavelengths that are being transmitted. Transmission is maximised at these resonant wavelengths and falls off rapidly as you move away from resonant wavelengths creating the spectrum depicted in Figure 1.2 (b).

While metal mirrors, like those used by Fabry and Pérot, are easily fabricated and cost effective, they have high absorption that translates to sacrificing full-width-at-half-maximum (FWHM) and maximum transmission of the filter [28]. An alternative option for the reflector (mirror) is the use of dielectric materials to form a quarter-wavelength distributive Bragg reflector. Using an alternating stack of high and low refractive index materials, all layers being a quarter-
Figure 1.2: Light incident on partially silvered plates with spacing $d$. (a) Transmitted light focused through a focusing lens to produce interference fringes. (b) Transmitted light collected by a photodetector to produce a transmission versus wavelength spectrum. Redrawn from Ref. [26].
wavelength in thickness, a "quarter-wave mirror" stack is realised. Such mirrors can have high reflectivity with very low loss (absorption) over a wavelength band [29]. By adding more and more layers one could theoretically reflect all of the incident light. Such a mirror stack is also known as a distributed Bragg reflector (DBR).

When DBRs forming an optical cavity are moved with respect to each other, the result is a wavelength-tunable Fabry-Pérot optical filter. If the separation of the micro-DBRs is controlled electrostatically (or magnetically), the result is a MEMS tunable Fabry-Pérot filter. Given the high and low refractive indices of the system, \( n_H \) and \( n_L \), the corresponding wavelength band of a DBR can be determined (or the refractive index requirements can be determined from the desired tuning range) via [29, 30]:

\[
\frac{\lambda_+^2 - \lambda_-^2}{4 \lambda_+ \lambda_-} = \Delta g = \frac{2}{\pi} \sin^{-1} \left( \frac{n_H - n_L}{n_H + n_L} \right)
\]

(1.2.1)

where \( \lambda_+ \) and \( \lambda_- \) are the upper and lower wavelengths desired for the tunable range of the mirror, and \( \Delta g \) is the normalised half-width of the desired tuning range. If, for example, it is desired to tune between 1.4 \( \mu \)m to 2.7 \( \mu \)m, \( \Delta g = 0.3525 \). Plotting \( \Delta g \) versus \( n_H/n_L \), as in Figure 1.3, it is shown that this requires \( n_H/n_L > 3.2 \). For the case of the air gap mirror giving \( n_L = 1 \), silicon with a refractive index of \( n_H = 3.44 \) and germanium with refractive index of \( n_H = 4.06 \) would both be suitable.

![Figure 1.3: Normalised half-width of the tuning range, \( \Delta g \), versus the ratio of high and low refractive indices, \( n_H/n_L \).](image_url)
1.2.2 *Current Fabry-Pérot filters*

Key components of various examples of tunable Fabry-Pérot interferometers are highlighted in Figure 1.4. Features ubiquitous to this type of device are the upper mirror, lower mirror, air gap, support features for the upper mirror, and an actuation mechanism. The upper mirror ranges in size from 10s of $\mu$m across to a few millimeters. In some instances the support for the upper mirror is decoupled from the actuation feature, as is the case in Figure 1.4(d). Most often, however, the actuation and support are an integrated feature as in Figure 1.4(a-c). While integration of support and actuation features can create a very stable upper mirror, as was observed for the device in Figure 1.4(c), this design leads to displacement dependent deformation in the upper mirror.

![Figure 1.4](image_url)

*Figure 1.4:* Examples of Tunable Fabry-Pérot interferometer with key design features noted (a) Ref. [21] 1995, (b) Ref. [23] 1998, (c) Ref. [31] 2003, (d) Ref. [32] 2009
Fabry–Pérot filters have applications in industry as far reaching as telecommunications [22, 33–36], meteorology [37, 38], gas sensing [31, 39, 40], medicine [41, 42], and future applications in hyperspectral imaging [43–45]. The final example, hyperspectral imaging, is the end-goal of the larger study for which this work is a part of and is discussed briefly below. Some hyperspectral imaging systems are currently available. It is expected that hyperspectral imaging systems utilising Fabry–Pérot filters will find their way into every day use by the consumer in the form of portable imaging spectrometers for food contamination and spoilage detection [46].

1.2.3 Hyperspectral Imaging

The human eye is an example of a "multispectral" imaging system, detecting light in three bands as shown in Figure 1.5. The difference between multispectral and hyperspectral is the number of bands and the band widths. Just as monochrome or dichrome vision reduces the information available to the viewer compared to RGB colour, we can imagine if we saw many more wavelength divisions how we could gather additional information about our surroundings just by seeing [47]. Hyperspectral imaging is the term given to systems that divide light in to hundreds or thousands of bands. These narrowly defined bands give us the ability to "see" more and collect additional information about an environment. Spectrometer devices on the market are typically categorised based on operating wavelength range as defined in Figure 1.5. These include, visible at 400 nm - 700 nm wavelength, near-infrared (NIR) at 0.75 \( \mu \text{m} \) to 1.4 \( \mu \text{m} \) [48], short-wavelength infrared (SWIR) at 1.4 \( \mu \text{m} \) to 3 \( \mu \text{m} \), mid-wavelength infrared (MWIR) at 3 \( \mu \text{m} \) to 5 \( \mu \text{m} \) [48], long-wavelength infrared (LWIR) at 5 \( \mu \text{m} \) to 15 \( \mu \text{m} \), and far infrared (FIR) at 15 \( \mu \text{m} \) to 1000 \( \mu \text{m} \). Many hyperspectral (and multispectral) imaging devices function across several spectral ranges [49, 50].
Figure 1.5: Classification of wavelengths for visible and infrared with schematic of “multi-spectral” versus “hyper-spectral” imaging.

**1.2.4 Scaling up**

A goal of this research is to enable the scaling up from 0D (single-point) spectroscopy capabilities to 2D imaging without an array of individual spectrometers. Figure 1.6 shows the typical output generated by 0D (point), 1D (line), and 2D (area) spectrometer imaging/detection. A 0D system generates a single spectrum, and is useful when spatial information is not needed, such as gas sensing or testing of homogeneous samples. 1D and 2D spectrometers are both useful in the collection of changing information across a field. 2D filters can be coupled to large area IR imaging arrays [30, 51, 52].

To make the progression from 0D point detection to 2D imaging, one possible solution is to array 0D spectrometers as shown in Figure 1.7 (a) and (b). In the case of arrayed 0D spectrometers, a significant portion of the measurement field is disrupted by support structures. The ratio of optically active area to total pixel area is called the fill factor, and is an indicator of the sensitivity of a sensor. Arrays that utilise the traditional long arm supports as in Figure 1.7(a) or Figure 1.4(a) (b) and (d), fill factor is typically about 20 %, meaning such an array misses up to 80 % of the incident signal. These low fill factor arrays are typical of what is currently available on the market for 2D focal plane arrays used for hyperspectral...
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Figure 1.6: Visual representation of 0D single point spectrum, 1D spectral line scan showing change in peak wavelength with linear position, and 2D area map of spectral peaks showing peak wavelength variation represented by a colour map (image taken from Ref. [53])
imaging [30, 43, 44, 54, 55].

To address the loss of sensing and detection efficiency inherent in low fill factor arrays, Walmsley et. al developed a structure with a fill factor of 79 %, shown schematically in Figure 1.7(b). In this design, the mirror (blue in Figure 1.7) is attached to the supports (red) at a single point (green). However, this design had many instabilities that resulted from mechanical deformation due to thin film stress and lacked the robustness desired by the current project.

Moving away from arrayed spectrometers to single large 2D imaging structures dramatically increases the fill factor, as shown in Figure 1.7 (c), as well as a potential for improved stability of suspended membrane compared to the stability of the design presented by Walmsley [55]. The proposed new design has a fill factor of effectively 100 %, only limited by the fill factor of the IR imaging array optically behind the filter. However, scaling up from 200 µm x 200 µm filters to something with dimensions exceeding 10 mm introduces many challenges, especially when the suspended film is restricted to a thickness of less than 200 nm and a suspended height variation tolerance of less than ±20 nm. Translating this to a macro scale, we can picture suspending a trampoline (5 mm in thickness) over the entire expanse of an Australian rules football field, or three American gridiron fields, maintaining less than ±0.5 mm in height variation across the expanse, while only supporting the structure about the perimeter.

![Figure 1.7: Schematics of (a) array of 200 µm filters with low fill factor, (b) array of 250 µm filters with 80% fill factor, design taken from [55], and (c) proposed large area 2D imaging structure.](image-url)
To maintain an ultra flat suspended membrane, the most common technique is to maintain the structure in tension. Fundamentals of thin film material properties including tensile and compressive stress are covered in Chapter 3. Small structures under tension are easily secured to a substrate, however, scaling up requires unique solutions that can withstand the large tensile stress required to keep a millimeter scale membrane flat without delamination. The effects of residual stress on thin film microstructures and other mechanics of MEMS are covered in Chapter 4. Specialised support structures can also lead to extended actuation and tuning range of the spectrometer. This is covered in detail in Chapter 8.

1.3 Material selection

At present, no single material system exists that is applicable to spectroscopic imaging across the most important bands of the electromagnetic spectrum from ultra-violet (UV) to LWIR. The current dominant material systems are GaN-based for UV [56–58], Si-based for VIS [59, 60], InGaAs-based SWIR, and HgCdTe-based detectors and III-V-based InSb for MWIR/LWIR. A new material set, SiGeC, has been proposed. SiC is functional in the UV, visible, NIR, and SWIR wavelengths. Si is functional in SWIR and MWIR wavelengths. Finally, Ge is functional in the MWIR and LWIR wavelength. As such, a SiGe-based hyperspectral device, offers the unique opportunity for a material system capable of functioning across the electromagnetic spectrum from UV to LWIR [61–65].

The main challenges facing future hyperspectral imaging systems are concerned with complicated device structures and thicker/multilayer material growth. For such applications, it is essential to grow high quality SiGe thin films and have a good understanding of their fundamental mechanical properties. One such feature is thin film stress control with variance in Si and Ge content [66], which gives the opportunity to tune membrane tension.

An additional feature for material selection is the desire to operate movable, or tunable, filters across a wide range of temperatures. The simplest way to maintain form with out deformation is to match the coefficient of thermal expansion (CTE)
of the filter material to the substrate. With Si being a common substrate in MEMS applications, materials are selected which are anticipated to have similar CTEs to crystalline substrates.

Key mechanical and material properties are determined for amorphous silicon in Chapter 7 and for silicon-germanium alloys in Chapter 8. These material properties are used to enable predictive behavior analysis for MEMs structures as demonstrated in Chapter 9.

1.4 Thesis objectives

The goal of this research is to build on previous iterations of a room temperature operated Fabry-Pérot micro-spectrometer. This research is within a large project involving a number of researchers. The project involves research and development in several main areas including processing/fabrication optimisation, material characterisation, device fabrication, optical characterisation, and mechanical characterisation. The main objectives of my research are:

1. characterisation and testing of materials for the proposed devices,
2. designing and modeling of MEMS actuation structures capable of driving the specified (and unprecedented) large-area filter and operating at very low temperatures,
3. characterisation and optimisation of prototype systems fabricated based on the designs for proof of concept.

The details of these three objectives are as follows:

1. Material properties characterisation: The MEMS structures to be developed are constructed of thin film materials primarily consisting of deposited Si, Si$_x$Ge$_{1-x}$, and oxides. These thin film materials, owing to their very small physical dimensions, unique microstructures, and the formation mechanisms associated with thin film depositions, can exhibit very different mechanical properties from their bulk counterpart. To be able to use these ma-
terials in MEMS designs, it is essential to fully understand their properties, structures and responses to variations in application conditions and exposure to the ambient. The particular tasks will include the characterisation and determination of:

a. Physical and mechanical properties: Young’s modulus, Poisson’s ratio, thermal expansion coefficient, yield strength, creep and fracture resistance;

b. Thin film material status: physical, chemical or structural defects, residual stresses and stress gradients, effect of temperature on residual stress and stress gradients; effect of fabrication conditions on film status and properties

c. Environmental stability of thin films: effect of oxidation, effect of temperature and effect of moisture

d. Chemical structure: film composition stoichiometry and variations, stability of chemical makeup of the films

II. Design and modeling of devices

a. Design and simulate the mechanical performance of model MEMS structures: to investigate the effects of film stress, temperature, thermal cycling, vibration; to characterise the static force-displacement relationships, snap-down conditions

b. Examine the mechanical performance of the devices in terms of
   i. Mechanical force displacement
   ii. Vibrational Modes
   iii. Electrostatic displacement (actuation, pull-in, and lift-off voltage)
   iv. Thermal stability of structure and actuation curves
   v. Residual stress

III. Fabrication and Characterisation of prototype device

a. Mask design and device realisation

b. Mechanical performance of the devices
i. Low temperature actuation and transmission spectrum
ii. Specialised test structures for stress and strain calculations

The tangible and measurable outcome of the proposed research is to extend upon the performance demonstrated in previously reported iterations of the device structures through:

- Increasing the tuning range via utilising material stress and specialised device structure.
- Increase fill factor via large area mirror structures and well designed novel actuation and supports
- Develop a robust knowledge/database of material properties at low temperatures.
- Develop finite element models to understand mechanical behavior in the system across a large range of temperatures
- Develop a large area device with large wavelength tuning range (greater than $\lambda = 1.615 \, \mu m$ to $\lambda = 2.425 \, \mu m$) that can be stably actuated over a large range of temperatures.

1.5 List of publications

1.5.1 Publications resulting from this work


### 1.5.2 Publications related to the work


1.5.3 *Patents pending related to this work*

2.1 Thin film deposition techniques

Deposition methods of thin films falls into one of three classes: purely chemical, purely physical, and the intermediary physical-chemical method. These techniques are further divided into evaporative, glow-discharge, gas-phase chemical, and liquid-phase chemical. Specific techniques used in this thesis are: (1) biased-target ion-beam sputter deposition (BTIBD) 2.1.1, and (2) inductively-coupled plasma-enhanced chemical vapour deposition (ICPCVD) 2.1.2.

2.1.1 Biased-target ion-beam sputter deposition (BTIBD)

BTIBD falls into a deposition class termed physical vapour deposition (PVD), in which films are deposited via purely physical mechanisms in a vacuum environment [67]. The basics of deposition can be broken down into four stages: 1) formation of the vapour, 2) transport of atoms or molecules to a substrate, 3) deposition on the substrate, and 4) nucleation and growth of films on the substrate.

BTIBD is the technique used to deposit a-Si$_x$Ge$_{1-x}$ thin films in this thesis. This technique utilises an ion beam source (typically called an ion gun), which produces positive ions from an inert gas such as Ar. These ions are attracted to a negatively biased bulk material "target", ejecting or "sputtering" atoms from the target. Figure 2.1 shows a schematic of a multi-target BTIBD set up with biased targets, ion source, and substrate.
Figure 2.1: Schematic of a 6-target biased-target ion-beam deposition (BTIBD) system.

Sputtering is a process by which an atom of molecule is ejected from a bulk material when the surface is struck by a fast incident particle [68]. Figure 2.2 shows a schematic of the process of sputtering. To efficiently knock-off target atoms or molecules, the incident particle needs to be of similar size to the desired ejection particle. An electron, for instance, would be much too small and not carry enough momentum to successfully eject a target atom. On the other hand, if the object is too large it will not be able to interact with the surface molecules that are to be ejected. As such, the typical source of fast incident particles are ions.

Ions needed for sputtering come from a plasma which is either produced via a direct electrical current (DC) or a high frequency alternating current (RF). The plasma is either produced within the chamber and referred to as the "glow discharge", or sequestered to an ion gun. For plasmas produced within the chamber, the ionising gasses flood the chamber and are ionised by the biased electrical current, typically located at the target. This deposition technique involves increased
Chapter 2: MEMS Fabrication

Figure 2.2: Schematic of the sputtering process redrawn from Ref. [68] showing the collision process for (a) implantation of a bombardment ion and (b) ejection (sputtering) of a target atom.

Chamber pressures compared to ion gun sputtering techniques and requires careful consideration of mean-free-path of the target particles moving through the chamber to maintain sufficient deposition rates. In contrast to a plasma plume, using an ion-beam source allows for low chamber pressure as the ions are produced within the ion gun requiring only a localised volume of ionising gasses. This method allows for much greater control through directed ion bombardment.

Biased-targets are used in addition to the ion beam to accelerate the ions toward the target. With plasma, the ions produced are not sufficiently energetic to sputter atoms from a target. A biased target increases the momentum of the incident ions which leads to greater ejection rates. Biasing a target for deposition also acts as a "getter" for the ions; attracting the ions to the target, thereby reducing ion bombardment of undesired materials that may be present elsewhere in the chamber.
2.1.2 Inductively-coupled plasma-enhanced chemical vapour deposition (ICPECVD)

ICPECVD falls into the deposition class of chemical vapour deposition (CVD). CVD relies on reacting volatile compounds to create a non-volatile solid deposit [69]. This is the main technique used to deposit a-Si:H thin films reported in this thesis. The CVD reactions necessary to precipitate the desired solid deposit can be classified as pyrolysis, reduction, oxidation, or compound formation. In the case of a-Si:H, the driving mechanism is pyrolysis which involves the thermal decomposition of the gaseous species on a hot substrate [70].

\[
\text{SiH}_4(g) \rightarrow \text{Si}(s) + 2\text{H}_2(g)
\]  
(2.1.1)

for SiO\(_2\), the driving chemical reaction is oxidation.

\[
\text{SiH}_4(g) + 2\text{N}_2\text{O}(g) \rightarrow \text{SiO}_2(s) + 2\text{H}_2(g) + 2\text{N}_2(g)
\]  
(2.1.2)

However, these reactions require elevated temperatures of 650 °C and 450 °C, respectively. These elevated temperatures are not compatible with temperature sensitive substrates or temperature sensitive processing steps.

The solution to this compatibility issue is plasma-enhanced CVD (PECVD). In PECVD processing, glow-discharge plasmas are excited by an RF field. In a low pressure environment of 0.5 to 5 Torr, electron and positive ion densities of the plasma range between \(10^9\) and \(10^{11}\) cm\(^{-3}\) with average electron energies ranging from 1 to 10 eV [69, 71]. This high energy plasma transfers energy to the reactant gasses, resulting in dissociations and promoting surface reactions at significantly reduced temperatures. The reaction steps that occur during PECVD processing (shown in Figure 2.3) are [69–71]:

1. electron impact ionisation and dissociation of the source gasses to create film precursors (for example Equations 2.1.3 and 2.1.4)

2. transport of reactants and products to the substrate
3. adsorption of film precursors to substrate surface (or reabsorption of precursors); surface reactions

4. heterogenous nucleation and film growth

5. desorption of volatile bi-products

For a-Si:H the ionised species created from the input gases SiH\textsubscript{4} and He are:

\[
\begin{align*}
\text{Ionisation} & : \text{SiH}_4 \xrightarrow{\text{energy}} \text{SiH}_n^+ + (4-n)\text{H} + e^- , \quad n = 1, 2, 3, 4 \\
\text{He} & \xrightarrow{\text{energy}} \text{He}^+ + e^- \\
\text{Dissociation} & : \text{SiH}_4 \xrightarrow{\text{energy}} \text{SiH}_n + (4-n)\text{H}, \quad n = 1, 2, 3, 4 \\
\end{align*}
\]

\[(2.1.3)\]

Figure 2.3: Sequence of ICPCVD reaction steps. Redrawn from Ref. [69, 71].
For SiOₓ, the ionised species created in the plasma from the input gases SiH₄, He, Ar, and N₂O (in addition to the ones listed above for a-Si:H formation) are:

\[
\text{Ionisation} \quad \text{N}_2\text{O} \xrightarrow{\text{energy}} \text{N}_n\text{O}^+_m + \text{N}_{(2-n)\text{O}}^{(1-m)} + e^- , \quad n = 0, 1, 2 \quad m = 0, 1
\]

\[
\text{Ar} \xrightarrow{\text{energy}} \text{Ar}^+ + e^-
\]

\[
\text{Dissociation} \quad \text{N}_2\text{O} \xrightarrow{\text{energy}} \text{N}_n\text{O}^+_m + \text{N}_{(2-n)\text{O}}^{(1-m)} , \quad n = 0, 1, 2 \quad m = 0, 1
\]  

(2.1.4)

In the synthesis of a-Si:H (Equation 2.1.3) the most common Si ions and neutral species that deposit on the substrate surface are SiH₄⁺ and SiH₃. Surface reactions with dangling Si bonds result in the subsequent release of hydrogen and the formation of a-Si:H films [70, 72]. The addition of inert gasses like He, Ar, and Xe works to increase the energy of the ions impinging on the substrate and have been shown to reduce film disorder and defect density [73, 74]. The implanted hydrogen present in the film comes from the incomplete dissociation of the SiH₄ gas as well as fully dissociated atomic hydrogen. The latter is noted as the dominant hydrogen species in a-Si:H thin films, desorption of which begins at annealing temperatures as low as 300 °C [75].

To produce a high frequency (RF) discharge plasma, the electrode impulse stimulus must be coupled to the gas that is to be excited into a plasma. This can be done through capacitive coupling or inductive coupling. With inductively coupled plasmas (ICP), the plasma energy is supplied by an electromagnetic current produced from RF-current being passed through an electric coil. Figure 2.4 shows a schematic of the ICP unit and the ICPCVD chamber.
2.1.3 Nucleation and Growth

When deposition species impinge on a substrate, the adsorption occurs as either physisorption or chemisorption. PVD relies predominantly on physisorption where impinging particles adhere to the surface via van der Waals forces. CVD relies on chemisorption, as described in section 2.1.2, where by the impinging particle chemically bonds to atoms on the substrate surface. In either case, the subsequent thin film growth is governed by heterogeneous nucleation, or the formation of a new phase on the surface of another material [76]. Heterogeneous nucleation of PVD and CVD films is most often triggered by a decrease in the temperature of deposit, which leads to vapour-phase condensation, solidification, or solid-state phase transformations [69].

Figure 2.5 shows a schematic of the two growth modes observed for PVD and CVD. For both PVD and CVD we can assume growth of a species in the vapour...
Figure 2.5: Growth modes of vapour deposited thin films. (a) Island growth "Volmer-Weber mode" and (b) layer-by-layer "Frank-van der Merwe mode". (Dots do not necessarily represent an ordered structure, as would be the case with molecular beam epitaxy)

When the vapour particles impinge on the substrate the thin film growth mode depends on the surface energies of the vapour deposit and substrate [77]. If the vapour surface energy is lower than the wafer, the vapour will fully "wet" the surface producing layer-by-layer growth. If the vapour surface energy is higher than the wafer, the vapour will form growth islands. Si films deposited on oxides most typically grow via islands (or Volmer-Weber growth) and low temperature deposits are amorphous in structure [69]. However, layer-by-layer (Frank-van der Merwe) growth has been achieved through high temperature CVD, giving rise to epitaxial growth of crystalline silicon [68].

2.2 Micromachining

Micromachining is the term given for the processing steps used to create MEMS and other microelectronic and micromechanical devices. It is the umbrella term encompassing patterning, material deposition, and etching. The fabrication of MEMS can be divided into either bulk micromachining or surface micromachining. Though the dividing line between bulk micromachining or surface micromachining is often blurred, the terms as used throughout the experimental chapters
CHAPTER 2: MEMS FABRICATION

will be described below.

2.2.1 Photolithography

The starting point for bulk or surface micromachining is to deposit a masking layer that protects areas that are not to be etched. This step is called patterning. Various masking techniques exist, but the most popular of them is photolithography. With contact photolithography (the method used for masking in this thesis), a photo reactive material (photoresist) is deposited on to the wafer, a graphic mask is placed over and in direct contact with the deposited photoresist, and high intensity UV light is used to transfer the graphic mask image into the photoresist. Figure 2.6 shows the micromachining steps for creating a fully released cantilever, which are described below.

2.2.2 Bulk and surface micromachining

Bulk micromachining refers to the process of bulk substrate removal through various etching techniques. The most common substrate material in MEMS is crystalline Si, with bulk micromachining of Si employed in the fabrication of pressure sensors [78], shape memory structures [79], and released membranes for material testing [80].

Surface micromachining is the process of adding or removing thin film or surface deposits. Similar methods to those employed by bulk micromachining are also used during surface micromachining. These include the methods describe in wet etching (section 2.2.2.1) and dry etching (section 2.2.2.2). Unlike bulk micromachining which involves the etching of substrate wafers, surface micromachining also involves the layer-by-layer deposition and patterning of thin films on the surface of a wafer [81].

The following sections detail wet and dry etching techniques used in bulk micromachining of substrate wafers and surface micromachining of thin film. These steps are illustrated schematically in Figure 2.6 for reference.
Figure 2.6: Process for machining of a fully released cantilever. (1) deposition of photoresist on prepared wafer (2) transfer of graphic mask to photoresist layer (3) removal of exposed resist (4) etching through a mask the deposited thin film (5) removal of remaining resist (6) etching through a mask to release of the cantilever detailed further in Figure 5.2.
2.2.2.1 *Wet Etching*

The most common form for the bulk micromachining of Si is wet etching. This treatment is performed by masking areas of the wafer with an etch resistant material and then exposing the unprotected areas of the wafer to a liquid etchant. Depending on the chemistry, a wet etch can either be isotropic, meaning etching at the same rate in all directions, or anisotropic, meaning preferential etching in a particular direction.

Figure 2.7 highlights the difference between isotropic and anisotropic characteristic of Si(100) wafers. For the isotropic case (Figure 2.7 (a)) we see the etch front propagating evenly in all directions and the resultant under-etch at the Si(100)-thin film interface. For the anisotropic case (Figure 2.7(b)) we see that the Si(100) wafer resists etching in the (111) crystal plane with preferential etching of the (100) planes. As shown in the picture, masking width will determine etch depth in this configuration of anisotropic etching.

*Figure 2.7:* Etching of Si(100) for the (a) isotropic case and (b) anisotropic case.
A major draw back to wet etching in terms of this thesis, however, is the fact that any etchant that etches Si(100) wafer, will also etch an a-Si:H or Si$_x$Ge$_{1-x}$ thin film. Dry etching techniques provide a solution to this, with greater ability to control sample exposure to the etchant.

### 2.2.2.2 Dry Etching

Dry etching, as is implied by the name, uses gaseous etchants instead of liquid. As with wet etching, dry etching occurs as either isotropic or anisotropic etch, but the anisotropy is not solely controlled by crystallographic plane preferential etching. Dry bulk micromachining has been used for backside release of suspended membranes for bulge testing [82], as a processing step in the fabrication of silica waveguides [83, 84], and for the development of high aspect ratio Si nanopillars for single electron transport [85]. In addition to easier preservation of the deposited a-Si$_x$Ge$_{1-x}$ films, dry bulk micromachining allows for greater aspect ratios in Si(100) wafers than what can be achieved with wet etching techniques [86].

The most common means of dry etching is plasma etching. As with the plasmas in CVD and PVD systems, etchant plasmas are generated through RF electric fields applied between two electrodes. The mechanism that results in plasma etching can be chemical, purely physical, or a combination chemical-physical depending on the various operational parameters.

For a purely chemical plasma etch, the etching directional characteristics are quite similar to wet etching. Depending on the chemistry of the gas the etch will either be isotropic or anisotropic as shown in Figure 2.7, with the anisotropic etch being controlled by the same preferential orientation etching as described in wet etching. For a purely mechanical plasma etch, the material is removed through sputtering. With this technique, ions accelerated by the plasma strike the surface of the wafer taking some of the wafer material with it. This etch is also highly anisotropic, etching primarily in the direction of ion flux. However, a purely mechanical etch is not material selective. If it is desired to stop at a specific point (as is the case for release of thin membranes and microstructures), the etch must be
carefully timed or a combination of chemical and physical etch processes must be used.

Reactive ion etching (RIE), considered a type of dry etching, uses chemically reactive plasmas to remove substrate or thin film material. Using a technique originally developed by researchers at Robert Bosch GmbH [87] of varying the gas chemistry of the etch, it is possible to alternate between sidewall passivation and deep wafer etching [88, 89]. As a result, vertical sidewalls and large aspect ratios can be achieved for the backside release of MEMS [90].
CHAPTER 3

Material properties of thin films and their substrates

While a great deal is known about the material properties of bulk materials, there is a continued need for the characterisation of materials in their thin-film form. For semiconductor thin films, most characterisation focuses on their electrical properties [91]. With current technologies, such thin film materials are used as mechanical components in MEMS devices. As such, their mechanical properties must be evaluated as they can differ greatly from those of their bulk counterparts [92–96]. Differences in the properties between bulk and thin film materials arise due to many different reasons, but mainly size, composition, and processing conditions.

Size differences between bulk and thin films relate to an increase in the ratio of surface area to volume with a decrease in dimension. A reduction in dimension has shown to result in increased strength as is the case observed in nanowires due to the elimination of defects with in the structure [52, 95]. However, reduction in dimension can also negatively impact material performance as compared to bulk [92]. Such an example would be the observation of reduced tensile strength in thin films compared to bulk due to process induced defects [97].

Composition, in the terms of material phase and microstructure, often differs between bulk and thin film. These differences are largely due to formation/deposition techniques being vastly different for thin films compared to bulk and has been shown to effect the material properties [92, 94, 96]. Additionally,
composition variants unique to thin films, such as processing gas interstitials and inclusions, can alter elastic constants, stress, and thermal expansion [98–105].

In addition to deposition, any post deposition processing of the thin film, such as photolithography and etching, used to create the MEMS microstructures can alter the final material properties. Such processing steps are prone to inducing local areas of concentrated surface defects that reduce the ultimate strength and alter the mechanical performance of the thin film [106–109].

As the characteristic material (mechanical, thermal, and electrical) properties of a system are explored, the interrelatedness of the properties are revealed. Originally drawn by G. Heckmann and later published by Nye in Ref. [110], Figure 3.1 is an interesting diagram that shows the multifunctional relationships that exist between electrical, thermal, and mechanical properties. The primary ‘forces’ applied to the crystals and corresponding to the three areas of material properties are temperature, electric field, and stress with the direct results of these forces (or

Figure 3.1: The interrelatedness of mechanical, electrical, and thermal properties in materials. Numbers in [] and () show the tensor rank of the property. Material properties covered in this thesis are highlighted. Redrawn from Ref. [110].
principal effects) being entropy, displacement, and strain. When a primary ‘force’ is applied to a material, what Nye refers to as ‘coupled effects’ occur. One such example is the application of temperature resulting in a strain in the material, the effect being known as thermal expansion. This thesis will explore the properties highlighted in the lower portion of the diagram for the SiGe material system including: stress, strain, elasticity, and thermal expansion coefficient. Additionally, the effect of displacement due to electric field is utilised, but not explored fundamentally, as this has been covered extensively by others [111–120].

3.1 Single crystal wafers

The wafer substrates used in this study are single crystal Si(100) and Ge(100) wafers. These single crystal wafers are the most commonly used substrates for thin film deposition and MEMS device fabrication. The crystal structure of Si and Ge, which have identical crystal structure but different lattice constants, are shown in Figure 3.2 (a). The naming conventions for the Miller indices and directions used in this thesis are also included in the figure.

These wafers, Si(100) and Ge(100), are cut such that the top surface of the wafer is the (100) crystal plane with the [100] direction normal to this surface. To identify the in-plane orientation, a wafer is cut with a straight edge, which is usually in the (110) plane for a Si(100) wafer. Figure 3.2 (b) shows the planes and direction for a (100) oriented wafer. The x and y directions defined by the fore-cut flat are in the <110> directions while the 45° diagonals are in the <100> directions.

The directionality of crystals plays a large role in their material properties. While amorphous thin films are generally described as isotropic (having the same characteristics in any direction) [121], crystalline materials can be highly anisotropic (having different characteristics dependent upon the crystallographic direction) [122]. As such, it is important to take careful consideration of the placement of microstructures as well the orientation relative to the substrate for which material properties of thin films are measured. The following sections will outline material properties for both the isotropic (thin film) and anisotropic (substrate) cases.
CHAPTER 3: MATERIAL PROPERTIES OF THIN FILMS AND THEIR SUBSTRATES

3.2 Mechanical properties

3.2.1 Elasticity

When a sample is placed under a load there are two types of deformation that can occur: elastic and plastic. Elastic deformation is a reversible behavior and corresponds to the linear region of the stress strain curves in Figure 3.3. Plastic deformation occurs after the elastic region has been passed (beyond the yield point), after which permanent deformation remains even after the load is removed. When loaded under tension at room temperature, thin film semiconductor materials like Si and Ge demonstrate brittle fracture with no plastic deformation [96, 123–125].
The relationship between the stress $\sigma$ and strain $\varepsilon$ of a material is described by Hooke’s law:

$$\sigma = C\varepsilon$$  \hspace{1cm} (3.2.1)

$$\varepsilon = S\sigma$$

where $S$ is the material compliance and $C$ is the material stiffness. In many cases these parameters are directionally dependent. As such, for an anisotropic material with cubic symmetry, Hooke’s law is written as:

$$\sigma_i = c_{ij}\varepsilon_j$$  \hspace{1cm} (3.2.2)

$$\varepsilon_i = s_{ij}\sigma_j$$

where the subscripts $i$ and $j$ describe the orientation of the face and vector according to Table 3.1 [110, 127–129]. Where $\sigma_{xx} = \sigma_1$ refers to the stress in the x-direction, and $\sigma_{yz} = \sigma_{zy} = \sigma_4$ refers to a shear stress in the yz-plane.

<table>
<thead>
<tr>
<th>Direction</th>
<th>xx</th>
<th>yy</th>
<th>zz</th>
<th>yz/zy</th>
<th>zx/xz</th>
<th>xy/yx</th>
</tr>
</thead>
<tbody>
<tr>
<td>Numerical Equivalent</td>
<td>11</td>
<td>22</td>
<td>33</td>
<td>23/32</td>
<td>31/13</td>
<td>12/21</td>
</tr>
<tr>
<td>Reduced Numerical Equivalent</td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
</tr>
</tbody>
</table>
Due to symmetry in the Si and Ge crystals, the compliance and stiffness coefficients can be written as:

\[
\begin{align*}
\mathbf{s}_{ij} &= 
\begin{bmatrix}
  s_{11} & s_{12} & 0 & 0 & 0 \\
  s_{12} & s_{11} & s_{12} & 0 & 0 \\
  s_{12} & s_{12} & s_{11} & 0 & 0 \\
  0 & 0 & 0 & s_{44} & 0 \\
  0 & 0 & 0 & 0 & s_{44} \\
  0 & 0 & 0 & 0 & 0 & s_{44}
\end{bmatrix} \\
\mathbf{c}_{ij} &= 
\begin{bmatrix}
  c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\
  c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\
  c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\
  0 & 0 & 0 & c_{44} & 0 & 0 \\
  0 & 0 & 0 & 0 & c_{44} & 0 \\
  0 & 0 & 0 & 0 & 0 & c_{44}
\end{bmatrix}
\end{align*}
\]

(3.2.3)

For the isotropic case, \(s_{44}\) and \(c_{44}\) can be rewritten as:

\[
\begin{align*}
  s_{44} &= 2(s_{11} - s_{12}) \\
  c_{44} &= 2(c_{11} - c_{12})
\end{align*}
\]

(3.2.4)

Using Equation 3.2.2 with the tensor matrix from Equation 3.2.3 we can get an equation for the stress in the x-direction:

\[
\sigma_1 = c_{11}\varepsilon_1 + c_{12}\varepsilon_2 + c_{12}\varepsilon_3
\]

(3.2.5)

which tells us stress in x (\(\sigma_1\)) is a function of the strain in the three orthogonal directions x, y, and z (\(\varepsilon_1, \varepsilon_2, \varepsilon_3\)). Given the elastic constants for Si and Ge in Table 3.2, we solve a set of simultaneous equations similar to Equation 3.2.5, and the relationship of stress to strain of the material is determined. These relationships
Table 3.2: Elastic constants for Si and Ge at 298 K

<table>
<thead>
<tr>
<th>Material</th>
<th>( c_{11} )</th>
<th>( c_{12} )</th>
<th>( c_{44} )</th>
<th>( s_{11} )</th>
<th>( s_{12} )</th>
<th>( s_{44} )</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon</td>
<td>165.7</td>
<td>63.9</td>
<td>79.6</td>
<td>7.68</td>
<td>-2.14</td>
<td>12.6</td>
<td>[127, 128]</td>
</tr>
<tr>
<td>Germanium</td>
<td>129.2</td>
<td>47.9</td>
<td>67.0</td>
<td>9.64</td>
<td>-2.6</td>
<td>14.9</td>
<td>[128]</td>
</tr>
</tbody>
</table>

give us the elastic properties of the material such as poisson’s ratio \( (\nu) \), Young’s modulus \( (E) \), and the Shear modulus \( (G) \).

One caveat does exist. Equation 3.2.3 is in a form that gives us the elastic properties in the [100] direction of Si and Ge. As discussed earlier, this is the out-of-plane direction, but we require the in-plane [110] elastic properties of the substrate and subsequent in plane properties of the isotropic thin films. To achieve this, the tensor matrices in Equation 3.2.3 must be rotated towards the [110] direction. Such a transformation quickly becomes mathematically tedious, therefore results are computer generated [110, 127–130]. The computer generated transforms for elastic modulus, poisson’s ratio, and shear modulus are discussed in the following sections.

### 3.2.1.1 Elastic Modulus

When a material is deformed elastically, the slope of the linear region of the stress strain curve (Figure 3.3), is the Young’s modulus, \( E \). In an isotropic material like a-Si, Young’s modulus fully describes the uniaxial stiffness of the material. As such, the full uniaxial isotropic equation for Hooke’s law (Equation 3.2.1) becomes [127]:

\[
\sigma = E\epsilon
\]  

(3.2.6)

For the case of an anisotropic material such as crystalline Si and Ge, the Young’s modulus is computationally derived from the rotation of the anisotropic tensor (Equation 3.2.3) as discussed above. Figure 3.4 (a) shows the Young’s modulus as a function of direction in the (100) plane. From the figure we see that for the [110] direction, the Young’s modulus is 169 GPa for Si and 138 GPa for Ge.
CHAPhTER 3: MATERIAL PROPERTIES OF THIN FILMS AND THEIR SUBSTRATES

Figure 3.4: (a) Young’s modulus in the (100) plane for Si and Ge. From Ref. [127, 128]. (b) Shear modulus for Si and Ge (100) crystals with $i$ fixed in the [110] direction and $j$ rotating in the (110) plane. $G_{yz}$, or $i$=[110] and $j$=[001] is noted in the figure for reference. Redrawn from Ref. [128].

The Young’s modulus is anisotropic within the plane of Si(100) and Ge(100) wafers. However, there is a symmetric boundary condition along the orthogonal axes in the (100) plane and is often referred to as having "orthogonal anisotropy". This symmetry allows for the behavior of the wafer to be described by the biaxial modulus, $M$, given by [127]:

$$M_{100} = c_{11} + c_{12} + \frac{c_{12}^2}{c_{11}}$$  \hspace{1cm} (3.2.7)

Using the values from Table 3.2, $M_{100} = 180$ GPa for Si and $M_{100} = 142$ GPa for Ge.

Due to the variable nature of thin film deposition techniques, there is a great deal of variation in reported values for Young’s modulus of amorphous silicon and germanium [98, 102, 103, 131, 132]. The use of hydrogen containing gas mixtures is a common as a precursor for various CVD and PVD deposition techniques. The inclusion of bonded and unbonded hydrogen molecules within the a-Si and a-Ge lattices has been shown (in controlled single experiments) to result
in a reduced Young’s modulus with increasing hydrogen content [98, 103]. Table 3.3 gives the Young’s modulus values reported for hydrogen containing a-Si and a-Ge thin films. In the studies that included both a-Si and a-Ge deposited under similar conditions, the Young’s modulus of a-Si was demonstrated to be greater than that of a-Ge [102, 132].

| Table 3.3: Literature values of elastic modulus, $E$, for thin film Si and Ge |
|-----------------|-----------------|-----------------|-----------------|
| $E$ (GPa)       | Hydrogen (at. %) | deposition method | Ref.           |
| 100             | <0.1            | rf sputtering    | [98]           |
| 65              | 1               | rf sputtering    | [98]           |
| 130             | –               | rf sputtering    | [102]          |
| 150             | 20              | PECVD            | [131]          |
| 140             | –               | ion-beam sputtered | [132]       |
| a-Ge            | 83              | –               | rf sputtering    | [102]          |
| 110             | variable 4 - 20 | PECVD            | [103]          |
| 120             | –               | ion-beam sputtered | [132]       |

3.2.1.2 Shear Modulus

Shear Modulus, $G$, also called the modulus of rigidity, defines the response of a body to a shear stress, $\tau$. The applied shear stress will result in elongation, or strain ($\gamma$), in the two opposing directions. Just as Hooke’s law (Equation 3.2.1) relates normal stress to strain, the relation of $\tau$ to $\gamma$ in an isotropic sample is given as:

$$\tau = G\gamma$$  \hspace{1cm} (3.2.8)

Shear modulus is given as a function of the Young’s modulus and Poisson ratio ($\nu$ described in detail in Section 3.2.1.3) in an isotropic sample via [110]:

$$G = \frac{E}{2(1+\nu)}.$$  \hspace{1cm} (3.2.9)

In an anisotropic crystal, shear modulus is given by [110, 127]:

39
Figure 3.4 (b) shows the shear modulus of Si (100) and Ge (100) crystals for \( i \) fixed in the [110] direction and \( j \) varying in the plane. For a long narrow beam oriented with the long axis in the [110] direction and shearing occurring in the [001] direction (twisting of a beam that lies in the plane of the wafer), we can see from the figure that \( G_{yz} = 80 \text{ GPa} \) for Si, and 65 GPa for Ge. None of the material property testing of the a-Si:H and a-Si\(_x\)Ge\(_{1-x}\) thin films in this thesis require consideration of the substrate’s shear modulus as any testing of shearing in this thesis involves fully released thin films. However, there are no literature reports on the shear modulus of a-Si or a-Ge thin films and as such, bulk crystalline values give the best indication of expected values for the thin films.

### 3.2.1.3 Poisson Ratio

Generally, a material deformed elastically in compression or tension will have a corresponding dimensional change that is orthogonal to the direction of applied stress. The orthogonal length change is described by Poisson ratio, \( \nu \). In an isotropic material, it is most often the case that a compressive load applied in the \( x \)-direction will have a negative strain in the \( x \)-direction and corresponding positive strain in the \( y \)- and \( z \)-directions. If instead a tensile load is applied, there will be a positive strain in the \( x \)-direction and a negative strain in the \( y \)- and \( z \)-directions. As such, Poisson ratio in an isotropic material is written as:

\[
\nu = \frac{-\varepsilon_i}{\varepsilon_{jk}}
\]

where \( i, j, \) and \( k \) correspond to the three orthogonal directions \( x, y, \) and \( z \) in any combination. For a-Si this value is in the range of 0.2 to 0.3. Table 3.4 lists published values of Poisson ratio for a-Si thin films from various deposition methods. Poisson ratio for amorphous Germanium is not reported in the literature. A paper reporting on the poisson ratio in epitaxially grown thin film Ge is listed in Table 3.4.
As with Young’s modulus, for orthogonal anisotropic Si(100) and Ge(100), Poisson ratio can be computed from the rotated tensor equation. When describing anisotropic materials, Poisson ratio is given two subscripts ($\nu_{ij}$) with the convention that $i$ is the direction of applied extension and $j$ is the corresponding direction of contraction. For the purpose of this thesis, we are curious about an extension in the [110] direction and the corresponding in-plane orthogonal ([110] and [110]) contractions as well as the out-of-plane orthogonal ([100] and [100]) contractions.

Figure 3.5 has two plots: (a) showing the Poisson ratio for $i$ fixed at [110] and $j$ rotating in the (110) plane giving $\nu_{xz}$ and $\nu_{xy}$ and (b) for $i$ and $j$ both fixed in the (110) giving $\nu_{zy}$ and $\nu_{yz}$. The key values of published Poisson ratio for crystalline Si and Ge are listed in Table 3.5 [127, 128].

### Table 3.4: Literature values of Poisson ratio, $\nu$, for thin films of Si

<table>
<thead>
<tr>
<th>$\nu$</th>
<th>form</th>
<th>deposition method</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.22</td>
<td>amorphous Si</td>
<td>none listed</td>
<td>[133]</td>
</tr>
<tr>
<td>0.2-0.24</td>
<td>amorphous Si</td>
<td>ICPCVD</td>
<td>[134]</td>
</tr>
<tr>
<td>0.23-0.32</td>
<td>amorphous Si</td>
<td>laser CVD</td>
<td>[134]</td>
</tr>
<tr>
<td>0.3</td>
<td>nanocrystalline Si</td>
<td>ICPCVD</td>
<td>[135]</td>
</tr>
<tr>
<td>0.22</td>
<td>polycrystalline Si</td>
<td>LPCVD</td>
<td>[136]</td>
</tr>
<tr>
<td>0.25</td>
<td>amorphous Si</td>
<td>ICPCVD and laser CVD</td>
<td>[137]</td>
</tr>
<tr>
<td>0.25</td>
<td>epitaxial Ge</td>
<td>LPCVD</td>
<td>[138]</td>
</tr>
</tbody>
</table>

### Table 3.5: Poisson ratio for (100) oriented Si and Ge wafers

<table>
<thead>
<tr>
<th>$i$</th>
<th>$j$</th>
<th>Si</th>
<th>Ge</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\nu_{yz}$</td>
<td>0.36</td>
<td>0.36</td>
<td>[1-10]</td>
</tr>
<tr>
<td>$\nu_{xz}$</td>
<td>0.36</td>
<td>0.36</td>
<td>[110]</td>
</tr>
<tr>
<td>$\nu_{zy}$</td>
<td>0.28</td>
<td>0.26</td>
<td>[001]</td>
</tr>
<tr>
<td>$\nu_{xy}$</td>
<td>0.064</td>
<td>0.022</td>
<td>[110]</td>
</tr>
</tbody>
</table>

*see Figure 3.2 for orientation directions on a Si(100) wafer*
3.2.2 Strength of Si and Ge

Literature reports of tensile strength focus on single-crystal silicon [139, 140], polycrystalline silicon [141–144], and silicon nitride and carbide films [80, 145–147]. Comparatively little has been reported on the strength of germanium based materials, with reports focusing on the strength benefits of Ge in Si wafers [148, 149] and Ge nanowires [96, 123]. Additionally, little is known of the strength of a-Si and a-Ge type films, despite these films are becoming common structural/mechanical component materials of many MEMS devices.

Silicon has been reported to exhibit different strengths depending on its form, for example, $10 \sim 12$ GPa for silicon nanowires [150, 151], $1 \sim 3$ GPa for single crystalline thin films bulk-machined from crystalline wafers [152], $1 \sim 3.5$ GPa for polycrystalline thin films fabricated by various CVD techniques [97, 130, 142, 152, 153], and 1.75 GPa reported for LPCVD amorphous Si thin films [152].

Ge nanowires have a reported strength of 15 GPa [96, 123]. This is the theoretical maximum normal stress at failure in single crystal Ge. Silicon has a theoretical
maximum of 20 GPa [125, 154] but the largest experimentally reported strength is only 12 GPa. Compared to Si, very little has been reported on the strength of Ge in any form.

Crystalline materials often demonstrate an increase in strength with a reduction in dimension into the nanometer domain, possibly due to a reduction or absence of dislocations and defects [130, 155]. However, thin amorphous films demonstrate reduced strength compared to their thin film crystalline counterparts [97, 147, 153, 156, 157]. This reduced strength in amorphous thin films compared to crystalline thin films can be attributed to several factors, including:

- the lower average densities of the amorphous materials [158, 159],
- the inclusion of deposition gas atoms inherited from the deposition processes that attach to dangling Si bonds and ultimately reduce the population density of Si-Si bonds [160, 161],
- process or environment induced physical flaws such as micro cracking, pin-holes, and surface roughness, which create stress concentration locations [130, 160].

Due to the difficulties in handling and gripping extremely thin samples, direct tensile testing of thin film materials has been challenging. To overcome this difficulty, several alternative testing and measurement procedures have been trialled in the literature, including three point bending [162, 163], cantilever bending [152], membrane bulge testing [80, 164, 165], and suspended microbridge fracturing [145, 153, 157, 166–169].

It is noted that the three point bending, cantilever bending, and membrane bulge tests all characterise the material in bending mode, and are thus indirect measures of tensile properties. Fracturing of suspended microbridges is usually performed by loading the suspended microbridge perpendicular to the bridge length using a nanoindenter, thus creating a tensile stress state along the length of the bridge either side of the contact point. However, bridge failure (rupture) in such cases often occurs at the point of contact where the maximum bending occurs [145, 168–173]. As recognised by Zhang et al. [145] and Su et al. [170], this
method effectively measures only the bending strength of the material and does not provide a means for true tensile strength measurement. In the case of a typical microbridge bending test, for a thin film sample of 500 nm thickness curving around a blunt wedge-shaped indenter of 1 µm radius, the bending induced maximum tensile strain at the film surface will be 25%. This value is far greater than what would be needed to fracture the film at the point under the indenter tip prior to any possible failure elsewhere. In this regard, many of the above-mentioned micromechanical testing techniques reported in the literature operate in bending mode, and do not provide a reliable measurement technique for tensile strength of thin film materials in true tension [80, 145, 147, 152, 153, 157, 162, 163, 166–169].

A novel microstructure developed by Espinosa et al. results in an area of uniaxial tension within a microbridge [174, 175]. The unique geometry, shown in Figure 3.6, moves the failure point away from the contact area of the load tip, into a narrowed gauge region [174–176]. This design, however, requires vertical displacements greater than 10 µm. Such a displacement requirement may be out of reach of some popular nanoindentation tools [177, 178].

Figure 3.6: Optical micrograph of the test structure design by Espinosa et al. highlighting the two gauge sections and the central loading region. Image from Ref. [175]
3.3 Deposition induced stress and wafer-film distortion

Deposition processes often result in films experiencing a stress mismatch between the deposited film and the substrate. This stress mismatch results in distortion of the thin film and substrate. Considering that a film is often amorphous and has in-plane isotropy whereas the substrate is anisotropic in mechanical properties, the wafer distortion is expected to be anisotropic and nonuniform. In addition, for a MEMS device, structures often need to be released from the substrate. Internal stresses in films also cause shape distortion or bending of thin film structure. Therefore, understanding the causes of internal stresses in films and their influences on film and wafer distortion is critical for MEMS design and application.

3.3.1 Defining stress

*Pre-deformation* or *Stoney stress* is the stress present in an as-deposited film before the substrate is allowed to deform under the influence of the thin film.

*Residual stress* can arise from intrinsic or extrinsic factors and is the stress present in a film after the substrate and film is allowed to deform.

*Intrinsic* or *Growth stress* is a stress not due to lattice mismatch or thermal mismatch, instead it arrises from energetic or non-equilibrium deposition processes [179].

*Extrinsic* or *Induced stress* is a stress arising due to thermal mismatch or lattice mismatch [179].

3.3.2 Internal stress of thin films

When a stress mismatch between thin film and substrate arrises from the deposition processes, and if the substrate is allowed to deform under the influence of the thin film, the assembly may bend as shown in Figure 3.7. In the case of an
CHAPTER 3: MATERIAL PROPERTIES OF THIN FILMS AND THEIR SUBSTRATES

amorphous film, lattice parameter mismatch does not dictate the ultimate stress

Figure 3.7: Curvature in films as a result of thin films deposited with (a) their preferred orientation, (b) spacing less than preferred (thin film lattice parameter > substrate) giving rise to compressive stress, and (c) spacing greater than preferred (thin film lattice parameter < substrate) giving rise to tensile stress. Redrawn from Ref. [180].

state of the film. Instead, if the atoms in the deposited film have a separation less than their preferred separation, $a_o$, the film will have a compressive stress as shown in Figure 3.7(b). If the atoms in the deposited film have a separation greater than their preferred separation, $a_o$, the film will have a tensile stress as shown in Figure 3.7(c). Similarly, a film that is epitaxially grown on a substrate with mismatched lattice parameters will be under tension if the deposited film lattice parameter is less than the substrate (Figure 3.7(c)) and under compression if the deposited films lattice parameter is greater than the substrate (Figure 3.7(b)).

3.3.3 Substrate distortion

The damaging effects of stress in thin films deposited on substrates was noted as early as 1858, when Gore et. al found that electrochemically deposited antimony was prone to cracking when gently struck (vibrated) or heated [181, 182]. There are many ways in which stresses can arise in thin films [133, 183–185]. However, the greatest contributing factors are lattice mismatch and thermal expansion differences between the substrate and film.
Delamination and cracking are a common outcome of stress in a deposited film. Similar to thin film induced substrate bending, delamination and cracking is a mean of releasing pre-deformation intrinsic and extrinsic stress. If the stress is highly compressive, buckling delamination like that observed for BTIBD a-Si:Ar deposited on Ge(100) substrate, can occur. Figure 3.8(a) and (b) show an example of ribbons of delamination in an a-Si:Ar thin film. This particular a-Si:Ar film deposited via BTIBD on Ge(100) substrates had an as deposited compressive stress of over 1 GPa. Delamination was not observed in films deposited during the same run on Si(100) substrate indicating some substrate dependent adhesion issues.

Cracking in thin films typically occurs due to high tensile stresses of both intrinsic and extrinsic nature, or from tensile stress developed during crystallisation of the film. BTIBD films are highly compressive as deposited, only approaching zero stress at long anneal times or high anneal temperatures and the ICPCVD films used in this thesis are relatively stress neutral. Because of this, no cracking is observed for the films contained in this thesis. For reference, however, Figure 3.8(c) shows a film cracking due to a large tensile film stress and Figure 3.8(d) shows film cracking caused by stress induced through crystallisation of the film.

### 3.3.4 As-deposited thin film stress

One method for the evaluating as-deposited stress is through thin-film induced substrate bending, which provides a means for determining even relatively low levels of stress (on the order of tens of MPa) in a deposited film. This method of calculating stress has been used since 1909 when Stoney discovered that metal films deposited on thick substrates induced substrate bending. From this he was able to determine a relationship (the “Stoney” formula) between the curvature of the substrate and the pre-deformation stress in the thin film [188]. This technique continues to be used to characterise film-on-substrate or bi-layer structures [102, 131, 189, 190].

The relationship between the stress in the film, $\sigma_f$, and the curvature, $c$, of the bilayer structure is given by Stoney’s equation [188]:

\[
\sigma_f = \frac{E_s c}{2(1+\nu_s)}
\]
Figure 3.8: (a-b) Ribbons of delamination in an a-Si:Ar thin film deposited on a Ge(100) substrate. Cracking observed in thin silicon films due to (c) tensile film stress (Ref. [186]) and (d) stress induced from crystallisation of the thin film (Ref. [187])
where $M_s$ is the biaxial modulus of the substrate, $t_s$ is the substrate thickness, $t_f$ is the deposited film thickness, $R$ is the radius of curvature, and curvature is simply defined as $\kappa = 1/R$. The convention is to denote tensile stress as a positive stress and compressive stress as a negative stress. Knowing the thin film thicknesses, Stoney’s equation gives us a means for evaluating the stress in a thin film without knowledge of other material properties of the thin film assuming:

1. $t_s$ and $t_f$ are uniform and $t_f << t_s << R$
2. The film and substrate are homogeneous, isotropic, and linearly elastic
3. The film stress are in-plane and isotropic or orthogonally symmetric
4. The film stress is uniform across the surface

However, it is often the case that Stoney’s equation of stress are used even if all these assumptions are not met [191, 192]. While $E$ is variable with in the (100) plane of Si and Ge substrates, it has been shown that $M$ for Si and Ge is invariant in the (100) plane [193]. For the substrates used in this work, as discussed in Section 3.2.1.1, $M$ is a more appropriate means of quantifying the in-plane elastic behavior, as compared to $E$, for Si(100) and Ge(100) substrates. As such, all of the above assumptions hold true for the films and substrates used in this thesis.

Two popular techniques for non-contact determination of the curvature of a sample for use in Equation 3.3.1 are "three-beam" measurement and optical profilometry. The basic principles of the three-beam technique are shown Figure 3.9 (a). With this method three laser beams are reflected off the surface of a thin film and the change in spacing from $d_1$ and $d_2$ to $d'_1$ and $d'_2$, as shown in Figure 3.9, allows for the calculation of the curvature.

Optical profilometry uses light interference to measure height variation on the surface of a sample. Unlike three-beam which results in a 1D analysis of the sample surface, this technique gives a 2D surface profile for which radius of curvature
can be measured across the entirety of a sample as shown schematically in Figure 3.9 (b). From this curvature, Equation 3.3.1 is used to determine the stress in the as-deposited pre-deformation thin film material.

3.4 Thermal Properties

Thermal properties describe how a solid responds to thermal energy. There are three characteristic thermal properties of materials including: heat capacity, thermal expansion, and thermal conductivity. For MEMS devices operating across a wide range of temperatures, the most important thermal property is thermal expansion and its potential to cause device failure.

3.4.1 Coefficient of Thermal Expansion

Control of the internal stresses in thin films has long been critical for realisation of reliable and stable MEMS devices [194]. For the design of MEMS devices expected to function under wide temperature variations, the coefficient of thermal expansion (CTE) of its construction material is an important thermal property of concern. Thin film materials often exhibit different thermal expansion coef-
coefficients and behavior relative to their bulk counterparts, owing to the different chemical makeups and structural variations between thin film and bulk. This is of particular relevance for applications in which optical MEMS are combined with infrared sensing devices, which are typically cooled to cryogenic temperatures. In addition, some materials show abnormal thermal expansion behavior, such as a negative CTE, with single crystal Si at below 100 K being a typical example [195]. Thus thermal expansion behavior of SiGe thin films considered in this study requires careful characterisation.

The CTE of Si and Ge substrates are well known [196, 197]. Like other material properties of thin films, the CTE is highly dependent on deposition method. Reports of thin film CTE center around metal deposits and the few literature reports for a-Si and a-Ge thin films only report room temperature or elevated temperature values of CTE. Figure 3.10 shows the CTE values reported in the literature for thin film a-Si and a-Ge. It is often the case that the CTE for thin film silicon is greater than that of crystalline silicon and the same is true for thin film germanium compared to crystalline germanium [103, 131].

Figure 3.10: CTE for crystalline Si and Ge wafers, and thin film Si and Ge.


### 3.4.2 Thermal stress between film and substrate

The thin film stress induced substrate bending described in section 3.3 can be utilised to evaluate the change in stress with temperature of the deposited film. The change in stress with temperature enables the determination of the CTE. If a bilayer structure such as the one shown schematically in Figure 3.7 is cooled, the mismatch between the CTE of the two materials will induce additional stress and change the curvature of the sample [190, 194, 198–200]. This effect can be described by [190]:

\[
\frac{d\sigma}{dT} = -M_f(\alpha_s - \alpha_f)
\]  

(3.4.1)

where \(M_f\) is the biaxial modulus of the deposited film, and \(\alpha_f\) and \(\alpha_s\) are the CTEs of the film and the substrate, respectively.

By knowing the CTE of the substrate and measuring the curvature change (thereby determining the stress change) with temperature, the CTE of the thin film can be determined using Equation 3.4.1. However, this equation still contains two unknown parameters, \(M_f\) and \(\alpha_f\). Therefore, a second independent condition needs to be established. For this we deposit the same film on two different substrates, and Equation 3.4.1 holds true for the following two simultaneous equations:

\[
\frac{d\sigma_1}{dT} = -M_f(\alpha_s - \alpha_f)
\]

(3.4.2)

\[
\frac{d\sigma_2}{dT} = -M_f(\alpha_s - \alpha_f)
\]

(3.4.3)

where the subscripts 1 and 2 refer to substrates 1 and 2, respectively. By combining Equation 3.4.2 and Equation 3.4.3, an equation for the CTE of the film, \(\alpha_f\), can be derived:

\[
\alpha_f = \frac{\alpha_{s2}\frac{d\sigma_1}{dT} - \alpha_{s1}\frac{d\sigma_2}{dT}}{\frac{d\sigma_1}{dT} - \frac{d\sigma_2}{dT}}
\]

(3.4.4)
which requires the experimental determination of stress versus temperature for the film deposited on two different substrates, giving $d\sigma_1/dT$ and $d\sigma_2/dT$. It is assumed that (1) the choice of substrate does not result in any epitaxy in the film, (2) the substrate-film compositions have biaxial symmetry, and (3) the film thickness is much less than the substrate.
Chapter 4

Mechanics of MEMS

The mechanical and material properties of thin film a-Si:H and a-Si_xGe_{1-x} and crystalline Si(100) discussed in Chapter 3 can be related to the observed mechanical behavior of MEMS microstructures enabling predictive modeling of MEMS devices. The elastic properties of a material will affect any corresponding shape deformations and motion. The thermal properties, specifically CTE, give rise to stress in thin film microstructures when cooled or operated across a wide range of temperature. The effect of thin film stress on deformation and motion of thin film microstructures is the dominant property of concern in many applications and specifically of great concern when actuating large thin suspended membranes. This chapter will summarise the effect of stress on various thin film structures used in this thesis for the exploration and determination of thin film material properties. The vibrational modes in microcantilevers and the mechanisms for electrostatic actuation, will also be covered. Lastly, this chapter will discuss finite element modeling (FEM) for the prediction of mechanical behavior in MEMS.

4.1 Effect of thin film stress on microstructures

Stress in multi-layer structures can result in catastrophic device failure as well as rendering the device unusable due to mechanical stiffening or distortion. Stresses in such structures can result from many factors: lattice mismatch between deposited layers, lattice mismatch between the substrate and deposited layers, different CTE in materials, and variable thermal history inherent in ICPCVD pro-
Understanding the mechanisms that contribute to the observed structural deformation, and developing ways to mitigate them, is critical in MEMS design and application. Huang et al. studied the effects of residual stresses in PECVD silicon nitride cantilever and fixed-fixed beam structures [201]. Using finite element modeling (FEM) they identified a symmetric out-of-plane (across the thin film thickness) stress gradient of 275 MPa/µm ranging from 55 MPa compressive to 55 MPa tensile stress in cantilever beams of 0.4 µm in thickness. Many studies have reported on the intrinsic and residual stress in thin films. Since the work by Huang et al. there have been some reports on stress gradients through the thickness of the film [202–204]. Such a stress gradients can impact distortion, obscuring results from traditional residual stress measurement techniques such as fixed-fixed beam buckling and Guckel ring deformation [205].

### 4.1.1 Micro-cantilevers

The observed post-release deformation of a cantilever can reveal the stress that was present in the film prior to release. If the stress is in the plane of the thin film and uniform across the thin film thickness, upon release the microcantilever will lengthen (for compressively stress unreleased films) or contract (for tensile stressed unreleased films) while remaining in the plane of the deposited film. This is shown schematically in Figure 4.1 (a).

Additionally, as shown in Figure 4.1 (b), anchor effects can cause a cantilever with constant in-plane stress to angle up or down. This is because one surface of the deposited film is constrained, while the other is free to deform. If the deposited film prior to release is compressively (tensile) stressed, and the bottom surface of the anchor is constrained while the top surface of the anchor is free to deform as shown in Figure 4.1 (b), the structure will bend down (up) at the anchor point. The released portion of the film is free to expand (contract) as in Figure 4.1 (a).

An addition consideration for stress in thin films that is very common in ICPCVD film deposition is a stress gradient. The stress gradient, shown in Figure 4.1 (c),
Figure 4.1: Residual stress in the thin film prior to release will cause a released cantilever to (a) expand or contract, (b) angle down or up from anchor effects, and/or (c) curl from a stress gradient.

 upon release results in a curl up if the top surface of the film is more tensile (pre-release) than the bottom surface and a curl down if the top surface is more compressive (pre-release) than the bottom. The pre-release stress gradient, $\Delta \sigma$, in a film can be calculated from the radius of curvature of a released cantilever via:

$$\Delta \sigma = E \left( \frac{1}{R} \right) t_f$$  

(4.1.1)
where $E$ is the Young’s modulus of the film, $R$ is the radius of curvature, and $t$ is the film thickness.

### 4.1.2 Fixed-fixed beams

Stress behavior of a fixed-fixed beam mirrors that of the cantilever. If stress of the thin film is in plane and uniform across the thickness, a tensile stress in the unreleased beam will cause the beam to want to contract upon release. This will lead to no noticeable deformation, with the released beam remaining in tension or a crack in the film as a means to relieve this tension. However, if the stress of the unreleased thin film is in-plane and compressive, upon release of the fixed-fixed beam the expanding film will cause the microstructure to buckle as shown in Fig 4.2 (c). This buckling is often referred to as Euler buckling, which describes the minimum force required to cause the beam to buckle, and is given by [206]:

$$F_{\text{bucking}} = \frac{\pi^2 EI}{(KL)^2} \quad (4.1.2)$$

where $F_{\text{bucking}}$ is the critical force required to buckle the beam, $E$ is the Young’s modulus, $I$ is the moment of inertia of the cross section, $L$ is the suspended length, and $K$ is the effective length factor equal to 0.5 for a fixed-fixed beam.

It is important to note that buckling will only occur after a critical level of compressive strain is present in the beam. For a rectangular beam of uniform thickness critical strain, $\varepsilon_c$, is given by [205]:

$$\varepsilon_c = \frac{P}{AE} = \frac{\pi^2 t^2 f}{3L^2} \quad (4.1.3)$$

where $P$ is the load at buckling, $A$ is the cross sectional area, $E$ is the Young’s modulus of the thin film, $t$ is the film thickness, and $L$ is the length. For a a-Si:H film $E \approx 130 GPa$, $t = 1 \mu m$, and $L = 200 \mu m$, $\varepsilon_c = 8 \times 10^{-5}$ and $\sigma_c = 10.7 MPa$. So, for a typical microbridge, even small compressive residual stresses can induce some degree of buckling.
Equation 4.1.2 was developed by Euler to describe the buckling in large columns with vertical applied loads. Fang et al. showed that for a suspended thin film, without imperfections, the buckling deformation profile, $h$, can be described by [207]:

$$h = \frac{1}{2} h_{\text{max}} \left( 1 - \cos \frac{2\pi x}{L} \right)$$  \hspace{1cm} (4.1.4)

where $h_{\text{max}}$ is the maximum height of the profile, $x$ is the location along the length of the microbridge, and $L$ is the original suspended length.

Once the profile $h$ is determined, it can be used to calculate the increase in length of the microbridge, $\Delta L$. Given that $\Delta L = \varepsilon L$, if the Young’s modulus of the thin film material is known, the pre-release stress in the film can be determined. This pre-release stress would be the residual stress present in any unreleased areas of the thin film.

When a significant pre-release stress gradient is present across the thickness of the thin film, the deformation profile post-release may appear to be similar to that of a beam that has buckled upon release due to compression. However, a film
with average zero stress (or even slightly tensile stress) and a large stress gradient will buckle upon release, as shown in the Figure 4.3. This is due to the competing forces present in the film including anchor effects and rules pertaining to Gauss’ "Theorem Egregium" which states that the Gaussian curvature of a surface does not change if one bends it without stretching it [208]. In this case, witness wafers are best used to determine the average deposition stress of the thin film and FEM can be used to determine the level of gradient required to achieve the observed deformation.

**Figure 4.3:** (a) FEM of a fixed-fixed microbridge, with average pre-release tensile stress of 80 MPa and out-of-plane stress gradient of 160 MPa/µm showing upward “buckling” as is expected for a thin film microbridge with net compressive stress. Film is 1 µm thick. Image is exaggerated 10× in the z-direction to highlight deformation features. (b-g) show the progression of deformation for 0 MPa average pre-release tensile stress to 100 MPa.
4.1.3 Guckel rings

The fixed-fixed microbridges described above demonstrate a means for determining the pre-release compressive stress in a film with constant in-plane stress and uniform stress across the film thickness. However, they are not suited for determining tensile stress, as a released fixed-fixed beam under constant in-plane tension will generally remain flat. A microstructure referred to as the Guckel ring, named after developer H. Guckel, solves this problem and gives a means for measuring in-plane tensile stress [209].

For a Guckel ring, as pictured in Figure 4.4, points ‘A’ are affixed to the substrate. Figure 4.4(b) and (c) show the two primary modes of buckling that occur in the central beam of a Guckel ring under tensile strain. If the thickness of the central beam, $t_f$, is greater than the width $w_b$, the buckling will occur in the plane of the film. If, instead, the $t_f < w_b$ the buckling will occur out of plane. The latter style of Guckel compatible ring is most commonly used for MEMS diagnostic structures as it is most compatible with surface micromachining processes.

To determine the thin film tensile stress from a Guckel ring (Figure 4.4 (a)) with $w_r << R$, we must fist look at the forces present. When tensile strain is present in the film, the points at ‘A’ will be displaced by $\varepsilon_o R$ and points B are displaced $-\varepsilon_o R g(R)$ where $g(R)$ is the ratio of contraction to expansion given by [205]:

$$g(R) = -\left(\frac{2w_r f_2}{2w_r f_1 + w_b f_1^2 - w_b f_2^2}\right)$$

(4.1.5)

for $f_1$ and $f_2$ given by,

$$f_1 = \left(\frac{\pi}{4} - \frac{2}{\pi}\right) \left(\frac{R}{e}\right) - \frac{2e}{\pi R} + \frac{4}{\pi} - \frac{\pi}{4} + \frac{\pi k_f (1 + \nu)}{2}$$

(4.1.6)

$$f_2 = \left(\frac{1}{2} - \frac{2}{\pi}\right) \left(\frac{R}{e}\right) - \frac{2e}{\pi R} - \frac{1}{2} + \frac{\pi}{4} - k_f (1 + \nu)$$

where $R$ is the average radius ($R = (R_i + R_o)/2$ as shown in Figure 4.4), $k_f$ is a
form factor, $\nu$ is Poisson ratio, and $e = R - w_r/\ln(R_o/R_i)$ is the eccentricity.

As was the case for the fixed-fixed microbridge, the central beam of a Guckel ring will only buckle if the compressive strain, $\epsilon_c$, in the beam reaches the critical value which is given by [209]:

$$\epsilon_c = \frac{P_c}{Ew_b t} \tag{4.1.7}$$

where $P_c$ is the load for critical buckling and $E$ is the Young’s modulus of the film. The film tensile strain for a material with $\nu$ between 0.2 and 0.3, is then given by:

$$\epsilon = \frac{\epsilon_c}{g(R)} = \frac{9.985(t/R_c)^2}{12g(R)} \tag{4.1.8}$$

where $R_c$ is the critical radius and can be determined by placing Guckel rings of various radii (all with $w_b = w_r << R$) on the test wafer.

![Diagram](https://via.placeholder.com/150)

**Figure 4.4:** (a) Schematic of a Guckel ring showing defining values. Tension between points ‘A’, which are affixed to the substrate, results in the central beam buckling as (b) if the deposited film thickness $t$ is greater than the beam width $w_b$, or (c) if $t < w_b$. 

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As with the fixed-fixed microbridges, if a significant out-of-plane stress gradient is present across the thickness of the unreleased thin film, post-release the Guckel ring distortion will exaggerate the level of tensile stress determined from Equations 4.1.8 and 4.1.7. Figure 4.5 shows the characteristic deformation of a thin film Guckel ring with average pre-release stress of 80 MPa tensile and a 160 MPa/μm out-of-plane stress gradient, compared to the deformation of a Guckel ring with no pre-release stress gradient and a tensile stress of 1 GPa. From the discussion above on fixed-fixed microbridges, if a pre-release out-of-plane stress gradient is present, the deformation behavior of the diagnostic microstructures is altered from what is expected. FEM analysis is needed to evaluate the observed deformation due to stress gradient for a known average stress in the sample.

Figure 4.5: Deformation determined through FEM of a Guckel ring with pre-release residual stress of (a) 80 MPa average tensile stress with 160 MPa/μm out-of-plane stress gradient and (b) 1 GPa tensile stress and no stress gradient.
4.1.4 Suspended Membranes

The simplest case for evaluating deflection in a membrane due to pre-release thin film stress is to look at a membrane that is has uniform boundary conditions. That is, either a completely free standing floating film that is no longer attached to a substrate or one that is fully supported/fully constrained about its perimeter. Figure 4.6 shows a schematic of a fully supported/constrained, released membrane under uniform in-plane compressive (a) or tensile (b) stresses.

If a thin film is deposited such that it is under compressive stress, when released to form a free standing membrane it will expand in all directions. If the resultant thin film is under tensile load it will contract in all directions once released. If the membrane is fully supported about the perimeter as in Figure 4.6, a tensile load will pull the membrane flat, and could potentially cause tearing or warping. If the pre-released film is compressively stressed, the membrane will bulge up or down. If the compressive load becomes very large the membrane will buckle similar to the observed buckling in fixed-fixed beams.

4.2 Vibrational modes in microcantilevers

One method for determining the Young’s modulus and Poisson ratio of a deposited thin film is through the resonance method. The resonance method involves the excitation of the bending and torsional modes of a microcantilever. The Young’s modulus of a rectangular beam can be determined from the bending resonance frequency of the beam, $f_b$ [210, 211] also known as the Euler-Bernoulli bending vibration [212].

$$f_b = \frac{\eta_i^2}{2\pi \sqrt{12}} \left( \frac{t_f}{L^2} \right) \sqrt{\frac{E}{\rho}}$$  \hspace{1cm} (4.2.1)

where $\rho$ is the density of the thin film, $L$ the length of the cantilever, $t_f$ is the film thickness, and $\eta_i$ is determined from $\cos \eta_i \cosh \eta_i = -1$. Numerical values for $\eta_i$ are shown in Table 4.1
Figure 4.6: FEM of a 500 µm by 500 µm and 1 µm thick square suspended membrane deforming from (a) 75 MPa pre-release compressive stress and (b) 75 MPa pre-release tensile stress. (c) section of (b) with scale adjusted to highlight deformation from pre-release tensile film stress.
### Table 4.1: Numerical values for ηᵢ for the first 4 bending resonances

<table>
<thead>
<tr>
<th>η₁</th>
<th>η₂</th>
<th>η₃</th>
<th>η₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.8751</td>
<td>4.6941</td>
<td>7.8548</td>
<td>10.9955</td>
</tr>
</tbody>
</table>

Equation 4.2.1 holds true especially for long thin beams. As reported by Walm- sley [55], for small length to width \((L/w)\) ratios this equation results in a Young’s modulus that is slightly higher than the true modulus. This variation is, however, less than 5% and does not vary with thickness for cantilevers with \(L/w > 1\).

From the torsional resonance the shear modulus of the thin film material can be calculated [210] via:

\[
G = \left(\frac{2Lw}{\pi f_i} \right)^2 \rho \quad i = 1, 3, 5, \ldots
\]

(4.2.2)

where \(w\) is the beam width and \(f_i\) is the torsional resonance frequency. By experimental measurement of \(f_i\), the Poisson ratio, \(ν\), is calculated from the shear modulus and Young’s modulus via:

\[
ν = \frac{E - 2G}{2G}
\]

(4.2.3)

### 4.3 Electrostatic Actuation

The effects of stress on the deformation of thin film microstructures is of critical importance when dealing with actuable devices. Actuator devices are characterised by the mechanism of actuation: electrostatic, magnetic, thermal, and piezoelectric. When a voltage is applied across two conductors, an electrostatic force develops between them, which can be used to induce motion [8]. Nathanson developed one of the earliest examples of a micro-electrostatic actuator with the invention of a resonant gate transistor [213]. Figure 4.7 illustrates the basic principles behind electrostatic actuation in a coupled electro-mechanical device.

If we ignore fringing fields (assuming an infinite parallel plate) we can calculate the displacement due to an applied voltage in this system. In a movable plate system, once equilibrium displacement is achieved, the electrostatic force \(F_{electric}\)
is given by [8]:

\[ F_{electric} = \frac{1}{2} \frac{C(x) V^2}{(x_0 + x)} \]  

(4.3.1)

where \( C(x) \) is the displacement dependent capacitance between the two plates, \( V \) is the applied voltage, \( x_0 \) is the zero voltage displacement of the system, and \( x \) is the equilibrium distance between the two plates. The mechanical force, also called the restoring force, of the system is given by:

\[ F_{mechanical} = -k_m x \]  

(4.3.2)

where \( k_m \) is the mechanical spring constant. By combining equations (4.3.1) and (4.3.2) we get an expression for the equilibrium displacement of the parallel plate system:

\[ -x = \frac{F_{mechanical}}{k_m} = \frac{F_{electrical}}{k_m} = \frac{C(x) V^2}{2(x_0 + x) k_m} \]  

(4.3.3)

where \( K_m \) is the effective spring constant, which is a combination of the mechanical spring constant and the electrical spring constant.

The primary obstacle restricting the actuation range in electrostatic actuators is an effect known as pull-in or snap-down. This effect occurs when the electrostatic force overcomes the restoring force in the system and the top capacitor plate snaps down onto the bottom plate. In the basic case, as illustrated in Figure 4.7, the pull-in voltage, \( V_p \), of the system is given by [8]:

\[ V_p = \frac{2x_0}{3} \sqrt{\frac{k_m}{1.5C_0}} \]  

(4.3.4)

where \( x_0 \) is the initial separation of the plates, \( K_m \) is the effective spring constant, and \( C_0 \) is the initial capacitance between the two plates. Divergence from this ideal case is expected in practice due to fringing fields and non-linear mechanical spring constants in the material.
Figure 4.7: Coupled electro-mechanical device. $F_{\text{mechanical}}$ is the restoring force of the top plate as determined by the effective spring constant, $K_m$ (re-drawn from Ref. [8]).

4.4 Finite Element Modeling

While simple MEMS structures can be described through analytical models, when a system becomes complex it is often necessary to describe the behavior using computational methods. Finite Element Modeling (FEM) is one such computational technique. With FEM, a model is constructed by dividing a 3D rendering into many finite elements. Simplified behavior of each element is described and these elements interact with each other at nodes. A collection of nodes is referred to as a mesh. The FEM process results in a set of simultaneous equations and, as such, can be described as a piecewise polynomial interpolation [214]. Such a simulation does not result in a formula or a solution for a set of problems, but gives an approximate solution to the specific problem and set of conditions being tested.

Figure 4.8 shows the two most common mesh types used in FEM: the brick element mesh and the tetrahedral mesh. Brick elements are orthogonal and can only be used to mesh orthogonal structures. A tetrahedral mesh is composed of irregular tetragonal mesh elements and can be used to mesh curved or irregular shaped objects. To improve accuracy of a mesh, parabolic mesh elements can be used. These mesh elements include an extra node at the center of each element edge. Figure 4.8 highlights the difference between a linear element and a parabolic element. The use of parabolic elements can triple the total number of nodes in a mesh, and as such, are computationally expensive.
The complex behavior of a suspended membrane being actuated is not easily described by an analytical solution. The actuation of such a structure is analysed via FEM. The nature of the actuation and the impact of various support structures will be covered in detail in Chapter (8).

**Figure 4.8:** Schematic of a linear beam with brick mesh and tetrahedral mesh. Parabolic mesh elements include additional nodes on the element edges compared to linear mesh with nodes only at the element vertices.
Experimental Procedures

Evaluation of the material properties is imperative for the successful design of MEMS devices. Knowledge of the mechanical/material properties gives key insight into both device performance and reliability. This chapter will cover the preparation of the investigated ICPCVD a-Si and BTIBD a-Si$_x$Ge$_{1-x}$ thin films and specific processing steps used to create MEMS test structures. It will also cover the experimental set up for the techniques used to determine the thin film mechanical and material properties. Measurement techniques are best grouped according to the technique itself as many measurement techniques overlap in the properties they test.

5.1 Preparation of the investigated thin films and microstructures

Table 5.1 list the deposition, surface micromachining, and bulk micromachining steps required to fabricate the test structures described throughout this work. Overall fabrication process is shown schematically in Figure 2.6, with further details described in the following sections.
### Table 5.1: Processing steps for MEMS test structure fabrication

<table>
<thead>
<tr>
<th>Processing Step</th>
<th>Tool</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SiO$_x$ deposition† ‡</td>
<td>Sentech SI 500D</td>
</tr>
<tr>
<td>2a</td>
<td>Si deposition</td>
<td>Sentech SI 500D</td>
</tr>
<tr>
<td>2b</td>
<td>Si$<em>x$Ge$</em>{1-x}$ deposition</td>
<td>4Wave LANS</td>
</tr>
<tr>
<td>3</td>
<td>Photoresist deposition and patterning</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>a-Si:H, a-Si$<em>x$Ge$</em>{1-x}$ etch</td>
<td>Oxford Instruments Plasma Lab system 100</td>
</tr>
<tr>
<td>5</td>
<td>SU-8 backside deposition and patterning</td>
<td>Oxford Instruments Plasma Lab system 100</td>
</tr>
<tr>
<td>6</td>
<td>Si(100) cryogenic etching</td>
<td>Oxford Instruments Plasma Lab system 100</td>
</tr>
</tbody>
</table>

† no SiO$_x$ deposition for thin film induced substrate bending  ‡ Polyimide layer added between SiO$_x$ and Si layer for Guckel rings and fixed-fixed bridges

#### 5.1.1 a-Si:H

The hydrogenated amorphous Si (denoted a-Si:H) thin films used in this work were deposited using a Sentech SI 500D ICPCVD system with the deposition conditions summarised in Table 5.2. Deposition conditions for the SiO$_x$ stop etch layer are also summarised in the table.

The a-Si:H films were patterned using photolithography and a dry chemical etch in a Oxford Instruments Plasma Lab system 100 ICPRIE (inductively-coupled...
plasma reactive ion etch). The etch chemistry was 5 sccm O\textsubscript{2} and 160 sccm SF\textsubscript{6} with an RF power of 50 W and an ICP of 600 W. To etch a film 500 nm in thickness required 15 sec run time. The same etch recipe is used for the a-Si\textsubscript{x}Ge\textsubscript{1-x} films below.

### 5.1.2 a-Si\textsubscript{x}Ge\textsubscript{1-x}

The amorphous silicon germanium films (denoted a-Si\textsubscript{x}Ge\textsubscript{1-x}) are sputter deposited using an Ar plasma and as such contain trace amounts of Ar. In keeping with the nomenclature above for a-Si:H, the amorphous argon containing silicon-germanium thin films should be denoted as a-Si\textsubscript{x}Ge\textsubscript{1-x}:Ar, but for brevity, clarity, and consistency with literature the "Ar" identifier is dropped.

The a-Si\textsubscript{x}Ge\textsubscript{1-x} thin films characterised in this chapter where deposited via BTIBD (see Section 2.1.1 for detailed description) on a 4Wave Laboratory Alloy and Nanolayer System (LANS). Elemental sputtering targets, 4 inches in diameter, of pure Si and Ge were used as the source material. To obtain the various ratios of Si to Ge, sputtering rates from the individual targets were controlled via varying the target duty cycles according to the process described in detail by Krishnan et al. [215] with Table 5.3 summarising the adopted values. Figure 5.1 depicts the target duty cycle. The Si-Ge alloy has complete mixing without the formation of any secondary microstructures [216].

<table>
<thead>
<tr>
<th>Thin film composition (Si\textsubscript{x}Ge\textsubscript{1-x})</th>
<th>Target duty cycle</th>
<th>Deposition rate (nm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si\textsubscript{0.75}Ge\textsubscript{0.25}</td>
<td>OFF</td>
<td>2.07</td>
</tr>
<tr>
<td>Si\textsubscript{0.61}Ge\textsubscript{0.39}</td>
<td>12/2</td>
<td>2.45</td>
</tr>
<tr>
<td>Si\textsubscript{0.48}Ge\textsubscript{0.52}</td>
<td>12/2</td>
<td>3.07</td>
</tr>
<tr>
<td>Si\textsubscript{0.33}Ge\textsubscript{0.66}</td>
<td>12/2</td>
<td>3.96</td>
</tr>
<tr>
<td>Si\textsubscript{0.22}Ge\textsubscript{0.78}</td>
<td>12/2</td>
<td>5.78</td>
</tr>
<tr>
<td>Ge</td>
<td>OFF</td>
<td>3.83</td>
</tr>
</tbody>
</table>

Ar flow: 53 sccm Target bias: Si -800 V, Ge -500 V

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5.1.3 Through wafer etching

For the release of a-Si:H and a-Si_xGe_{1-x} suspended structures for mechanical testing, a multi-phase approach that utilises RIE, anisotropic-chemical etching, and isotropic-chemical etching is employed. Figure 5.2 shows schematically the desired etch process for deep through-wafer etching and Figure 5.3 shows a flow chart of the etch steps and gas chemistries used for the removal of the Si(100) substrate.
**CHAPER 5: EXPERIMENTAL PROCEDURES**

Figure 5.3: Cryo etch flow chart with (a)-(d) corresponding to steps in Figure 5.2.
In this process, first a RIE dry etch is employed to remove the bulk of the Si(100) substrate wafer. This etch requires large biasing power and relatively high oxygen flow. The high oxygen flow tends to passivate the side walls as well as the horizontal etch front, while the large bias results in ion bombardment of the etch front. This energetic ion bombardment removes any passivation on the horizontal etch front while leaving the passivation layer on the vertical side walls. SF$_6$ along with the bombarding ions push the etch front through the Si wafer. Due to the high voltage biasing this etch did not show any selectivity between the Si(100) wafer and the SiO$_2$ stop etch layer. As such, this etch is timed to reach within a few 100 nm of the SiO$_2$ stop.

While the specific gas flows that result in straight sidewalls may vary tool-to-tool and with sample geometry, the etch profile gives insight to what modes are dominating the etch. Figure 5.4 shows SEM micrographs of selected processing outcomes with (d) being the desired straight sidewall etch. In general four factors contribute to etch profile: temperature, SF$_6$ and O$_2$ flow, and RF biasing. SF$_6$ is responsible for chemical etching of the Si(100). As such, reduction in SF$_6$ flow slows the etch rate, while increases in SF$_6$ flow lead to outward sloping side walls. O$_2$ is responsible for the passivation of the surface, with low O$_2$ flow will lead to outward sloping sidewalls and high O$_2$ flow resulting in over-passivation. This leads to the development of "grass" at the bottom of the etch trench.

Once the vertical etch traversed the bulk of the Si(100) wafer, a passivation step consisting of relatively high oxygen flow + a set SF$_6$ flow is alternated with an etching step consisting of reduced oxygen flow + a set SF$_6$ flow. During the passivation step, high oxygen concentration allows for passivation of the side walls as well as the horizontal etch front. During the etching portion of the cycle the oxygen passivation on the horizontal layer is removed through ion bombardment allowing the SF$_6$ (plus the bombarding ions) to etch the Si wafer while the side walls are protected from chemical etching. This etch is more isotropic than the main etch and results in a sloped sidewall as pictured in Figure 5.2, but is selective between Si(100) wafer and the SiO$_2$ due to reduced biasing. The cyclical etch steps and gas chemistries for this portion of the etch process are shown in the flow chart, Figure 5.3(c).
Finally, a dry etch of the SiO₂ using CF₄ is used to clear the stop etch layer and release the MEMS structure. This process step and gas chemistry is included in the flow chart in Figure 5.3.
5.2 Preparation of finite element models

The commercial FEM suite CoventorWare 2014 was used for the simulation of MEMS microstructures for validation of experimental CTE (Section 5.6) and ultimate tensile strength (Section 5.7). Figure 5.5 gives an example of the two most common meshes: (a) tetrahedral mesh and (b) brick mesh. For the microstructures investigated, some meshes result in improved computational performance (faster solution times) and improved outcome accuracy. Increased mesh density, of any mesh form, will improve the accuracy of a run. However, there is a point where additional mesh elements lead to no improvement in simulation accuracy, but the computation time continues to increase. Mesh optimisation is used to find the intersection between minimum computation time and maximum accuracy.

(a) Tetrahedral Mesh

(b) Brick Mesh

Figure 5.5: Square and round top-hat anchor fixed-fixed microbridges meshed using (a) tetrahedral and (b) brick mesh. Mesh statistics are shown in Table 5.4.
To give a comparison of the two meshes shown in Figure 5.5, a square anchor and round anchor fixed-fixed microbridge with the same volume and surface area were meshed with the tetrahedral and brick where applicable. Mesh elements of 7.5 \( \mu \text{m} \) (tetrahedral mesh) and 5\( \mu \text{m} \times 5\mu \text{m} \times 0.25 \mu \text{m} \) (brick mesh) were used. The number of volume \((N_v)\), surface \((N_s)\), and total \((N_{tot})\) mesh elements, as well as the ratio of volume to surface elements \((N_v/N_s)\) for each mesh time are listed in Table 5.4. We can see from the Table 5.4 that the tetrahedral mesh results in a high \(N_v\) to \(N_s\) ratio, whereas the brick mesh gives a \(N_v\) to \(N_s\) ratio of about 1 for a mesh with 2500 elements. If the height of the mesh element is increased from 0.25\(\mu \text{m}\) to 1\(\mu \text{m}\) (the thickness of the thin film structure) for the brick mesh variant, \(N_{tot}\) is reduced by more than 50\% (Table 5.4 *reduced brick). Additionally, the number of surface elements dominates for the reduced brick mesh compared to all others. Such a reduction in total mesh elements and a reduced number of volume elements can lead to a loss in accuracy. However, mesh optimisation ensures that accuracy is retained while reducing total run time.

**Table 5.4:** Mesh statistics for round and square top-hat anchor fix-fixed micro-bridges

<table>
<thead>
<tr>
<th>mesh</th>
<th>(N_v)</th>
<th>(N_s)</th>
<th>(N_{tot})</th>
<th>(N_v/N_s)</th>
</tr>
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<tbody>
<tr>
<td>Round</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>tetrahedral</td>
<td>1408</td>
<td>1144</td>
<td>2552</td>
<td>1.23</td>
</tr>
<tr>
<td>Square</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(a) tetrahedral</td>
<td>1413</td>
<td>11138</td>
<td>2551</td>
<td>1.24</td>
</tr>
<tr>
<td>(b) brick</td>
<td>1270</td>
<td>1264</td>
<td>2534</td>
<td>1.00</td>
</tr>
<tr>
<td>*reduced brick</td>
<td>328</td>
<td>790</td>
<td>1118</td>
<td>0.42</td>
</tr>
</tbody>
</table>

Mesh optimisation was performed to determine a mesh element density that reduced computation time while maintaining solution accuracy. The square fixed-fixed beam is used as an example. A low density brick mesh, such as that in Figure 5.5(b) with one layer of through thickness elements, was chosen for the structure. To increase the number of mesh elements the element size was incrementally increased over a number of simulations. A voltage bias was placed on the fixed-fixed beam and the displacement and run time were recorded for various mesh element totals \((N_{tot})\).

Figure 5.6 shows the as-modelled displacement at the centre of the microbridge for a set voltage bias deflecting the microbridge as a function of the number of
Figure 5.6: Optimisation of brick mesh density for maximum accuracy and minimum computation time on a square top-hat anchor fixed-fixed microbridge.

mesh elements. Also plotted in the figure is the computation time versus total mesh elements. It is seen that as the number of mesh elements increases there is a point at which the solution remains the same but computation time continues to increase. At this point any reduction in element size or increase in mesh density will not result in an improvement in the FEM accuracy. For the brick mesh optimum meshing occurred between $N_{tot} = 2500$ and $N_{tot} = 3000$, with a run time of approximately 1500 s. Comparatively, the tetrahedral mesh with 8000 mesh points and a total run time of 3000s had not yet converged on a solution. As such, the brick mesh is superior to the tetrahedral mesh and is preferred where applicable.

5.3 Quartz crystal microbalance for the density of thin films

A quartz crystal microbalance (QCM) is used to determine the density of a deposited film, through measuring the change in resonant frequency of a quartz crystal. The change in frequency of the quartz crystal due to the deposited film is given by the Sauerbrey equation [217]:

$$80$$
\[ \Delta f = -\frac{2f_i^2}{A\sqrt{\rho_q \mu_q}} \Delta m \]  

(5.3.1)

where \( \Delta f \) is the change in resonant frequency due to thin film deposition on the quartz crystal and the associated change in mass \( \Delta m \), \( f_o \) is the pre-deposition frequency, \( A \) is the active area of the crystal, and \( \rho_q = 2.65 \text{ g/cm}^3 \) and \( \mu_q = 31.14 \text{ GPa} \) are the density and shear modulus of the quartz crystal, respectively. Since the mass of the film is the film density, \( \rho_f \), times the volume of the film, \( V_f = t_f \times A \), the density of the thin film can thus be calculated for a known film thickness, \( t_f \), or:

\[ \rho_f = \frac{\Delta f \sqrt{\rho_q \mu_q}}{2f_o^2 t_f} . \]  

(5.3.2)

The resonant frequency of three quartz crystals was taken prior to deposition. The three QCMs were then placed in the deposition chamber along with witness wafers for determining the deposited film thickness. Film thickness was measured using a Dektak 150 surface profilometer. For a-Si:H films the quartz crystals used had no previous depositions. Post deposition, the frequency of the QCMs was again measured giving \( \Delta f \) for a-Si:H and \( f_o \) for any subsequent runs. Depositions of Si\(_x\)Ge\(_{1-x}\) films were deposited on the same quartz crystals as the a-Si:H following the recipes in Table 5.3. The resonant frequency of the QCMs were recorded between each deposition giving \( \Delta f \) for each run and \( f_o \) for the subsequent run. Given that a-Si:H and Si\(_x\)Ge\(_{1-x}\) deposited films are amorphous and shown to have very low surface roughness, subsequent density characterisation for Si\(_x\)Ge\(_{1-x}\) can be performed on the same crystal assuming the quartz surface area, \( A_q \) and thin film surface area \( A_f \) are equal. (\( A_q = A_f \) must be true for Equation 5.3.2 to hold true).
5.4 Nanoindentation

The most common method for the determination of Young’s modulus and hardness of a material is nanoindentation. Variations of hardness testing began at the turn of the 20th century [218]. Modern nanoindentation is a variation on Rockwell hardness testing [219] for the measurement of smaller volumes of material and was popularised in the early 1970s [220]. With the addition of depth sensing technologies for nanoindentation, Young’s modulus could be determined in addition to hardness [221].

Various indenters were trialled throughout history [218, 222], but the most commonly used version today is a diamond Berkovich tipped indenter (shown in Figure 5.7). This particular tip has gained popularity due to the relative ease of fabrication of having the three faces join at a single apex point, compared to the Vicker’s indenter composed of four faces. The angle of the faces from vertical on the Berkovich tip is 65.35 degrees, giving it a relatively flat profile. For the indentation testing throughout this thesis, a diamond Berkovich tip is attached to the loading arm of a Hysitron TI950 Triboindenter.

Figure 5.7: SEM micrograph of a diamond Berkovich nanoindentation tip.

During nanoindentation testing, the Berkovich tip is vertically loaded and driven into the sample to a given depth or load value. Then the load is removed and the sample material relaxes. During testing the applied load, $P$, and the depth, $h$, are recorded giving the load-displacement (or $P - h$) curves schematically shown in Figure 5.8. The tip area function describes the contact area, $A_c$ as shown in Figure 5.8, as a function of contact depth $h$. 
Figure 5.8: Schematic of a typical nanoindentation P-h curve with key measurement parameters. Redrawn from Ref. [180, 223].

The majority of materials undergo an elastic-plastic deformation during the loading segment of nanoindentation. However, upon unloading, it is assumed that only elastic displacements are recovered [223]. Following the methods proposed by Oliver and Pharr, in the absence of plastic deformation, stiffness of a sample is given by the unloading portion of the $P - h$ curve as the load, $dP$, per change in contact depth, $dh$:

$$S = \frac{dP}{dh}.$$  \hspace{1cm} (5.4.1)

In Figure 5.8, stiffness ($S'$) is given as the slope of the top 60 - 95% (or the linear region) of the unloading portion of the $P - h$ curve.

Given $S'$, the reduced modulus, $E_r$, of the material can be calculated from the equation:

$$E_r = \frac{\sqrt{\pi}}{2\sqrt{A_c}} \times S'.$$  \hspace{1cm} (5.4.2)

The reduced modulus is used to determine the Young’s modulus, $E$, of the sample from:

$$\frac{1}{E_r} = \left(\frac{1 - \nu^2}{E}\right)_{\text{sample}} + \left(\frac{1 - \nu^2}{E}\right)_{\text{indenter}},$$ \hspace{1cm} (5.4.3)
where $E_{\text{indenter}}$ and $\nu_{\text{indenter}}$ for a standard diamond indenter are 1140 GPa and 0.07, respectively.

The hardness, $H$, of the material is given as the maximum load divided by the contact area at maximum load, or:

$$H = \frac{P_{\text{max}}}{A_c} \quad (5.4.4)$$

There are a few limitations to nanoindentation. Due to the tip area function not being well defined and rapidly changing at less than 50 nm, all data in this thesis is restricted to contact depths greater than 50 nm. Second, we are limited to the top 10 - 20% of the film thickness. If contact depths exceed this limit, the characteristic material properties of the substrate start to significantly influence the reading of the nanoindenter. Figure 5.9 shows an example of the variation in Young’s modulus with thickness taken from the work by Jung et al. [224].

To enable the use of contact depths beyond 20% of film thickness, Jung et al. expanding on work by Hu et al., showed that Young’s modulus and hardness can be expressed as a function of film and substrate values [224, 225]:

**Figure 5.9:** Plot of Young’s modulus versus relative contact depth for SiN$_x$ and SiO$_x$ films deposited on Si substrates. Dashed lines are asymptotic limits for Si (right) and the thin film materials (left). Figure taken from Ref. [224].


$E = E_s \left( \frac{E_f}{E_s} \right) \zeta$  \hspace{1cm} (5.4.5)

$H = H_s \left( \frac{H_f}{H_s} \right) \xi$  \hspace{1cm} (5.4.6)

where subscripts $f$ and $s$ denote the film and substrate, respectively. The indices $\zeta$ and $\xi$ are defined as dimensionless sigmoidal functions given by:

\[
\zeta = \frac{1}{1 + A \left( \frac{h_c}{t_f} \right)^C}
\]  \hspace{1cm} (5.4.7)

\[
\xi = \frac{1}{1 + B \left( \frac{h_c}{t_f} \right)^D}
\]  \hspace{1cm} (5.4.8)

with $h_c$ referring to indentation contact depth, $t_f$ the film thickness, and $A$, $B$, $C$, and $D$ being fitting coefficients. In Figure 5.9, the solid line represents the best fit of the data to Equations 5.4.5, 5.4.6, 5.4.7, and 5.4.8.

In order to accurately extract Young’s modulus and hardness from the P-h curves using the techniques described above, a sufficiently large sample set must be taken. Figure 5.10(a) shows an example of a 25 point array used for sample testing. A trapezoid load function test consisting of a load-hold-unload segmented routine was used. Figure 5.10(b) shows examples of the load function for 1,000 $\mu$N, 5,000 $\mu$N, and 10,000 $\mu$N maximum loads. Samples were indented at load rate of 200 $\mu$N per second to a maximum load varying from 500 $\mu$N to 10,000 $\mu$N, subsequently held constant for five seconds, and unloaded at a rate of 100 $\mu$N per second. At least three arrays of 25 load points were taken from disperse locations on each sample to ensure sample uniformity and measurement accuracy.

To determine the nanoindentation tool tip area function and establish a baseline for standard deviation in the data, indentation data is collected on a standard fused quartz sample. The data from this test is shown in Figure 5.11. A tip area function is fit to the data between each of the 6 runs shown. Even with extensive fitting of the tip area function it is seen that the hardness and Young’s modulus

85
Figure 5.10: (a) AFM micrograph taken on the Hysitron triboindenter showing an example of a 25 indent array in a thin film of a-Si:H. (b) Prescribed load functions for max loads of 10,000 µN, 5000 µN, and 1000 µN.

has an apparent tailing off at a contact depth of approximately 40 - 50 nm. This tailing off is attributable to the tip area function changing slightly with every indentation and as such not being well defined at small contact depths. A minimum indentation depth of 50 nm is required for this particular instrument to be able to reliably determine both hardness and Young’s modulus. Therefore, in this thesis reporting will be limited to indentations deeper that 50 nm.
Based on the data collected at above 50 nm of indentation depth, $H$ and $E_r$ of the fused quartz was measured to be 8.8 GPa and 69.9 GPa, respectively. Both values agree with listed values for the standard sample used and with literature [177, 221, 223].

### 5.5 Microcantilever vibration

An optical vibrometer is used to obtain the bending and torsional resonance frequencies of cantilever beams of various lengths and width such as those shown in Figure 5.12. The cantilevers are vibrated using a piezo stack in rigid contact with the sample. Two incident laser beams are focused on distinct locations on a cantilever as shown for various cases in Figure 5.13. The comparative amplitude between the two locations is determined versus the vibrational frequency of the piezo stack. The piezo stack is excited with a 3 V peak-to-peak sign wave and frequency is swept from 1 kHz to 1,000 kHz.

By placing the laser beams in the configurations shown in Figure 5.13 (a), the bending mode of the cantilever is captured. Placing the beams in the configuration shown in Figure 5.13(b) allows for the determination of the torsional res-
Onances. If the beams are not correctly aligned as in Figure 5.13(c), cross talk will occur and both torsional and bending mode resonances will show up in the frequency spectrum.

Figure 5.12: SEM micrograph of fully released a-Si:H cantilevers of various widths and lengths for vibrometry testing.

Figure 5.13: Schematic of incident laser beam configuration for (a) bending mode (b) torsional mode and (c) bending plus torsional mode which results in frequency spectrum with both sets of resonance peaks.
Figure 5.14 shows an example of (a) the frequency spectrum for a bending mode configuration and (b) torsional mode configuration from Ref. [210]. Slight miss alignment of the laser beams as shown in Figure 5.13(c), is observed experimentally as cross-talk in the frequency spectrum as shown in Figure 5.14(a). Squeeze film damping leads to peak broadening and high frequency peak shifting as observed for the torsional frequency in Figure 5.14(b) [210, 226].

![Frequency spectrum](image)

**Figure 5.14:** Frequency spectrum from (a) bending configuration and (b) torsional configuration, for thin film Si₃N₄ microcantilevers. Figures form Ref. [210].

### 5.6 Thin film induced substrate bending

A Zygo NewView 600K optical profilometry system was used for the capture of surface profile data of thin film-on-substrate samples. Curvature data was taken from the surface profile images of 2 mm x 15 mm samples prior to and after thin film deposition events. The change in curvature was used to determine the residual stress of the deposited films according to Stoney, introduced in Section 3.3.
5.6.1 Low Temperature Measurements

In Section 3.3.4 it was shown that thin film induced substrate bending can be used to determine the as-deposited, pre-released stress in a thin film. Section 3.4.2 discussed utilising changes in thin film stress with changing temperature for determination of coefficient of thermal expansion in a thin film. As such, a cryostat coupled to an optical profilometer provides a mean for determining the CTE of a thin film material from the experimentally determined change in bi-layer film curvature across a large range of temperatures. This system also provides a means of observing device and microstructure behavior across a wide range of temperatures.

For the low-temperature curvature measurements, a cryostat with a 3 mm thick BK7 window was used, requiring an objective with dispersion compensation (OMP-0553A) for the Zygo optical profilometer. A vacuum base pressure of $10^{-6}$ Torr was achieved for the cryostat setup using an Edwards T-station high vacuum system with turbo-molecular pump (EXT75DX) and dry backing pump (XDD1). Figure 5.15 shows the setup for cryogenic optical profilometry system. Thermal control of the cryostat was achieved via a LakeShore Model 335 PID temperature controller coupled to an copper coil heater and a Si diode temperature sensor.

A sample is placed at the end of the cold finger and affixed with thermal epoxy. For calibration, a thermocouple was placed at TC1 (fixed end of the sample) and TC2 (free end of the sample). For curvature measurements a thermocouple is placed at TC1 only. The sample is covered with a copper thermal shield to reduce temperature gradient along the length of the sample. The sample is enclosed in all three directions apart from 3 small circular openings (visible in Figure 5.15 through the viewing window) through which measurements are performed. Test locations are indicated in Figure 5.15 inset. The chamber is evacuated to vacuum ($10^{-6}$ Torr) to eliminated condensation on the viewing window or sample and the sample is cooled via liquid nitrogen. Temperature is set via the PID controller with a control loop between the copper heat coil and Si diode temperature sensor. Once the sample has reached a minimum temperature, the sample is left to equilibrate for 15 minutes. Optical profilometry measurements are taken at three
Figure 5.15: Experimental setup for the measurement of curvature at low temperatures. The sample is placed at the end of the cold finger, being mounted with thermal epoxy, and a thermocouple is placed at position TC1 and TC2 as shown in the inset. TC2 is only placed during thermal gradient characterisation.
locations (indicated in Figure 5.15) for each temperature increment. The temperature was incremented by 5 K steps between 85 K and 110 K and then 10 K steps from 110 K to 300 K allowing five minutes at each set point for the sample to equilibrate.

Thin film stress is deduced from the film induced curvature. Thus, the change in stress as a function of sample temperature is recorded for each sample. As per Equation 3.4.1, the change in stress with temperature is related to the difference in CTE of the film and the substrate. If the modulus of the film is well known across the full temperature range, Equation 3.4.1 can be used to determine the CTE of the deposited film. However, as is often the case, if the modulus across the whole temperature range is not known a second substrate is used and the CTE of the deposited film can be determined as per Equations 3.4.2, 3.4.3, and 3.4.4. So, each film to be tested was deposited on a thin substrate of Si(100) and Ge(100).

5.6.2 Validation of low-temperature curvature technique

To accurately determine the temperature dependency CTE, the temperature at the sample needs to be calibrated to the silicon diode temperature reading (or set point). Temperature readings were taken from the sample at two locations, one near the fixed mounting point and another at the free end, to determine if any thermal gradient was present along the sample. The two T-type (-270 °C to 350 °C) thermocouples (TC) used for these measurements are labeled Figure 5.15 as TC1 and TC2. The TCs were attached to the sample with silver epoxy.

For the thermal calibration, the temperature was set by the diode-heater control loop. Thermal equilibrium was reached for the set point and temperatures were recorded for TC1 and TC2. Figure 5.16 shows a plot of the temperature calibration data between 87 K and 300 K. The maximum difference between TC1 and TC2 is 2 K, and was achieved by using the copper thermal shield. The difference between the two thermocouples is temperature dependent. At low temperatures TC2 reported a reading of 1 K greater than TC1. The two readings are equal at 190 K followed by a steady increase in disparity with a TC2 reading 2 K less than TC1 at 300 K. The difference between the diode reading and TC2 increased.
monotonically with decreasing temperature, and reached a maximum $\Delta T$ of 15 K at a nominal diode temperature of 87 K.

Observation of the radius of curvature of a SiN$_x$ thin film on a GaAs substrate and subsequent calculation of the temperature dependent change in stress of SiN$_x$ is detailed as a proof of concept for later use with Si$_x$Ge$_{1-x}$ thin films. Figure 5.17 shows the evolution of the radius of curvature with temperature for the ICPCVD SiN$_x$ thin film sample. A sample of 700 nm SiN$_x$ on 100 $\mu$m GaAs (100) substrate, previously tested [180, 227] with the three-beam laser reflection technique described in Section 3.3.4, was tested on the cryogenic optical profilometry system to validate the measurement tool.

Sample curvature data were taken for temperatures ranging from room temperature (295 K) to the cryostat minimum sample temperature of 95 K. The thin film residual stress was then calculated using the Stoney equation as given by Equation 3.3.1. For comparison, Martyniuk’s original measurement [180], which
Figure 5.17: (a) Radius of curvature and (b) the stress versus temperature for a SiN$_x$ thin film on a GaAs substrate using the three-beam setup (Ref. [180]) and the cryogenic optical profilometry system.
was made using a three-beam laser reflection technique, is also shown in Figure 5.17. The stress calculation for the SiN\textsubscript{x} thin film on GaAs substrate based on curvature taken from the cryogenic optical profilometry system showed good agreement with the three-beam curvature measurement technique.

5.7 Tensile testing of thin film microstructures

The fracturing of suspended microbridges is used for the measurement of tensile strength in thin films. The first criterion for evaluating tensile strength is to create a testing condition of pure tension, in this case using a nanoindenter tool. To achieve this, double necked suspension bridge samples were considered, as illustrated in Figure 5.18. In this design, the two narrow sections of width \( w_1 \) are the gauge sections for testing, and the wide auxiliary sections of width \( w_2 \) support the gauge sections and allow application of the load. The load is applied perpendicular to the microbridge surface using a wedge shaped indenter as schematically illustrated in Figure 5.18. Having \( w_2 \gg w_1 \) also ensures that the tensile strain is concentrated in the two narrow gauge sections during elongation. The central (auxiliary) region of width \( w_2 \) allows the application of a load away from the two narrow gauge sections where pure tension allows for true tensile stress testing.

For microbridge fracture testing, samples of eight different \( w_2/w_1 \) width ratios were designed, resulting in a nominal \( w_2/w_1 \) ratio ranging from 2 to 9 in integer steps. The microbridges were fabricated from 500 nm thick a-Si:H or a-Si\textsubscript{x}Ge\textsubscript{1–x} thin films on 300 \( \mu \)m thick Si(100) wafer substrates (see Table 5.2 for a-Si and 5.3 for a-Si\textsubscript{x}Ge\textsubscript{1–x} deposition conditions). Further processing followed that outlined in section 5.1 Table 5.1. The fabricated microbridges were tested using a Hysitron TI 950 TriboIndenter fitted with a wedge-shaped diamond tip indenter having a 200 \( \mu \)m length edge with an edge tip radius of 650 nm. The deformation was conducted under load control, and the load-displacement curve for each of the microbridges was recorded.
Chapter 5: Experimental Procedures

Figure 5.18: Schematic illustrations of a double-necked suspension microbridge (a) top view; (b) side view. The images are not drawn to scale.

The orthogonal load applied by the nanoindenter, $F_i$, was translated to a tensile force in the microbridge, $F_T$, using the relation [175]:

$$ F_T = \frac{F_i}{2 \sin \theta} \tag{5.7.1} $$

where $\theta$ is the angle of bridge deflection as shown in Figure 5.18 and $F_i$ is the load applied by the nanoindenter. Assuming the stress is uniform across the width of the microbridge and knowing the cross-sectional area of the narrow gauge section ($w_1 \times t$), the tensile stress in the gauge section can be computed as:
\[ \sigma_1 = \frac{F_T}{w_1 t} \]

\[ = \frac{F_i}{2w_1 t \sin(\theta)} \quad (5.7.2) \]

\[ = \frac{F_i \sqrt{(\frac{1}{2}L)^2 + (h)^2}}{2hw_1 t} \]

where \( L \) is the length of the microbridge, \( h \) is the displacement at the center of the microbridge, and \( t \) is the film (microbridge) thickness. For later reference, \( z \) is the coordinate for through the thickness of the film as indicated in Figure 5.18.

The design proposed by Espinosa et al. [174] results in uniform stress across the width of the microbridge, \( w \), at any point along the length, \( L \). For such a sample, with gradual changing width profile, the strain in any given volume can be determined expressly through an analytical solution [228, 229]:

\[ \varepsilon_1 = \frac{\varepsilon^o L_T}{w_1} / \int_0^l \frac{1}{w(l)} dl \quad (5.7.3) \]

where \( L_T \) is the total length of the microstructure, \( w(l) \) represents the width function of the microbridge along the length, \( \varepsilon^o \) is the effective strain of the entire structure, \( \varepsilon_1 \) is the true strain in the test region, and \( w_1 \) is the specified width at the test region. Given the stress and strain correlation at any point the Young’s modulus of the thin film material can also be determined using this technique. The full derivation of Equation 5.7.3 is given in Appendix B.

The shortening of the structure to enable fracturing within 5 \( \mu \)m of vertical displacement is accomplished by reducing the distance over which \( w_2 \) steps down to \( w_1 \). This rapid change in width can result in non-uniform stress across the width of the microbridge [228, 229]. As such, finite element modeling (FEM) results were evaluated to determine maximum stress in the gauge region for microbridge deformation at rupture. Post process, scanning electron microscope (SEM) micro-
graphs of the released microbridges were used to define the geometry of models for FEM. Due to the potential for slight variations from the photolithography mask geometry during surface micromachining, FEM simulations were run for each released microbridge. The CoventorWare2014™FEM suite was used for the simulations. Load was applied mimicking a nanoindentation load giving a FEM load-displacement (P-h) curve. From the FEM output, stress distribution across deflecting microbridges at the moment of rupture were analysed. The stress was determined for the microbridge following a 1D analytical approximation (Equation 5.7.2) and through evaluation of the FEM results at a displacement equal to the experimental rupture displacement.
Properties of ICPCVD a-Si:H thin films

A popular material for microelectromechanical systems (MEMS) is a-Si, both for structural and optical applications. A vast number of studies have been done for Si-based thin films focusing on SiN$_x$ or SiC [190, 194]. Extensive studies of SiN$_x$ long-term stability have been conducted by others [190, 194, 230, 231], as well as for crystalline Si. However, the body of work done on amorphous Si, particularly as a structural material, is sparse.

The research on a-Si:H thin films focuses primarily on the deposition conditions and how they relate to hydrogen concentration in the resultant film [100, 232, 233], or the optical and electron transport properties of the film [99, 234–242]. Some papers have also reported on room temperature thermomechanical properties [131] and residual stress [232, 243]. However, the long-term stability of a-Si:H thin films as a structural component for MEMS is largely unreported. The deposition techniques commonly used for a-Si:H result in hydrogen inclusions that may diffuse out with time and could result in changes to mechanical properties. Also missing from the literature are additional material properties that are required when building accurate and predictive models. This section aims to fill in material property gaps by characterising residual stress, Young’s modulus, poisson ratio, CTE, and ultimate tensile strength all for the same a-Si:H thin films.
6.1 Density of ICPCVD a-Si:H thin films

A quartz crystal microbalance was used to determine the density of the a-Si:H thin films. The method is described in section 5.3. The density of the a-Si:H film is determined to be 2.08 g/cm$^3$, less than the 2.33 g/cm$^3$ reported for crystalline Si [158, 159].

6.2 Young’s modulus and hardness of a-Si:H thin films

Nanoindentation technique has been used to determine the Young’s modulus and hardness of as-deposited ICPCVD a-Si:H thin films. It has also been used to probe the long-term stability of thin films through timed heat treatment experiments. In this study, a Hysitron 950 TI triboindentation system fitted with a diamond Berkovich tip was used.

The indentation data of the ICPCVD a-Si:H deposited on 300 µm Si (100) wafers are shown in Figure 6.1 including Young’s modulus and hardness as a function of contact depth. Seven ICPCVD a-Si:H film samples were tested. The samples were heat treated at 300 °C in an Ar atmosphere for various times up to 24 hours. The control for this experiment being a sample that did not undergo any heat treatment. For comparison the hardness and Young’s modulus of the bare Si(100) were also measured and are shown in the figure.

The Young’s modulus of Si (100) wafer is measured to be 175 GPa ± 3 GPa. This value is comparable to Si(100) reported in literature [127, 128]. The Young’s moduli of the ICPCVD a-Si:H film samples are apparently all well below that of Si (100) wafer, all found to be in the range of 124 GPa to 133 GPa. Young’s modulus describes a material’s ability to deform reversibly. In crystalline silicon the atoms are placed in densely packed arrangement with rigid bonds resulting in high Young’s modulus. Comparatively, a-Si:H atoms are less densely packed and the amorphous structure with dangling Si-H bonds provides more degrees of freedom to shift under an applied load. This results in a lower value for Young’s modulus.
Figure 6.1: Plot of Young’s modulus versus contact depth for bare 300 µm Si (100) substrate and ICPCVD a-Si:H thin films. The Young’s modulus, calculated from the reduced modulus via Equation 5.4.3 is 175 GPa for Si (100) substrate and approximately 130 GPa for the deposited a-Si using a $\nu$ of 0.28 and 0.23, respectively.

The hardness for the Si(100) wafer substrate is measured to be 11.4 GPa ± 0.2 GPa, which compares well to reported values [223]. The hardness of the ICPCVD a-Si:H film samples were within 9.5 GPa to 10.3 GPa, about 87 % that of the Si(100) wafer. Hardness, similar to Young’s modulus, measures the resistance of a material to shape change when a load is applied. There are many factors that contribute to the value of hardness of a material, one being the strength of inter atomic bonds. Crystalline Si being composed of densely packed Si-Si bonds is more able to resist load induced deformation than the less dense structure of
a-Si:H which is also ripe with weaker Si-H bonds. As such, a lower hardness is expected in an amorphous material compared to its crystalline counterpart.

The Young’s moduli for the a-Si:H thin film samples annealed at 300 °C are plotted in Figure 6.2(a) versus anneal time. The variation in observed Young’s moduli versus anneal is within the error of the measurement. Figure 6.2(b) plots the hardness versus heat treatment time for the ICPCVD a-Si:H films. As was the case with Young’s modulus, any change in hardness with time is within the experimental error.

The lack of change in Young’s modulus or hardness over time, but an increase in tensile stress (discussed later in Section 6.3.1) point to unbonded hydrogen out gassing from the films. Hydrogen exists in low temperature, low pressure ICPCVD a-Si:H as unbonded H₂ and monohydrogen bonded to dangling silicon bonds [244–247], with dihydrogen bonded to silicon occurring in high pressure depositions [246]. Most hydrogen exists as bonded hydrogen, with 2% to 40% existing as unbonded H₂ [244, 245]. The temperature ranges for which various hydrogen species evolve and diffuse from the a-Si:H film are 50 °C for unbonded

![Figure 6.2](image_url)

**Figure 6.2:** (top) plot of reduced modulus versus annealing time for ICPCVD a-Si:H thin film. (bottom) plot of hardness versus annealing time for ICPCVD a-Si:H thin film. The band shows the average ± one standard deviation of the data from any single sample. Bare Si substrate is plotted for reference.
hydrogen [247], 300 - 350 °C for dihydrogen bonded to Si [75, 248, 249], and 600 - 900 °C for monohydrogen bonded to Si. As the fully dissociated H₂ inclusions are few in number according to literature reports [70, 100, 105, 233, 244, 245] and do not have any associated bonds, it is reasonable that their exclusion from the film does not impact the thin film ability to deform freely. Also, the dominant bonded hydrogen species in low temperature, low pressure ICPCVD a-Si:H films is Si-H, which does not evolve until well above the annealing temperature used. Thus, there is no change in Young’s modulus with heat treatment duration. Hardness is dependent on many of the same mechanisms as Young’s modulus. As such, it is reasonable that exclusion of the fully dissociated hydrogen inclusions from the a-Si:H material would not be associated with any change in hardness.

6.3 Thin film induced substrate bending

6.3.1 Curvature and stress

Films deposited via ICPCVD can contain in-built internal stress as a result of the deposition process. The stress may be tensile or compressive, with the magnitude dependent on the deposition conditions. Full or partial relief of stress in films can cause distortions of the substrate or the released free standing fabricated MEMS structures. Thus it is important for MEMS design to determine these stresses. This knowledge will also allow prediction of shape distortion of MEMS structures when subjected to large temperature variations.

Internal stresses of thin films can be determined by measuring the curvature of its substrate, as explained in Section 3.3. Radius of substrate-film bi-layer curvature was measured for a number of as-deposited ICPCVD a-Si:H thin films to evaluate the distribution of as-deposited pre-release stress, or the Stoney stress, in a single ICPCVD deposition event. Films were deposited on 70 μm Si(100) substrates according to the deposition conditions reported in Table 5.2. These deposition conditions have been selected for the optical properties they produce in the films.
Figure 6.3 shows a histogram of the 20 deposited films, as well as the calculated normal distribution for a single deposition event. The as-deposited films were found to be compressive. The normal distribution of a single ICPCVD event following the outlined deposition conditions, is observed to have a mean residual stress value of -27 MPa and a standard deviation of 9 MPa.

Radius of curvature data were collected again after the samples were annealed in Ar at 300 °C for different times up to 48 hours. Figure 6.4 plots the Stoney stress versus annealing time for all 20 deposited films. As Figure 6.4 shows, the films become increasingly tensile with increasing annealing time. The level of tensile stress appears to be saturating at a maximum value of approximately 400 MPa with annealing time. However, the films annealed for 48 hours delaminated completely with only a loose dusting of a-Si:H films remaining. This indicates a stress of 400 MPa is enough to cause delamination in the ICPCVD a-Si:H films. As 300 °C is not a sufficiently high temperature to cause a-Si:H to begin crystallising [250], or to result in the dominant bound hydrogen species to evolve and diffuse.

![Figure 6.3: Histogram of the post deposition stress in 1 um-thick ICPCVD a-Si:H films on 70 µm Si(100) substrates. Deposition conditions contained in Table 5.2. The red line indicates the normal distribution, with a mean of -27.02 MPa and a standard deviation of 9.11 MPa](image)
as discussed in Section 6.2, the increase of tensile stress upon heat treatment is attributed to the dissociated hydrogen inclusions out-gassing from the samples [79, 251, 252]. This out-gassing is known to occur at room temperature [247]. As such, post process treatment of the films to eliminate unbonded hydrogen inclusion would result in improved stability of the MEMS devices over time.

### 6.3.2 Coefficient of thermal expansion

Coefficient of thermal expansion (CTE) is determined for a-Si:H thin films by measuring film stress as a function of temperature. This film stress is determined by measuring the curvature of the film-substrate bi-layer samples as described in Section 3.3 and Section 5.6. Two different substrates were used in this study, including 90 μm thick Si(100) and 110 μm thick Ge(100) wafers. The wafer substrates were cut to approximately 2 mm x 15 mm in dimension with the length aligned to the [110] crystallographic direction and the width aligned to the [110] crystallographic direction, labeled as y and x directions, respectively. Curvature of the substrate wafers were measured prior to film deposition.

The a-Si:H thin films were about 1μm thick. The film-on substrate strip samples
were subjected to cooling from room temperature (300 K) down to 85 K. At each
temperature, the curvature was measured and stress determined. A polynomial
function was used as a least-squares fit to the stress versus temperature data, and
the resulting change in stress with temperature $d\sigma/dT$, was used to determine
the CTE of the film, $\alpha_f(T)$, using Equation 3.4.4. To allow the use of Equation
3.4.4 to determine film CTE from the measured $d\sigma/dT$ data, material properties
of the Si(100) and Ge(100) substrates are required, as listed in Table 6.1.

Figure 6.5 shows the effect of temperature on the measured curvature and the
corresponding change in stress with temperature of the ICPCVD a-Si:H thin films.
Figures 6.5 (a) and (b) represent a-Si:H films deposited on the Si(100) substrate
and Figures 6.5 (c) and (d) represent a-Si:H deposited on Ge(100) substrates. It is
seen in Figures 6.5 (a) and (c) that there is a clear separation in curvature value
between the y and x directions. The separation results from the substrates having
slightly different curvatures in the two directions prior to deposition. This is
accounted for in the calculation of stress, and subsequent plots of stress data over
lay in the x and y directions.

Figures 6.5 (b) and (d) present the change in stress with change in temperature
for the Si/Si(100) and Si/Ge(100) samples, respectively. The data points are the
average of four measurements taken at each temperature with two standard de-
viations in the data indicated by the error bands (red band corresponding to the
error in the y-direction stress measurement and the grey band corresponding to

---

### Table 6.1: Material properties of a-Si, Si (100), and Ge(100)

<table>
<thead>
<tr>
<th>Material</th>
<th>M&lt;110&gt;</th>
<th>CTE Equation</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge(100)</td>
<td>138 GPa</td>
<td>$\alpha = 2.483 \times 10^{-6} + (-1.58 \times 10^{-7})T$ +$(3.18 \times 10^{-9})T^2 + (-2.17 \times 10^{-11})T^3$ +$(6.43 \times 10^{-14})T^4 + (-7.04 \times 10^{-17})T^5$</td>
<td>[128, 197]</td>
</tr>
<tr>
<td>Si(100)</td>
<td>180 GPa</td>
<td>$\alpha = 1.88 \times 10^{-6} + (-7.45 \times 10^{-8})T$ +$(7.45 \times 10^{-10})T^2 + (-2.51 \times 10^{-12})T^3$ +$(2.95 \times 10^{-15})T^4$</td>
<td>[127, 195]</td>
</tr>
<tr>
<td>a-Si:H</td>
<td>130 GPa</td>
<td>$\alpha = -3.231 \times 10^{-6} + (7.201 \times 10^{-8})T$ +$(6.29 \times 10^{-10})T^2 + (3.66 \times 10^{-12})T^3$ +$(1.12 \times 10^{-14})T^4 + (1.31 \times 10^{-17})T^5$</td>
<td>this work</td>
</tr>
</tbody>
</table>
Figure 6.5: Effect of temperature on curvature of ICPCVD a-Si:H on Ge(100) and Si(100) substrates. (a) and (b) correspond to ICPCVD a-Si:H deposited on a Si(100) substrate. (c) and (d) correspond to ICPCVD a-Si:H deposited on a Ge(100) substrate. Two standard deviations of the data are shown as bands for x (in grey) and y (in red).
the error in the x-direction stress measurement). Error propagation analysis from known sources of error provides a lower value than the two standard deviation values plotted in Figure 6.5, the larger value was adopted.

Within the temperature range of the measurement, ICPCVD a-Si:H on Ge(100) substrates demonstrate a positive correlation between thin film stress and temperature indicating a lower CTE of the film relative to that of the substrate, as per Equation 3.3.1 and Equation 3.4.1. This is consistent with the findings of earlier studies for ICPCVD a-Si:H [102, 131, 132].

A polynomial approximation of the temperature dependence of stress, \( \sigma = f(T) \), was best fitted using the least-squares method for the data shown in Figure 6.5(b) and (d). The polynomials \( d\sigma_1/dT \) for the Si/Ge(100) sample and \( d\sigma_2/dT \) for the Si/Si(100) sample were then used in Equation 3.4.4 to calculate the temperature dependent CTE, \( \alpha_f(T) \), for the a-Si:H films on two different substrates. Figure 6.6 shows the \( \alpha_f(T) \) for the a-Si:H films. Also plotted in Figure 6.6 are the CTEs of Si(100) and Ge(100) wafers in the <110> directions, as expressed in Table 6.1, for comparison. The polynomial equation that best describes the CTE for ICPVD a-Si:H is also reported in Table 6.1. It is noted that the deposited a-Si:H thin film has a temperature dependent CTE very similar to that of the Si substrate.

The most notable feature of the curves shown in Figure 6.6 is that the CTE of the deposited a-Si:H crosses the value for the substrate CTE at approximately 210 K. This is an interesting finding as a crossing of CTE would be apparent in reversible deformation behavior observed in an a-Si:H microstructure on Si(100) wafer as it is cooled. As the temperature is reduced the apparent stress in the deposited material would become increasingly compressive (or decreasingly tensile) down to 210 K. In contrast, below this temperature the thin film stress would become increasingly tensile (or decreasingly compressive).

Using the experimentally determined CTE of a-Si:H and Equation 3.4.1, the elastic modulus, \( M_f \), for the deposited material can be determined. Using the data in Figure 6.5(b) and (d) for \( d\sigma/dT \), \( M_f = 120 \text{ GPa} \pm 20 \text{ GPa} \) is found across the range of 70 K to 300 K. This is a very indirect measurement of elastic modulus compared to other methods, however it is in agreement with the values for elas-
Figure 6.6: Temperature dependence of the CTE of ICPCVD a-Si:H thin films, in comparison with those of Si (100) and Ge(100) wafers [127, 195, 197]. The CTE for ICPCVD a-Si:H is observed to cross the CTE of the Si substrate at 210 K.

6.4 Effect of CTE mismatch on MEMS microstructures

To validate the temperature dependent CTE values presented in Table 6.1, cooling-induced structure deformation of a number of MEMS test structures was characterised by means of both experimentation and finite element modeling (FEM) using the CTE data determined in Section 6.3.2. Figure 6.7 shows the designs of the test structures, which include fixed-fixed beams (Figure 6.7 (a)), cantilever beams (Figure 6.7 (b)), and Guckel rings [209] (Figure 6.7 (c) and (d)). The a-Si:H films were 1 \( \mu \)m thick with structures aligned along the <110> directions of the Si substrate as indicated in the Figure 6.7. A sacrificial spacing layer of polyimide was used to create the space between the substrate and the free-standing MEMS structures, with anchor points being used to fix the structure to the substrate. The
structures were released by dry etching of the polyimide in an oxygen plasma in a barrel asher at 150 Watts for 15 minutes.

When depositing films via ICPCVD at elevated temperatures, an out-of-plane stress gradient often develops through the thickness of the film [201], which can be further enhanced during release steps when the deposited a-Si:H is exposed to an oxygen plasma. Analysis of the deformation of the Guckel rings often assumes negligible stress gradient across the film thickness. Stress gradients may confound information obtained from Guckel rings and fixed-fixed beam test structures. The cantilever structures, deformation of which tends to be indicative of stress across film thickness, were used to evaluate the stress gradient. The Guckel rings and fixed-fixed beams were used to evaluate stress changes with temperature due to CTE mismatch between the film and the substrate.

Figure 6.8 shows the observed distortion of an a-Si:H cantilever beam, with Figure 6.8 (a) showing the experimental optical profilometry data of a cantilever beam at room temperature (300 K). Using the stress strain equation, \( \sigma = \varepsilon E \), with Young’s modulus, \( E \), of 130 GPa for the ICPCVD a-Si:H (as determined in Section 6.2) and strain in the cantilever determined from the curvature of the beam in Figure 6.8 (a), the stress gradient through the thickness of the film is determined to be \( d\sigma/dz = 160 \text{ MPa/\( \mu \)m} \). Figure 6.8 (b) shows the result of FEM simulation of the profile of the cantilever, using a film modulus of 130 GPa, the experimentally determined film stress gradient of 160 MPa/\( \mu \)m, and parameters from Table 6.1 for the Si(100) substrate. Good agreement between the model and the experimental data is observed, confirming the value of calculated stress gradient. Figure 6.8 (c) plots the profile along the length of the cantilever as measured at three different temperatures. It is seen that the deflection in the cantilever did not change with temperature, indicating that the observed curvature is purely a feature of stress gradient within the deposited film and not a feature of CTE mismatch at the anchor.

The first structure used to evaluate thin film deformation due to CTE mismatch was the fixed-fixed beam shown in 6.7 (a). The beam was cooled in the system shown in Figure 5.15, and optical profile data was taken as a function of tem-
**Figure 6.7:** Test structures used to evaluate the effect of CTE mismatch between the a-Si:H MEMS structural layer and its substrate in experiments and in FEM simulations: (a) fixed-fixed beam, (b) cantilever beam, (c) and (d) Guckel ring structures. The Si film is 1 µm thick, and the heights in the drawings have been exaggerated 5x for ease of viewing.
**Figure 6.8**: Bending of an a-Si:H cantilever beam caused by an out-of-plane thin film stress gradient of 160 MPa/μm. (a) Optical profilometry image of the fabricated cantilever; (b) FEM modeling of the cantilever; (c) comparison of the height displacement profiles of the cantilever along the length between experimental measurements at three different temperatures and FEM.
Temperature, ranging from 85 K to 300 K. Using the stress gradient determined by the method shown in Figure 6.8 of 160 MPa/µm and the experimentally determined CTE (Figure 6.6) of the ICPCVD a-Si:H film, along with the film Young’s modulus determined through nanoindentation, and substrate parameters in Table 6.1, the fixed-fixed beam was modelled to evaluate the effect of CTE mismatch on cooling-induced shape distortion.

Modeling results were compared to the experimental data, and Figure 6.9 shows the change in beam shape with temperature. Figure 6.9 (a) shows experimental optical profilometry data for the fixed-fixed beam and the FEM beam at 160 K. Figure 6.9 (c) shows the profile of the beam along the center of the length direction (from a to a’ as indicated in Figure 6.9 (a)) at various temperatures, for both the experimental and FEM data. It is noted that at ambient temperature (300 K), the released beam shows a positive bowing upwards along the length direction. While upward bending of a beam with zero out-of-plane stress gradient would indicate a compressive film, FEM indicated that the previously determined out-of-plane stress gradient of 160 MPa/µm was sufficient to cause the beams to bend up in the observed manner. In fact, an average 80 MPa tensile stress was determined through FEM analysis of the fixed-fixed beam microstructures.

The results in Figure 6.9(c) and (d) indicate a slight discontinuity in height change with temperature around 200 K to 220 K. Above 220 K there are only slight changes observed in maximum beam height. Below 220 K the maximum beam height, \( z_{\text{max}} \) begins to reduce rapidly. This flattening implies that the film is becoming more tensile due to the film contracting more than the substrate, indicating a film CTE that is greater than that of the substrate. This experimental observation is consistent with the measured data presented in Figure 6.6. Figure 6.9 (b) shows the profile across the width of the beam, as measured from b to b’ as indicated in Figure 6.9 (a). The negative bowing across the beam width results from the film being relatively more compressive on the bottom and more tensile on the top due to the 160 MPa/µm out-of-plane gradient present in the films. Since the width is not constrained and free to deform, no large variation in profile with change in temperature due to differences in CTE is expected. Experimentally, as the temperature changed, no corresponding change in curvature of
Figure 6.9: Effect of cooling on profile distortion of a fixed-fixed beam. (a) Measured optical profile (left) and FEM model (right) images of the fixed-fixed beam at 160 K. (b) Beam profile across the width, b – b’, at different temperatures. (c) Beam profile along the length, a – a’, at different temperatures.
the beam across its width was observed.

Figure 6.10 shows two Guckel ring structures, which were also used to evaluate the CTE mismatch induced thin film deformation. The Guckel rings pictured in Figures 6.10 (a) and (d) are designed such that the central beam buckles when the film is under tensile stress, or remains flat when the deposited film is under compressive stress, with the amount of bowing indicating the magnitude of tensile stress in the film as discussed in Section 4.1.3 assuming negligible stress gradient. However, bowing will also be induced by any out-of-plane stress gradient in the film. Since two mechanisms for bowing are present, FEM was used to evaluate the room temperatures stress state.

The overall deformation of the structures indicated that the films were under an average tensile stress of 80 MPa at room temperature with a through thickness stress gradient of 160 MPa/µm. This is in agreement with the value obtained from analysis of the fixed-fixed beam test structures. Figure 6.10 (b) and (e) show the optical profilometry and FEM data for the cross beam of the 100 µm and 112 µm diameter Guckel ring structures at 180 K. Figure 6.10 (c and f) plot the change in curvature of the central cross beam with change in temperature. Note that a change in the direction of deformation is observed in the 100 µm Guckel ring and in the larger 112 µm ring below 200 K.

Referring back to Figure 6.6, the curve of the CTE of the deposited Si crosses that of the silicon substrate at approximately 210 K. This crossing causes the deformation behavior of the deposited a-Si:H film to switch about this point. When cooling from 300 K to 210 K the bowing radius of curvature of the cross beam in the Guckel ring increases, indicating a reduction in tensile stress in the deposited film due to the fact that the film CTE is lower than that of the substrate. For cooling below 210 K the bowing radius of the cross beam reduces, indicating an increase in tensile stress in the film due to the film CTE now being greater than the substrate. A similar behavior is observed in the larger 112 µm beam shown in Figure 6.10 (f), where the radius of curvature increases as temperature is reduced to 210 K, and then remains relatively constant when cooled below this value. The FEM data shows good agreement with the experimental results.
Figure 6.10: Effect of cooling on cross-beam distortion of Guckel ring test structures. (a) and (d) Optical image (upper) and FEM model image (lower) of the 100 µm and 112 µm diameter Guckel rings, respectively. (b) and (e) Plots of the profile along the length of the central beam of the 100 µm and 112 µm diameter Guckel rings, respectively. (c) and (f) Plots of the change in radius of curvature with temperature of the central beam of the 110 µm and 112 µm cross-beams, respectively.
These results impact the development of large area MEMS design. Tensile stress sufficiently large to cover the change in stress mismatch between ICPCVD a-Si:H thin film and Si(100) from 300 K down to 85 K is required for a membrane to remain flat for 300 K and for cryogenic operation. This is a stress value of greater than 20 MPa as indicated by the magnitude of stress change across the temperature range for a-Si:H on Si substrate as shown in Figure 6.5(b). Additionally, a design feature that amplifies effects of tensile stress in a similar manner to a Guckel ring such as a fixed-fixed beam with perpendicular tension from a suspended membrane (see Section 8.2.3), will result in unstable temperature dependent actuation. A design that does not deform due to substrate film CTE mismatch over a wide range of stress is desired.

6.5 Resonance of thin film microcantilevers

Determination of Young’s modulus, shear modulus, and Poisson’s ratio was achieved through cantilever vibration using the methods outlined in Section 5.5. Thin film a-Si:H was deposited to a thickness of 500 nm preceded by an SiO\textsubscript{x} stop etch layer according to the deposition conditions outlined in Table 5.2. Many samples, each containing 9 microcantilever testing arrays (Figure 5.12) were fabricated. The cantilevers were defined through surface dry etching using the Oxford instruments PlasmaLab System 100 ICP-RIE, followed by a deep selective cryogenic etch of the Si(100) substrate wafer following procedures outlined in Section 5.1 and Figure 5.3.

Since a through wafer etch was used, no squeeze film damping was expected and so the cantilevers were tested at atmosphere. The samples were mounted to a piezoelectric stack and the lasers were positioned on the microcantilevers according to Figure 5.13. The piezoelectric stack was then excited by a square wave with a peak-to-peak voltage of 5 V over a frequency range from 1 kHz to 600 kHz.
The length of the beams tested varied from 70 µm to 410 µm with widths of 20 µm or 30 µm. Figure 6.11 shows an observed frequency spectrum for bending and torsional mode vibration detection. A total of 72 frequency spectrum in bending configuration and 34 frequency spectrum in torsional configuration, were taken for microcantilevers of various lengths and widths. The set of observed frequency spectra were analysed to determine the 1st fundamental torsional and 1st, 2nd, and 3rd fundamental bending resonant frequencies for the various microcantilevers.

### 6.5.1 Bending resonance of a-Si:H microcantilevers

Resonant frequencies for the first three bending modes of the a-Si:H microcantilevers are plotted versus $t_f/L^2$ in Figure 6.12, where $t_f$ is the deposited film thickness and $L$ is the length of the microcantilever. It is seen that for all the three modes the data obtained for the samples of different geometries each are linearly correlated. Young’s modulus of a-Si:H is thus determined from fitting the frequency versus $t_f/L^2$ data in Figure 6.12 using Equation 4.2.1. The determined Young’s modulus values are given in Figure 6.12 next to the corresponding vibrational mode. It is seen that there is a small variance in the calculated Young’s modulus ranging from 109 GPa ± 3 GPa for the 3rd bending mode to 123 GPa
Figure 6.12: Plot of the first three bending resonances for cantilever beams of various lengths plotted versus $t/L^2$. The data is fitted using Equation 4.2.1 and the resulting Young’s modulus values ($E$) are shown.

± 2 GPa for the 2nd bending mode. Because 3rd bending mode resonances were not apparent at large $t/L^2$, this data is not a good representation of the material properties and is not considered in the determination of Young’s modulus of a-Si:H. The average Young’s modulus given the first two bending modes is 120 GPa ± 7 GPa. This value is a comparative to the value determined through nanoinindentation (Section 6.2, $E = 128$ GPa ± 9 GPa) and in the CTE study (Section 6.3.2, $E = 120$ GPa ± 20 GPa) for the ICPCVD a-Si:H material.

### 6.5.2 Torsional Resonance of a-Si:H microcantilevers

Figure 6.13 plots the resonant frequency versus $t/Lw$ for the 1st torsional mode, where $t_f$ film thickness, $L$ is the microcantilever length, and $w$ is the microcantilever width. To determine the shear modulus of a-Si:H, the frequency versus $t_f/Lw$ data is fitted using Equation 4.2.2. The shear modulus from the 1st torsional resonance is 49.7 GPa ± 2.3 GPa. This value is comparable to literature reports for thin film Si [253, 254].
6.5.3 Poisson ratio from a-Si:H microcantilever resonance

Poisson’s ratio of the a-Si:H thin film material can be determined from the Young’s modulus and shear modulus via Equation 4.2.3. Using a shear modulus and a Young’s modulus as determined above, the Poisson’s ratio is calculated to be $\nu = 0.2 \pm 0.04$. This value is in agreement with previously reported values for thin film Si materials [133–137].

6.6 Direct tensile testing of thin film strength

Strength of a-Si:H thin films is determined using the techniques described in Section 5.7 Microbridge samples of eight different $w_2/w_1$ step-down ratios were fabricated, including $w_2/w_1$ ranging from 2 to 9 in integer steps. The microbridges were fabricated using 500 nm thick a-Si:H thin films deposited on 300 $\mu$m thick Si(100) wafer substrates. Prior to a-Si:H deposition, a 200 nm thick SiO$_x$ layer was deposited on the bare silicon substrate. This layer provides the required stop-etch layer during back-side etching of the silicon substrate. Deposition con-
ditions for the two thin film layers are summarised in Table 5.2.

The microbridge samples were fabricated using the deposited a-Si:H films by means of reactive ion etching in SF$_6$ and O$_2$ plasma through a negative photoresist mask. Release of the microbridges to form free standing microstructures was achieved though back-side bulk micromachining of the Si(100) wafer using a highly selective cryogenic etch process described in Section 5.1.3. The samples were cooled to -110 °C using a He-cooled substrate holder. The Si wafer substrate was back-side etched using a SF$_6$ and O$_2$ gas chemistry under DC bias, which stops on the SiO$_x$ etch-stop layer. The SiO$_x$ layer was subsequently removed using a CF$_4$ etch at room temperature. The etch recipes used for each layer are summarised in Table 5.3.

Post process, SEM micrographs of the released microbridges were used to define the geometry of models for FEM. Due to the potential for slight variations from the photolithography mask geometry during surface micromachining, FEM simulations were run for each released microbridge. The CoventorWare2014™ FEM suite was used for the simulations with input material property parameters as determined in the previous sections. A load was applied mimicking a nanindentation load giving a FEM load-displacement curve. From the FEM output, stress distribution across deflecting microbridges at the moment of rupture were analysed.

Three identical wafers were processed, each containing two arrays of eight microbridges with eight $w_2/w_1$ ratios. For all microbridges $L = 110 \ \mu m$ and $w_1 = 9 \ \mu m$ were held constant, where as $w_2$ was varied from $16 \ \mu m$ to $90 \ \mu m$, giving a $w_2/w_1$ ratio range of 2 to 9. Figure 6.14 shows SEM micrographs of three fully released microbridges after testing, each having a different $w_2/w_1$ ratio.

A total 30 samples were tested, with at least 3 samples loaded to rupture for each $w_2/w_1$ ratio. The load was applied at a rate of 100 $\mu N/s$ until rupture or to a maximum displacement of 5$\mu m$. The load-displacement curves of a few selected samples of different $w_2/w_1$ ratios are shown in Figure 6.15. It is evident in Figure 6.15 that the load-displacement curves exhibit a progressive shift with respect to a change in the $w_2/w_1$ ratio, with samples having a higher $w_2/w_1$ ratio appearing
to be stiffer. That is, samples with higher \( w_2/w_1 \) ratios exhibit a smaller deflection displacement for any given load (or a higher load for any given displacement), which is related to the sample geometry. This behavior is expected since samples with a higher \( w_2/w_1 \) ratio have a wider \( w_2 \) section, which contributes a lower strain at any given load to the total elongation of the microbridge, leading to reduced displacement.

Among all the samples tested, 3 samples with \( w_2/w_1 = 2 \) were tested under both loading and unloading conditions without rupture. As evident from Figure 6.15, the non-ruptured sample (sample 2*) displayed negligible hysteresis between loading and unloading cycles, implying purely elastic deformation.

Considering the complex geometry, thus complex mechanical condition of the necked sections of the samples, FEM simulations of the deflection behavior of the samples were conducted using a Young’s modulus of 130 MPa (as determined for a-Si:H from traditional nanoindentation methods [255]) and a poisson ratio of 0.22 [133, 134]. The FEM simulated load-displacement behaviours of the samples are also shown in Figure 6.15 (discrete points).

For the FEM data presented in Figure 6.15, the microbridge stress distribution was evaluated at the displacement, \( h \), of experimental rupturing. Figure
Figure 6.15: Experimental load-displacement curves (solid lines) and FEM load-displacement (discrete points) for microbridges having different $w_2/w_1$ ratios, as labeled in the figure for each curve. Sample $2^*$ was loaded and unloaded without rupturing, whereas all other samples were loaded to rupture. The inset shows the prescribed load versus time for the test.

6.16 shows stress maps at the moment of rupture at three different cross-sections through the thickness of the microbridge (top, middle, and bottom) in comparison with the 1D analytical result calculated from Equation 5.7.2. Figure 6.16(a) shows the FEM stress at the top surface ($z = 0.5 \mu m$) of the microbridge, image(b) shows the stress distribution on the layer mid-way through the microbridge thickness at $z = 0.25 \mu m$, and image (c) presents the stress distribution at the bottom surface ($z = 0 \mu m$) of the microbridge.

It is evident in Figure 6.16(a) that a compressive (tensile) stress is present at any point of curvature, with a corresponding tensile (compressive) stress seen on the opposing surface in Figure 6.16(c). While significant in magnitude, the bending stress at the attachment points and the point of wedge indenter contact has a maximum value of 1.3 GPa, less than that observed at the gauge region. And, indeed, the magnitude of bending stress as determined by FEM was less for all samples than the stress at the point of failure. Additionally, lack of experimental failure in these regions confirms the reduced stress compared to the gauge region.

Due to the rapid reduction of the cross-section width from $w_2$ to $w_1$, a local
Figure 6.16: FEM simulation of stress distribution in a $w_2/w_1 = 2$ microbridge displaced to the experimental moment of rupture. (a) Stress distribution in the top surface ($z = 0.5 \mu m$); (b) stress distribution halfway through the thickness of the microbridge ($z = 0.25 \mu m$); (c) stress distribution on the bottom surface of the microbridge ($z = 0 \mu m$); (d) 1D analytical approximation of stress distribution in the microbridge overlaid on an SEM micrograph of the microbridge.

stress concentration occurs along the edges of the microbridge in the gauge region. However, comparing the stress distributions at the three height layers, it is clear that in the gauge region the stress is uniform through the thickness, demonstrating tension without bending was achieved for the sample geometry. For the $w_2/w_1 = 2$ microbridge shown, the FEM analysis indicates a failure stress of 1.49 GPa, which is the maximum stress value in the FEM map. Figure 6.16(d) shows the stress at rupture predicted by the 1D analytical equations (Equation 5.7.1 and 5.7.2). The load at rupture as determined from Equation 5.7.1 was used in Equation 5.7.2 to determine $\sigma(x)$ for any $w(x)$ along the length of the microbridge. The maximum stress using this technique occurs at the neck region, with a resultant value of 1.19 GPa for the microbridge shown in Figure 6.16(d). Indeed it is shown for all $w_2/w_1$ ratios that Equation 5.7.2 gives a conservative estimate of the strength of the material.

In a similar fashion, tensile strength of all ruptured a-Si:H samples was determined from the nanoindenter load-displacements and FEM stress maps of the microbridge samples at the moment of rupture. Figure 6.17 shows the ultimate
Figure 6.17: Rupture tensile strength for a-Si:H thin films measured from microbridges of different nominal $w_2/w_1$ ratios as determined via 1D analytical and FEM methods.

The rupture tensile strength of the a-Si:H microbridges as a function of $w_2/w_1$ ratio. The x-scale error bars represent the small measured deviation from the nominal $w_2/w_1$ ratios of the fabricated microbridges. The error in y corresponds to 2 standard deviations of the calculated rupture strength values. Correlation analysis of all 27 ruptured samples showed no statistically significant correlation between tensile strength and $w_2/w_1$ ratio for the thin film (1D) or FEM sample set. A conservative estimate of the tensile strength of ICPCVD a-Si:H is 1.3 GPa as given by the 1D analytical solution in Equation 5.7.2, with an upper bound of 1.6 GPa ± 0.1 GPa as determined by FEM.

The correlation between the 1D analytical solution for rupturing strength and $w_2/w_1$ ratio is determined to not be statistically significant with a p-value > 0.05. However, if the difference between $\sigma(FEM_{max})$ and $\sigma(1D)$ is taken and plotted against the radius of curvature of the gauge region of the microbridge, $R$, a strong correlation in the data emerges as shown in Figure 6.18. A significant correlation exists between $\sigma(FEM_{max}) - \sigma(1D)$ and $R$ with a correlation of -0.578 and a p-value < 0.01. The trend is for the 1D solution and the FEM analysis to converge
Figure 6.18: Difference between rupture strength determined from FEM ($\sigma_{\text{FEM}}$) and 1D ($\sigma_{\text{1D}}$) analysis versus radius of curvature in the gauge region, $R$. Correlation is -0.578 with a p-value < 0.001.

Literature reports of tensile strength have focused on single crystal silicon [139, 140], polycrystalline silicon [141–144], and silicon nitride and carbide films[80, 145–147]. Very little is known of the tensile strength of a-Si:H type films, which are becoming common structural/mechanical component materials for many MEMS devices. In addition, silicon has been reported to exhibit different tensile strength depending on its geometry. For example, $10 \sim 12$ GPa for silicon nanowires [150, 151], $1 \sim 3$ GPa for single-crystal thin films bulk-machined from crystalline wafers [152], $1 \sim 3.5$ GPa for polycrystalline thin films fabricated by various CVD techniques [97, 130, 142, 152, 153], and 1.75 GPa for low pressure-CVD (LPCVD) amorphous Si thin films [152]. Whereas conventional crystalline materials have been shown to increase in strength with a reduction in dimension to the nanometer domain, possibly due to a reduction or absence of dislocations and defects [130, 155], thin amorphous films tend to demonstrate reduced strength compared to their thin film crystalline counterparts [97, 147, 153, 156, 157]. This reduced strength in amorphous Si thin films compared to crystalline Si thin films can be
attributed to several factors. These include, the lower average density of amorphous materials [158, 159], the inclusion of precursor gas atoms inherited from the deposition processes that attach to dangling Si bonds and ultimately reduce the atomic density of Si-Si bonds [160, 161], and process or environment induced physical flaws such as micro cracking, pinholes, and surface roughness, which tend to create localised regions of stress concentration [130, 160].

Due to strain effects caused by the rapid change from \( w_2 \) to \( w_1 \), non-uniformity of stress across the width of the gauge region requires use of FEM to confirm maximum tensile stress at failure. The tensile strength of ICPCVD a-Si:H thin films tested in this study has been determined from FEM to be 1.6 GPa ± 0.1 GPa, which is slightly lower than the 1.75 GPa reported by Tsuchiya et al. [152] for LPCVD a-Si:H film tested in tension. This difference can be attributed to the deposition technique, since LPCVD occurs at elevated temperatures, which is likely to result in denser films with less hydrogen inclusions [70]. Therefore, mechanically stronger a-Si:H thin films would be expected in their experiment.

### 6.7 Summary

The material properties of ICPCVD a-Si:H thin films are determined using different techniques to provide a more in-depth appreciation of the properties by eliminating accidental bias of any one technique. The results obtained in this study are summarised in Table 6.2.

Heat treatment was found to cause variation in pre-release stress, or Stoney stress, of ICPCVD a-Si:H thin films, as presented in Section 6.3.1 and Section 6.2. The Stoney stress became significantly more tensile during the initial 5 minutes at an elevated temperature of 300 °C. This increase in tensile stress is attributed to hydrogen out-gassing from the thin film [232, 251, 252].

Evaluation of nanoindentation, reported in Section 6.2, showed that the Young’s modulus and hardness of ICPCVD a-Si:H are not significantly changed by exposure to elevated temperatures in an Ar atmosphere. Since the dominant hydrogen species targeted during heat treatment at 300 °C is fully dissociated hydrogen in-
clusions, these are not expected to impact Young’s modulus or hardness in the thin films. The determined value of Young’s modulus and hardness for ICPCVD a-Si:H is 128 GPa ± 9 GPa and 9.9 GPa ± 0.9 GPa, respectively.

The CTE of ICPCVD a-Si:H films, reported in Section 6.3.2 was determined to be lower than that of the Si(100) substrate at temperatures above 210 K and greater than the Si(100) substrate at temperatures below 210 K. The effect of the parity of CTE between the a-Si:H film and the Si(100) substrate at 210 K is evident in the behavior of fixed-fixed beams and Guckel rings. The analysis of these microstructures also showed that the out-of-plane stress gradient plays a large role in the room temperature deformation of these microstructures.

Young’s modulus, shear modulus, and Poisson ratio were determined from the resonant vibration of microcantilever beams in Section 6.5. Young’s modulus for ICPCVD a-Si:H thin films was determined to be 120 GPa ± 7 GPa. This value is just lower than values determined nanoindentation, but it is within the error of all the measurement techniques. The shear modulus was determined to be 49.7 GPa ± 2.3 GPa. Given the Young’s modulus and shear modulus a Poisson ratio of 0.2 ± 0.04 was determined.

Microbridge fracturing in Section 6.6 resulted in a direct measurement of tensile deformation behavior of thin film materials. The double-necked microbridge design ensures that tensile rupture occurs within the narrow gauge regions of the microbridge, which experience no bending during testing, thus allowing the true tensile properties of thin films to be measured. The ICPCVD a-Si:H thin films were found to deform in tension in a purely elastic manner to brittle fracture. Non-uniform stress across the width of the gauge section required use of FEM to determine maximum tensile stress at rupture. A comparison of the 1D analytical solution to the FEM results indicates a correlation between the rate of change from \( w_2 \) to \( w_1 \), quantified by the radius of curvature in the gauge region, and the disparity between the 1D approximation and the true tensile strength. A tensile strength of 1.6 GPa ± 0.1 GPa was determined for a-Si:H, which is comparable to reported values for polycrystalline silicon thin films deposited via LPCVD.
Table 6.2: Summary of material properties of ICPCVD a-Si:H

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Measurement Technique</th>
<th>Chapter section</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$</td>
<td>128 ± 9 (GPa)</td>
<td>Nanoindentation</td>
<td>6.2</td>
</tr>
<tr>
<td>$E$</td>
<td>120 ± 7 (GPa)</td>
<td>Cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td>$E$</td>
<td>120 ± 20 (GPa)</td>
<td>Substrate bending</td>
<td>6.3.2</td>
</tr>
<tr>
<td>$G$</td>
<td>49.7 ± 2.3 (GPa)</td>
<td>Cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td>$H$</td>
<td>9.9 ± 0.9 (GPa)</td>
<td>Nanoindentation</td>
<td>6.2</td>
</tr>
<tr>
<td>$\nu$</td>
<td>0.2 ± 0.04</td>
<td>Cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td>$\sigma_T$ (strength)</td>
<td>1.3 ± 0.1 (GPa)</td>
<td>Microbridge fracturing</td>
<td>6.6</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>$-3.231 \times 10^{-6}$ + $(7.20 \times 10^{-8})T$ $-(6.29 \times 10^{-10})T^2$ $+(3.66 \times 10^{-12})T^3$ $-(1.12 \times 10^{-14})T^4$ $+(1.31 \times 10^{-17})T^5$</td>
<td>Substrate bending</td>
<td>6.3.2</td>
</tr>
<tr>
<td>$\rho$</td>
<td>2.08 ± 0.25 (g/cm$^3$)</td>
<td>QCM</td>
<td>6.1</td>
</tr>
</tbody>
</table>
Properties of BTIBD $a$-Si$_x$Ge$_{1-x}$ thin films

Si and Ge are two materials frequently used in thin film fabrication for electron transport devices. Si and Ge also belong to the same group in the Periodic Table. They have the same crystal structure and form infinite solid solution alloys. This material is increasingly being employed in MEMS devices as the thin film stress can be controllable through variation of the Si to Ge compositional ratio [66]. For the purpose of hyperspectral sensing, SiGe-based devices offer the unique opportunity for a material system capable of functioning across the UV, visible, and IR spectrum as explained in Section 1.3 [61–65]. For such applications, it is essential to grow high quality SiGe thin films and have a good understanding of their fundamental mechanical properties. It is also desired for such a device to operate across a wide range of temperatures. With Si being a common substrate in MEMS applications, Si and Ge are anticipated to have similar CTEs to crystalline substrates. The $a$-Si$_x$Ge$_{1-x}$ films reported in this chapter were deposited according Table 5.3

7.1 Elemental analysis of $a$-Si$_x$Ge$_{1-x}$ thin films

Figure 7.1 shows and Energy-dispersive X-ray spectroscopy (EDS) spectrum for a nominally pure Ge sample deposited on sapphire substrates. The EDS spectrum, collected on a Hitachi TM3030 Tabletop Scanning Electron Microscope and
analysed with the EDX Quantax70 analysis software, was used to quantify the proportion of Si and Ge in the BTIBD films. The dominant contaminant is the deposition gas Ar at an estimated 5 atomic % for all compositions. Si to Ge ratios for the various BTIBD duty cycles, as determined through EDS, are reported in Table 5.3.

### 7.2 Density of BTIBD a-Si$_x$Ge$_{1-x}$ thin films

A quartz crystal microbalance (QCM) was used to determine the density of the a-Si$_x$Ge$_{1-x}$ thin films for the full range of compositions according to the methods described in Section 5.3. Densities of the a-Si$_x$Ge$_{1-x}$ films deposited via BTIBD as reported in Section 5.1 are shown in Figure 7.2 as a function of Si content.

The density of the (nominally) pure BTIBD Ge film is measured to be 4.7 g/cm$^3$. This value is greater than previously reported in literature for amorphous germanium [257–259], but lower than that of bulk single crystal Ge at 5.35 g/cm$^3$ [260]. In comparison, density of the (nominally) pure BTIBD Si film is measured to be 2.5 g/cm$^3$. This is greater than that of bulk single crystal Si at 2.33 g/cm$^3$ [260]. Crystalline Si and Ge both exhibit a diamond cubic crystal structure, atomic packing factor (ATP) of 0.34. This structure is not particularly dense when compared
to body-centered cubic (ATP = 0.68) or face-centered cubic (ATP = 0.74), leaving room for an amorphous form to have a higher density than the crystalline structure [261]. However, both a-Si and a-Ge are shown consistently in literature to have a density less than that of crystalline [158, 159, 257–259, 262, 263] with a theoretical maximum density for a-Si and a-Ge of 93 % that of crystalline [256]. A lower density in amorphous Si and Ge is indeed reasonable due to the imperfect packing and dangling Si or Ge bonds that exist in PVD type films [158, 159].

One possible explanation for the increased density of the a-Si compared to the single crystal form is the inclusion of Ar atoms as a byproduct of the deposition process [160, 161]. Ar is observed as being present in the films, though not perfectly quantifiable, in EDS characterisation of the a-Si\(_x\)Ge\(_{1-x}\) thin films. A sample EDS spectrum obtained for a pure Ge sample is shown in Figure 7.1, and a similar degree of Ar content is observed for all the BTIBD films. Ar could exist as interstitials or be attached to dangling Si and Ge bonds that are typical in PVD and CVD depositions. Ar atoms, being heavier than Si, could contribute to a thin film density for BTIBD Si that is slightly greater than bulk single crystal Si. The Ar atoms, being lighter than Ge, could contribute to a thin film density for BTIBD
a-Ge that is still less than bulk single crystal Ge. Further quantification of the Ar content and its nature would be beneficial, however, this is considered to be outside the scope of this thesis.

### 7.3 Young’s modulus and hardness of a-Si$_x$Ge$_{1-x}$ films

Nanoindentation technique was used to determine the Young’s modulus and hardness of as-deposited BTIBD a-Si$_x$Ge$_{1-x}$ thin films as a function of compositional variation. Films of 500 to 1000 nm thicknesses and different Si-Ge ratios were deposited via BTIBD (see Section 5.1) on 300 $\mu$m thick Si(100) substrates. Young’s modulus and hardness versus contact depth for selected Si-Ge ratios are shown in Figure 7.3. A dependence on contact depth can be observed for both Young’s modulus and hardness. As such, the data was analysed and a curve fitted using the methods prescribed by Jung et al. as outlined in Section 5.4 via Equations 5.4.5, 5.4.6, 5.4.7, and 5.4.8. In Figure 7.3 for low contact depths, $h_c$ less than approximately 100 nm, the fitted curves approach the Young’s modulus or hardness of the thin film. For large contact depths, $h_c$ generally deeper than 100 nm, the fitted curves tend to approach the elastic modulus or hardness of the Si(100) substrate.

In total, 19 a-Si$_x$Ge$_{1-x}$ films were evaluated. A summary of the obtained Young’s modulus values are plotted in Figure 7.4 as a function of Si percentage. Some additional measurements from work done in tandem with researcher Ruijing Ge [264] are also plotted in Figure 7.4 along with the Young’s modulus for single crystal (100) oriented Si and Ge [128]. A strong linear correlation is observed between composition and the obtained Young’s modulus values. The Young’s modulus of 160 GPa ± 4 GPa obtained for nominally pure BTIBD Si is very similar to the commonly reported value of Young’s modulus for bulk Si(100) of 169 GPa. A comparable difference is observed between the reported bulk single crystal Ge Young’s modulus of 138 GPa and that of nominally pure BTIBD a-Ge at 127 GPa ± 3 GPa. This consistent trend indicates a dependence of Young’s modulus on the deposition process as well as the form (amorphous thin film versus bulk single crystal) of the test species.
Figure 7.3: Young’s modulus, $E$ (left) and hardness, $H$ (right) versus contact depth for various compositions of 500 nm thick BTIBD a-Si$_x$Ge$_{1-x}$ thin films. Curves represent fitting of the data with Equations 5.4.5 through 5.4.8 according methods introduced by Jung et al. [Ref. [224]].
Figure 7.4: Young’s modulus versus Si content for BTIBD $a$-$\text{Si}_{x}\text{Ge}_{1-x}$ thin films. Young’s modulus, $E$, and biaxial modulus, $M$, are also plotted for bulk single crystal Si(100) and Ge(100) for reference as well as the elastic modulus of ICPCVD $a$-Si:H from chapter 5.

Figure 7.5 reports the hardness values measure for the BTIBD $a$-$\text{Si}_{x}\text{Ge}_{1-x}$ thin films as a function of percent Si, along with the data collected in tandem with Ge et al. and the hardness for crystalline Si and Ge [265]. The hardness values exhibited a clear linear correlation with composition. The hardness for nominally pure BTIBD Si and Ge are lower than those for their bulk single crystal counterparts by approximately 1.5 GPa. The reduced hardness can be attributed to the lack of a rigid crystalline structure, a reduced density compared to crystalline, and the associated reduction in crystalline bonds which contribute to a materials resistance to loading deformations.

Of additional notice is the outlying point of nominally pure Ge in both hardness (Figure 7.5) and Young’s modulus (Figure 7.4), with a reported hardness and Young’s modulus of 8.25 GPa and 142 GPa, respectively. These data for this Ge sample, reported in the work by Ge et al. [264], do not follow the linear trend of the other compositions in the sample set. Hardness and Young’s modulus versus contact depth for this sample are shown in Figure 7.3. Figure 7.6 (a) displays load-depth curves of this Ge film subjected to various maximum loads. It is seen that the behavior has a significant scatter. In contrast, the same measurement for
Si\textsubscript{0.5}Ge\textsubscript{0.5} film shown in Figure 7.6 (b), deposited under the same conditions is much more repeatable.

Figure 7.6(c) and (d) shows the atomic force microscope (AFM) scan images of the BTIBD Ge and Si\textsubscript{0.5}Ge\textsubscript{0.5}, respectively. The root mean square (rms) roughness of the Ge film is 25 nm and that of Si\textsubscript{0.5}Ge\textsubscript{0.5} film is 2.5 nm. This order of magnitude increase in surface roughness is attributed to the crystallisation of the Ge film when metal contaminants are present [266]. X-ray diffraction (XRD) analysis of the Ge sample presented in Figure 7.7 showed strong crystalline peaks compared to the characteristically amorphous XRD spectrum observed for the other films deposited under the same conditions. It is concluded that the scatter present in Figure 7.4(Ge), Figure 7.5(Ge), and Figure 7.6(a) for BTIBD a-Ge results from high surface roughness of the film. While crystallisation of the Ge film could account for the increased Young’s modulus and hardness, the scatter in the data means no confident conclusion can be drawn. Re-configuration of the 4wave BTIBD chamber after the depositions conducted by for the work by Ge et al. was aimed specifically at reducing sputtering of undesired metal species from the internal target shutter and resulted in metallic contamination below the measurement threshold of the EDS system. The EDS spectrum for Ge in Figure 7.1 is

![Graph showing hardness versus Si content for BTIBD a-Si\textsubscript{x}Ge\textsubscript{1-x} thin films.](image)

**Figure 7.5:** Hardness versus Si content for BTIBD a-Si\textsubscript{x}Ge\textsubscript{1-x} thin films. Hardness for bulk single crystal Si(100) and Ge(100) are also plotted for reference.
Figure 7.6: P-h curves for BTIBD films of (a) nominally pure BTIBD Ge and (b) BTIBD Si$_{0.45}$Ge$_{0.55}$ thin film. 25 x 25 µm AFM image of the surface of (c) nominally pure BTIBD Ge thin film and (d) BTIBD Si$_{0.45}$Ge$_{0.55}$ thin film.
post re-configuration and shows no metal contamination. Consequently, no as-deposited crystallisation of Ge films persisted once the contamination source was corrected and the Young’s modulus and hardness values for nominally pure contaminant free a-Ge are inline with rest of the sample set. As such, these outlying Ge data points are included in Figures 7.4 and 7.5, but are not used in determining the trend for Young’s modulus and hardness versus composition of the BTIBD a-Si<sub>x</sub>Ge<sub>1−x</sub> thin films.

**Figure 7.7:** XRD of BTIBD a-Si<sub>x</sub>Ge<sub>1−x</sub> samples on a glass substrate. The crystalline peaks are visible for (nominally) pure Ge film, while all other films exhibit only the amorphous hump.
7.4 Thin film induced substrate bending

7.4.1 Curvature and stress

Films deposited via BTIBD contain in-built internal stress as a result of the deposition process. The stress is often compressive due to the highly energetic deposition process. In this case the magnitude of this stress is dependent on the deposition conditions and, for $a$-$Si_xGe_{1-x}$, the composition.

Figure 7.8 shows the pre-deformation stress, or Stoney stress, versus silicon content for the films. Radius of curvature was measured for Si and Ge substrates cut into 3 mm x 15 mm test strips. Films of $a$-$Si_xGe_{1-x}$ of various compositions were deposited via BTIBD according to the recipes outlined in Table 5.3. Radius of curvature was measured post-deposition and the stress determined via Equation 3.3.1. The as-deposited films were highly compressive and showed, in general, an increase in compressive stress with silicon content.

![Stress vs Si Content](image)

**Figure 7.8:** Stoney stress for the BTIBD $a$-$Si_xGe_{1-x}$ thin films on thin Si (90 µm) and Ge (110 µm) substrates versus composition.
This increase in compressive stress with increasing Si content is as expected for the a-Si$_x$Ge$_{1-x}$ series. Compressive stress is often observed in BTIBD films due to the highly energetic deposition of the particles which forces more atoms into a given space, thereby compressing the interatomic spacing to less than their preferred orientation (see Section 3.3). The energies of a Ge-Ge and Si-Si bond present in the target material are 2.84 eV and 3.39 eV, respectively [260, 267]. These energies are far below the ion beam energy in a BTIBD system which ranges between 10 eV to 30 keV meaning bond strength is not a significant contributing factor in sputter species energies. Instead, momentum transfer is most efficient in elements of similar size resulting in maximum energy transfer for target element similar in size to the Ar$^+$ bombardment ions (Si in this case) and comparatively reduced energy transfer for target elements of dissimilar size (Ge in this case). Given this, it is expected that films composed predominantly of Ge will exhibit reduced compressive stress compared to film of predominantly Si composition.

### 7.4.2 Coefficient of thermal expansion

CTE is determined for BTIBD a-Si$_x$Ge$_{1-x}$ thin films in the same manner as for ICPCVD a-Si:H film as described in chapter 3 and chapter 5, through measuring the curvature of film-substrate bilayer samples as a function of temperature. Two different substrates were used, 90 $\mu$m thick Si(100) and 110 $\mu$m thick Ge(100). The wafer substrates were cut into strips approximately 3 mm x 15 mm in dimension with the length aligned to the <110> crystallographic direction. The a-Si$_x$Ge$_{1-x}$ thin films were deposited onto the substrates using the 4Wave BTIBD system following the deposition duty cycles and conditions summarised in Table 5.3, with a resulting thin film thickness of varying from 650 to 800 $\mu$m.

Curvatures of the substrate strips were measured prior to film deposition. The primary axes of the samples were aligned along the <110> directions of the substrates. Curvature data measured as a function of temperature from room temperature (300 K) down to 85 K. At each temperature curvature data were recorded at each of the measurement locations shown in Figure 5.15.

Figure 7.9 shows the effect of temperature on the measured curvature and the
corresponding change in stress with temperature of the BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} thin films of various compositions. Figure 7.9 (a) represents varying compositions of BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} deposited on the Si(100) substrate and Figure 6.5 (b) represents varying compositions of BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} deposited on Ge(100) substrates.

From the data in Figure 7.9 we can infer the nature of the CTE of the BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} films from the slope of the stress versus temperature. In both cases (a-Si\textsubscript{x}Ge\textsubscript{1−x} deposited on Si(100) and Ge(100) substrates) there is a negative correlations between stress and temperature. From Equation 3.4.1, a negative \(\frac{d\sigma}{dT}\) means the deposited film has a CTE greater than the substrate. The relatively flat slopes of BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} on Ge(100) (Figure 7.9(b)) indicates that deposited films of all compositions have a CTE relatively close to the value of CTE for the Ge(100) substrate. The relatively large negative slopes of the stress versus temperature data for BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} films on Si(100) (Figure 7.9(b)) indicate that deposited films of all compositions have a CTE significantly greater to the substrate.

A linear fit of the temperature dependence of stress, \(\sigma = f(T)\), was applied to the data shown in Figure 7.9. The temperature dependent stress \(d\sigma_1/dT\) for the a-Si\textsubscript{x}Ge\textsubscript{1−x}/Ge(100) sample and \(d\sigma_2/dT\) for the a-Si\textsubscript{x}Ge\textsubscript{1−x}/Si(100) sample were then used in Equation 3.4.4 to calculate the temperature dependent CTE, \(\alpha_f(T)\), for the films. Figure 7.10 shows the \(\alpha_f(T)\) for the a-Si:H films. Also plotted in Figure 6.6 is the CTE of Ge(100) wafer in the <110> directions, as expressed in Table 6.1, for comparison. It is observed that all films have a CTE value greater than the Ge(100) substrate and as the Si content of the BTIBD a-Si\textsubscript{x}Ge\textsubscript{1−x} films increases the CTE decreases.

It is postulated that the increased CTE of the deposited a-Si\textsubscript{x}Ge\textsubscript{1−x} films compared to crystalline silicon and germanium is attributed to the inclusion of deposition gasses. The gas inclusions typical of CVD and PVD techniques may have a profound impact on the CTE of both BTIBD films containing Ar and the ICPCVD films containing H. However, literature reports of the impact of plasma gas species on the thermal expansion behavior of thin films has not, as of yet, been presented. Of relevance to this work and the theme of reduction of stress varia-
Figure 7.9: Stoney stress versus temperature for the BTIBD a-Si$_x$Ge$_{1-x}$ films with Si content as indicated in the figure for (a) films on 90 $\mu$m Si(100) substrates and (b) films on 110 $\mu$m Ge(100) substrates.
Figure 7.10: Experimentally determined CTE for BTIBD a-Si$_x$Ge$_{1-x}$ thin films. CTE of the films decreases with increasing Si content. The CTE for Si(100) and Ge(100) wafers are also reported for reference [195, 197].
tion across temperature range, if BTIBD a-Si$_x$Ge$_{1-x}$ films are to be implemented as structural components for low temperature MEMS it may be necessary to use Ge or other alternative substrates.

## 7.5 Resonance of thin film microcantilevers

As with ICPCVD a-Si:H, determination of Young’s modulus, shear modulus, and Poisson’s ratio was achieved through characterisation of cantilever vibration using the methods outlined in Section 5.5. Thin films of various Si$_x$Ge$_{1-x}$ alloy compositions were deposited to a thickness of 500 nm on Si (100) substrates, preceded by an SiO$_x$ etch stop layer according to the deposition conditions outlined in Table 5.3. Many die, each containing 9 microcantilever testing arrays (as depicted in Figure 5.12) were fabricated for each alloy composition in a single deposition event. The cantilevers were defined in a-Si$_x$Ge$_{1-x}$ layers using photolithography and surface dry etching with an Oxford Instruments PlasmaLab System 100 ICP-RIE system. This was followed by a deep selective cryogenic etch of the Si(100) substrate wafer according to procedures outlined in Section 5.1.3 and Table 5.3. The BTIBD Si$_x$Ge$_{1-x}$ had very similar etching characteristics to the ICPCVD a-Si:H thin films, and no adjustments to processing conditions were required.

### 7.5.1 Young’s modulus

Resonant frequencies for the first four bending modes are plotted for 5 different Si$_x$Ge$_{1-x}$ compositions in Figure 7.11 versus $t_f/L^2$, where $t_f$ is the film thickness and $L$ is the length of the cantilever. For a-Si$_{16}$Ge$_{84}$ and a-Si$_{38}$Ge$_{62}$ the first bending mode could not be resolved above the noise of the measurement are thus not presented. The Young’s modulus for a-Si$_x$Ge$_{1-x}$ is determined from fitting the frequency versus $t_f/L^2$ data using Equation 4.2.1. These fits are represented as the solid lines in Figure 7.11.

Figure 7.12 plots the Young’s modulus values determined from the fit of the bending resonance data shown in Figure 7.11. The Young’s modulus of BTIBD
Figure 7.11: Frequencies of the first four bending resonances for cantilever beams of various lengths plotted versus $t_f/L^2$ for different compositions of BTIBD a-Si$_x$Ge$_{1-x}$ as indicated in the figures. The data are fitted using Equation 4.2.1.
Si$_x$Ge$_{1-x}$ monotonically increases from 105 GPa for nominally pure Ge to 160 GPa for nominally pure Si. The dashed line represents the Young’s modulus values determined via nanoindentation for a-Si$_x$Ge$_{1-x}$ in Section 7.3. Young’s modulus values determined from the 1st bending mode have significant error compared to the rest of the sample set. Excluding these, the data taken from the bending resonance of the cantilevers is in excellent agreement with the nanoindentation measurement data.

The first bending mode produces a fitting error typically 5 to 10 times greater than the higher bending modes. This results from the first resonant bending mode frequency being on the order of only a few kHz. The measurement resolution of the test is 1 kHz, while the change in first bending resonant frequency measured $t_f/L^2 = 2$ and $t_f/L^2 = 30$ is typically 10 to 15 kHz. As such, a potential error of $\pm$ 1 kHz has the greatest impact on low frequency measurements. Increasing the measurement resolution could clarify first bending mode resonant
frequencies or, as is presented here, higher resonances can be evaluated to confirm the fit of Equation 4.2.1.

7.5.2 Shear modulus

The resonant frequency for the 1st torsional mode for various compositions of a-Si$_x$Ge$_{1-x}$ are shown in Figure 7.13 versus $t_f/L_w$. To determine the shear modulus of a-Si:H, the frequency versus $t_f/L_w$ data is fitted using Equation 4.2.2. As the Si content of the samples increases there see a gradual increase in the slope of the data. This is indicative of a shear modulus that increases with increasing Si content and is as expected if we look at the shear modulus ofbulk crystalline Si(100) and Ge(100) discussed in Section 3.2.1.2

The shear modulus, determined from the 1st torsional resonance data in Figure 7.13 for the BTIBD a-Si$_x$Ge$_{1-x}$ thin films, is presented in Figure 7.14 versus Si content. It is shown that shear modulus varies monotonically with composition from 45 GPa for nominally pure BTIBD Ge to 65 GPa for nominally pure BTIBD Si. The shear modulus of BTIBD Si is significantly greater than the shear modulus for ICPCVD a-Si:H determined in Chapter 5. A larger value for the shear modulus of the BTIBD a-Si films compared to ICPCVD a-Si films is indicative of a deposition that results in fewer dangling bonds (Si-Ar for BTIBD films and Si-H for ICPCVD films) and more Si-Si bonds. An increased proportion of Si-Si bonds would correspond to reduced degrees of freedom of the constituent atoms and therefore an increase in rigidity of the film. Additionally, the reported shear modulus trend for BTIBD a-Si$_x$Ge$_{1-x}$ is less than the shear modulus given by Wortman et al. [128] and Hopcroft et al. [127] for bulk crystalline Si(100) and Ge(100). As such, it would appear that the value of shear modulus in the Si-Ge alloy is strongly dependent on formation conditions of the solid.
Figure 7.13: Plot of the first torsional resonance versus $t_f/L_w$ for beams of various lengths and widths for different compositions of BTIBD a-Si$_x$Ge$_{1-x}$ as shown in the figure. The data is fitted using Equation 4.2.2.
Figure 7.14: Shear modulus as determined from data in Figure 7.13 versus composition for BTIBD a-Si$_x$Ge$_{1-x}$. The shear modulus for crystalline Si(100), 80 GPa, and Ge(100), 65 GPa, are shown for reference [127, 128].
7.5.3 Poisson ratio

Poisson’s ratio of the a-Si$_x$Ge$_{1-x}$ thin films can be determined from the Young’s modulus and shear modulus via Equation 4.2.3. Using the shear modulus and a Young’s modulus determined above, the Poisson’s ratio versus composition is presented in Figure 7.15. The value of Poisson’s ratio, $\nu$, for BTIBD a-Si$_x$Ge$_{1-x}$ is constant with Si content with an average $\nu$ of 0.21 ± 0.06. For BTIBD a-Si$_x$Ge$_{1-x}$, $\nu$ is less than $\nu_{yz}$, $\nu_{xz}$, and $\nu_{zy}$ for bulk crystalline Si(100) and Ge(100), (see Section 3.2.1.3) which are plotted as a range in Figure 7.15, but is greater than $\nu_{xy}$ of bulk crystalline Si (100) and Ge (100). As amorphous films are isotropic, having no directionally dependent properties, it is reasonable that $\nu$ for BTIBD a-Si$_x$Ge$_{1-x}$ would fall between the range of possible values for single crystal Si and Ge. Additionally, this value is very similar to $\nu$ reported for ICPCVD a-Si:H and is in agreement with previously reported values for thin film Si and Ge materials [133–138].

![Figure 7.15: Poisson ratio as determined from the bending and torsional resonance frequencies via Equation 4.2.3 versus composition of the BTIBD a-Si$_x$Ge$_{1-x}$ thin films. Poisson ratio of ICPCVD a-Si:H reported previously and literature reported Poisson ratio of crystalline Si(100) and Ge(100) are shown for reference [127, 128, 153].](image)
7.6 Direct tensile testing of thin film strength

Strength of a-Si$_x$Ge$_{1-x}$ thin films is determined using the techniques described in Section 5.7. Microbridge samples of eight different \( w_2/w_1 \) step-down ratios were fabricated, including \( w_2/w_1 \) ranging from 2 to 9 in integer steps. The microbridges were fabricated of approximately 500 nm thick a-Si$_x$Ge$_{1-x}$ thin films with 5 different Si:Ge ratios (Si, Si$_{75}$Ge$_{25}$, Si$_{48}$Ge$_{52}$, Si$_{30}$Ge$_{70}$, and Ge), deposited by means of BTIBD on 300 \( \mu \)m Si(100) wafer substrate using a 4Wave BTIBD system. Prior to a-Si$_x$Ge$_{1-x}$ deposition, a 200 nm SiO$_x$ layer was deposited on the bare substrate. This layer provides the required stop-etch layer during back-side etching of the silicon substrate. Deposition conditions for the a-Si$_x$Ge$_{1-x}$ films are summarised in Table 5.3, and the SiO$_x$ layer is summarised in Table 5.2.

As with the a-Si:H samples from chapter 6, microbridge samples were fabricated from the deposited a-Si$_x$Ge$_{1-x}$ films by means of reactive ion etching in SF$_6$ and O$_2$ through a negative photoresist mask. Releasing of the microbridges was achieved though bulk micromachining of the Si(100) wafer using a highly selective cryogenic etch process described in Section 2.2.2.2. Samples were cooled to -110 °C with He backing to maintain sample temperature. The Si wafer substrate was etched using a SF$_6$ and O$_2$ gas chemistry under DC biasing. This etch stopped on the SiO$_x$ etch stop layer, which was later removed using a CF$_4$ etch at room temperature. Etch recipes are summarised in Table 5.1.

Following release, the samples were hotplate annealed at 350 °C for 30 minutes. This was sufficient to render the microbridges flat for all compositions. The exception being nominally pure Si, which retained a slight vertical displacement after hotplate annealing of approximately 1 \( \mu \)m for each microbridge as shown in Figure 7.16. This displacement corresponds to a pre-release compressive stress of 50 MPa as determined from thin film induced substrate bending for a witness BTIBD a-Si on 70 \( \mu \)m Si(100) wafer samples. Since the compressive samples are free to deform, thereby relieving any residual compressive stress, no residual stress must be accounted for when determining the film strength given the 1D analytical approximation in Equation 5.7.2. However, in Equations 5.7.2 '\( h \)' is equal to the displacement recorded by the nanoindenter plus any pre-test displacement.
of the sample determined from optical profilometry. In Equation 5.7.1, $\theta$ must also be determined from the sum of the indenter height and the pre-test displacement. For FEM analysis, additional pre-load stress was added to the model.

The fabricated microbridges were tested using a Hysitron TI 950 TriboIndenter fitted with a wedge-shaped diamond tip indenter. The wedge tip had an edge length of 200 $\mu$m width a tip-edge radius of 650 nm. The deformation was conducted under load control, and the load-displacement curves for the microbridges were recorded and later converted to stress-strain data for the necked gauge sections using Equation 5.7.1, Equation 5.7.2, and Equation 5.7.3.

For each a-Si$_x$Ge$_{1-x}$ composition, up to five identical wafers were processed, each containing two arrays of eight microbridges with the eight $w_2/w_1$ ratios. For all microbridges $L = 70 \mu m$ or 100 $\mu m$, $w_1 \approx 7 \mu m$, and $w_2$ varied from 16 $\mu m$ to 90 $\mu m$, giving $w_2/w_1$ a range of 2 to 12. As in chapter 6 for a-Si:H, post process SEM micrographs of the released microbridges were used to define the geometry of models for FEM of the a-Si$_x$Ge$_{1-x}$ microbridges. The CoventorWare2014$^\text{TM}$FEM suite was used for the simulations. A load was applied mimicking a nanoindentation load giving a FEM load-displacement curve. From the FEM output, stress distribution across deflecting microbridges at the moment of rupture were anal-
Chapter 7: Properties of BTIBD a-Si$_x$Ge$_{1-x}$ thin films

Figure 7.17 shows the load-depth curves for a range of $w_2/w_1$ ratios for various a-Si$_x$Ge$_{1-x}$ compositions. The solid line indicates the load-depth curves produced by the nanoindentation system and the discrete points represent the FEM simulation. The nominal $w_2/w_1$ ratio for each sample is listed in the figure next to the corresponding curve and FEM result. The FEM load versus displacement of the microbridges is well fitted to the experimental data. Strength of the microbridges was determined from the displacement at the moment of rupture. Equation 5.7.2 was used for the 1D analytical approximation of strength assuming uniform stress distribution across the width of the microbridge. For the FEM data presented in Figure 7.17, the microbridge stress distribution was evaluated at the displacement, $h$, of experimental rupturing.

Figure 7.18 shows the ultimate rupture strength of the various Si$_x$Ge$_{1-x}$ compositions versus $w_2/w_1$ ratio as determined from experimental rupturing. Correlation analysis for each of the five compositions showed no statistically significant correlation between strength and $w_2/w_1$ ratio. There is a significant spread in the strength of microbridges as evident in the error bars in Figure 7.18. Some microbridges had a measured strength over 2.5 GPa while some only showed a strength of 0.5 GPa. This large spread indicates a potential for increased strength in BTIBD films compared to ICPCVD a-Si:H films which demonstrated a maximum rupturing strength of less than 2 GPa. The mechanism for low load rupturing of the samples is unclear as no significant surface or edge defects were apparent in the samples prior to rupturing.

A summary of the tensile strength of BTIBD a-Si$_x$Ge$_{1-x}$ versus Si content is shown in Figure 7.19. The 1D analytical solution for strength for a-Si$_x$Ge$_{1-x}$ microbridges (Equation 5.7.2) gives an average of 1.4 GPa $\pm$ 0.5 GPa over the compositional range. The average maximum strength observed at the moment of rupturing as determined through FEM for the microbridges was 1.6 GPa $\pm$ 0.3 GPa. To determine if a statistically significant correlation exists between composition and strength, analysis is performed of the full data set. It is found that there is a statistically significant negative correlation between Si content of BTIBD
Figure 7.17: Load versus depth (P-h) curves for microbridges of various compositions of a-Si$_x$Ge$_{1-x}$. From top to bottom, Ge, Si$_{0.48}$Ge$_{0.52}$, Si$_{0.75}$Ge$_{0.25}$, and Si$_{0.98}$Ge$_{0.02}$. The solid line represents the experimental data and the discrete points represent the FEM data. Step down ratios are for $w_2/w_1 = 2, 4, 6,$ and 8 as indicated in the figure. The applied load function for the experimental data is shown in the inset.
Figure 7.18: Strength at rupturing as determined by the 1D analytical approximation versus $w_2/w_1$ ratio for various compositions of a-Si_xGe_1-x thin films.
a-Si$_x$Ge$_{1-x}$ thin films and tensile strength, r(58) = -0.38, p<0.05. While the correlation is weak due to significant spread in the data, ultimate rupture strength of a-Si$_x$Ge$_{1-x}$ reduces with increasing Si content.

Looking at bond energy of a Ge-Ge and Si-Si bond (2.84 eV and 3.39 eV, respectively [260, 267]) with silicon also having a stiffer bond and higher melting point [268], it would be expected that Si would demonstrate an increased strength over Ge films. However, strength is not perfectly correlated with bond stiffness in a material. Since strength of thin films is strongly dependent on the deposition process, and a-Si$_x$Ge$_{1-x}$ undergo slight variation in deposition conditions, it is reasonable to assume that the strength variation observed across the compositional range is a result of these varying deposition conditions. Deposition of a-Si$_x$Ge$_{1-x}$ via other methods such as ICPCVD or E-beam would be required to confirm if the correlation observed is a result of processing. With such large variation in strength for a given composition and deposition even, it would stand to reason that there is a potential for increase strength in Si:Ge film using BTIBD compared to ICPCVD techniques.

**Figure 7.19:** Strength versus composition for BTIBD a-Si$_x$Ge$_{1-x}$ thin films. The experimentally determined strength given by the 1D analytical solutions, as well as maximum and minimum FEM stress in the neck gauge region of the microbridge at rupturing displacement is shown.
7.7 Summary

A summary of the material properties determined in this chapter for BTIBD a-Si$_x$Ge$_{1-x}$ thin films is listed in Table 7.1.

The quartz crystal microbalance was used to determine the density of BTIBD a-Si$_x$Ge$_{1-x}$ thin films. Density ranged linearly from 4.68 g/cm$^3$ for nominally pure Ge to 2.63 g/cm$^3$ for nominally pure Si films. The density of the BTIBD a-Si film being greater than single crystal silicon is attributed to the Ar content of the film. This also explains the reduced density of BTIBD a-Ge compared to single crystal Ge, as the lower atomic weight of Ar inclusions compared to Ge in the film would reduce the overall film density.

The curvature measurements of as-deposited BTIBD a-Si$_x$Ge$_{1-x}$, reported in Section 7.4.1, show that there is a strong correlation between composition and pre-deformation stress. This is attributed to the energy transfer efficiency between Ar$^+$ bombardment ions and the sputtering targets atoms of Si and Ge. Si is most similar in size to the Ar$^+$ bombardment ions, resulting in greater energy transfer and a more energetic deposition species. As such, compressive stress increases with increasing Si content.

Evaluation of nanoindentation, reported in Section 7.3, showed that the Young’s modulus and hardness of BTIBD a-Si$_x$Ge$_{1-x}$ varies linearly with composition. The value of Young’s modulus for BTIBD a-Si$_x$Ge$_{1-x}$ is 115 GPa for nominally pure BTIBD Ge and 160 GPa for nominally pure BTIBD Si. The hardness follows a similar trend with BTIBD Ge having a hardness of 6.6 GPa and BTIBD Si having a hardness of 9.8 GPa.

Microbridge fracturing in Section 7.6 showed that BTIBD a-Si$_x$Ge$_{1-x}$ thin films deform in tension in a purely elastic manner to brittle rupture. A tensile strength of 1.6 GPa ± 0.4 GPa for BTIBD Ge and 1.1 GPa ± 0.3 GPa for BTIBD Si was determined. While the bond energy of Si-Si bonds is greater than Ge-Ge bonds, bond strength is only an indicator of material strength. The BTIBD Si had a reduced strength compared to ICPCVD Si. However, the large spread in data, ± 0.4 GPa for BTIBD films compared to ± 0.1 GPa for ICPCVD films, indicates a potential
to increase the strength in films deposited via BTIBD compared to ICPCVD if the source of early rupturing can be isolated.

The CTE of BTIBD a-$\text{Si}_x\text{Ge}_{1-x}$, reported in Section 7.4.2 was determined to be greater than the CTE value for both the Si (100) and the Ge (100) substrates. It is hypothesised that the deposition gas inclusions in the films may significantly impact the thermal expansion of the material. This would need to be explored as there are no literature reports on the effect of deposition gas inclusions on the thermal expansion of thin films. A CTE of BTIBD a-$\text{Si}_x\text{Ge}_{1-x}$ significantly greater than that of the Si(100) substrate is of critical importance in the greater theme of this work of reducing stress in MEMS that must be operational over large temperature ranges. Use of a Si (100) substrate would result in significant additional stress from the large CTE mismatch, and thus alternative substrates would need to be employed.

Young’s modulus, shear modulus, and poisson ratio were determined from the resonant vibration of microcantilever beams in Section 7.5. Young’s modulus for BTIBD a-$\text{Si}_x\text{Ge}_{1-x}$ was determined to range from 105 GPa for nominally pure Ge to 160 GPa for nominally pure Si. These values are in excellent agreement with the Young’s modulus determined through nanoindentation, which ranged from 115 GPa to 160 GPa. The shear modulus was determined to vary linearly with composition from 45 GPa for Ge to 65 GPa for Si. Given the Young’s modulus, shear modulus, and Equation 4.2.3, a Poisson’s ratio of $0.21 \pm 0.06$ was determined across the full compositional range for BTIBD a-$\text{Si}_x\text{Ge}_{1-x}$ with no strong correlation to Si content of the films.
Table 7.1: Summary of Material properties of BTIBD a-Si$_x$Ge$_{1-x}$ thin films

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Measurement Technique</th>
<th>Section</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$</td>
<td>115 to 160 (GPa)</td>
<td>Nanoindentation</td>
<td>7.3</td>
</tr>
<tr>
<td>$E$</td>
<td>105 to 160 (GPa)</td>
<td>Cantilever vibration</td>
<td>7.5</td>
</tr>
<tr>
<td>$G$</td>
<td>45 to 65 (GPa)</td>
<td>Cantilever vibration</td>
<td>7.5</td>
</tr>
<tr>
<td>$H$</td>
<td>6.6 to 9.8 (GPa)</td>
<td>Nanoindentation</td>
<td>7.3</td>
</tr>
<tr>
<td>$ν$</td>
<td>$0.21 \pm 0.06$</td>
<td>Cantilever vibration</td>
<td>7.5</td>
</tr>
<tr>
<td>$σ_T$ (strength)</td>
<td>1.6 to 1.1 (GPa)</td>
<td>Microbridge fracturing</td>
<td>7.6</td>
</tr>
<tr>
<td>$α$(T=300K)</td>
<td>$7.1 \times 10^{-6}$ to $6.38 \times 10^{-6}$ K$^{-1}$</td>
<td>Substrate bending</td>
<td>7.4.2</td>
</tr>
<tr>
<td>$ρ$</td>
<td>4.68 to 2.63 (g/cm$^3$)</td>
<td>QCM</td>
<td>6.5</td>
</tr>
</tbody>
</table>
CHAPTER 8

Actuation geometries for large area MEMS

The general MEMS microspectrometer concept developed at UWA consists of a tunable MEMS optical filter (described in detail in Chapter 1.2.1 placed optically ahead of an optical sensing element [18, 30, 32, 51, 52, 55]. Large area MEMS for actutable filter applications, allow for the expansion of such spectrometer function from 0D single-point sensing to 2D imaging.

A typical design for 200 µm x 200 µm filter using fixed-fixed beam support and actuation structure is shown in Figure 8.1(a) and the scaled up 1 mm x 1 mm filter is shown in Figure 8.1(b). To maintain a flat membrane, the film is deposited under some tension. Maintaining a flat membrane for increasingly large membranes increases the tension at the anchors resulting in delamination. Actuation and support structures that perform well for small filter sizes did not translate to scaled up versions. Early prototypes showed that more rigid supports are required to eliminate actuation beam buckling and failure from anchor delamination. The use of finite element modeling (FEM) enables the pre-fab investigation of specific design features, in order to reduce time and cost of device design refinement. Simplified modeling features are used to reduce computational time, enabling faster turnaround on proposed structures.

The devices presented are modelled on a 300 µm Si(100) substrate, with a 1 µm a-Si thin film as the structural layer, and 40 nm thick Au upper and lower electrodes for actuation. The CoventorWare 2013 FEM software package was used.
Figure 8.1: (a) typical size and form of a 0D point detection filter with membrane of 200 µm in diameter, (b) Scaled up filter using post and beam support construction, and (c) design proposed in this work for ridged bench style support and actuation.

for the simulation. The material properties for the Si substrate and Au electrodes were provided by CoventorWare software package. The material properties for the a-Si layer were taken from the work presented in Chapter 5.

8.1 Support and actuation structures

8.1.1 Post-and-beam support structures

One actuation and support structure often used for actutable filters is the post and beam with "top-hat" feature. Figure 8.2 shows a schematic of the key design features of the top-hat anchor. Previous work has shown this structure to be very strong and stable [269, 270]. It is desired to use such a feature in our design, but modeling it is computationally time expensive due to the round features. To evaluate the accuracy of simplifying modeling features, a post-and-beam with round features and one with square features were modelled. Various dimension of top-hat flanges were trial to investigate the strain reducing nature of this feature and potential for improved stability of actuation.
Figure 8.2: Schematic of a top-hat style post and beam with top hat, post, anchor, and beam labeled for reference throughout Chapter 8. The cross-section indicated by the red dashed line and shown in the bottom right, highlights the design profile of this style of anchor with 1, 2, and 3 indicating the 3 top-hat flange dimensions investigated.

The footprint, or area, of the post is the same for the round and square designs. For each feature class three examples were modelled as shown in Figure 8.3: 1) no top-hat, 2) top-hat flange with area of $1200 \, \mu m^2$, and 3) top-hat flange with area of $3200 \, \mu m^2$. The beam lengths were $250 \, \mu m$ from post center to post center. The mesh size was chosen at a point where the increase in computation time was much greater than the increase in result resolution for snap down approximation from the addition of mesh points as discussed in Section 5.2. For the round configurations, Fig 8.3 (α 1 - 3), the round features limit the options for meshing the structure. The computationally expensive tetrahedral mesh, with 10 $\mu$ size mesh elements, was chosen. For the square configuration, Figure 8.3 (β 1 - 3), a brick mesh of $5 \, \mu m \times 10 \, \mu m \times 1 \, \mu m$ was used for FEM. From this sample set the dynamic actuation range for an applied voltage under various conditions is investigated to probe the equivalence of the round and square featured structures and the over-all impact of the top hat flange.
Figure 8.3: Images of round (α) and square (β) post structures with 3 variations in top hat size. The footprint of features are the same for the round and square designs. The anchor footprint is 400 $\mu$m$^2$ for all models. The top-hat flange footprint, from top to bottom, is 0 $\mu$m$^2$, 1200 $\mu$m$^2$, and 3200 $\mu$m$^2$. The square configuration is meshed with a 5 $\mu$m x 10 $\mu$m x 1 $\mu$m brick mesh. The round configuration is meshed with a 10 $\mu$m sized tetrahedral mesh. The location of the tether for attaching the suspended membrane is highlighted for each fixed-fixed beam.
8.1.2 *Rail support*

Fabrication of early designs for large area filters demonstrated that scaling up of filters using the post and beam design resulted in delamination of the post anchors. To compensate for the additional stress in a large area suspended membrane one option is to increase the footprint of the post anchors. As you continue to increase the size of the suspended membrane, the size of the post anchor must also increase to a point where the radius of the post must be as large as the post center-to-center spacing, reducing the beam length to zero. This is obviously not feasible.

An alternative design to a post and beam configuration is the rail support structure pictured in Figure 8.4. The anchor spans the length of the actuator. The anchor can also extend out perpendicular to the sidewall as much as is required, or space allows, to further reduce delamination. The first iteration of the rail design consisted of bridging arms between the sidewall and the actuator and a tab connecting the filter to the support structure. The second iteration further simplified the design by creating one continuous device with the filter connecting to the support along the entire length.

![Diagram](image-url)

*Figure 8.4:* Images of the two rail type structures, $\delta$ an open rail, and $\gamma$ a closed rail or "bench". Membrane attachment point for rail $\delta$ and attachment edge area for rail $\gamma$ is highlighted.
8.2 Finite element modeling of dynamic actuation range

One of the major motivators of this study is reduced computation time of proposed device designs. In CoventorWare, round structures are computationally expensive due to the mesh options available for these shapes. Table 8.1 lists all the simulations discussed in detail later in this chapter and their total run times for each support configuration. It is observed that simulation run time for the round configurations is 5 to 20 times greater than that of the device structures with square features.

<table>
<thead>
<tr>
<th>Run time (hours)</th>
<th>round</th>
<th>square</th>
<th>rail</th>
</tr>
</thead>
<tbody>
<tr>
<td>α1</td>
<td>α2</td>
<td>α3</td>
<td>β1</td>
</tr>
<tr>
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<td>25</td>
<td>39</td>
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</tr>
<tr>
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<td>10</td>
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<tr>
<td>Section 8.2.3</td>
<td>4.5</td>
<td>7.0</td>
<td>10</td>
</tr>
</tbody>
</table>

8.2.1 Effect of temperature on snap-down

For devices that must be operational over a wide range of temperatures, the operating temperature will augment the actuation behavior of the device. The effect of temperature on pull-in (or snap-down) of the support structures was investigated using FEM. Eight different support structures were examined at temperatures ranging from 77 K to 300 K. A voltage differential is applied between the upper and lower electrode with the magnitude of the voltage used for each support structure depended on the force required to produce snap down for that structure. Figure A.1 (located in Appendix A) shows the temperature dependent snap-down behavior for the 8 different support structures as summarised in the following.

Figure 8.5 show the snap-down and maximum displacement versus temperature for the round and square post and beam actuators, respectively. In the pull-in versus temperature analysis, a correlation between snap down voltage and
Figure 8.5: (a) Voltage at snap-down versus temperature and (b) maximum displacement at snap-down versus temperature for round ($\alpha$ 1 - 3) and square top-hat ($\beta$ 1 - 3) style fix-fixed beams.

Top-hat size was observed. This was observed in both the round and square featured actuators. In both cases there is an increase in voltage requirements to reach snap down with increasing top-hat area. All configurations also show a slight reduction in snap-down voltage and maximum displacement at low temperatures. There is no clear correlation in the maximum displacement with top-hat area.

If we compare the square to the round configuration for each top-hat size, we see that the snap down voltage and maximum displacement are nearly identical. As the size of the top-hat increases the small discrepancy between square and round configurations reduces further. The small difference is likely due to the small difference between the brick mesh used in the case of the square top-hat
and the tetrahedral mesh used in the case of the round top-hat.

Figure 8.6 shows a) the maximum displacement versus temperature and b) the snap-down voltage versus temperature for the two rail style supports. Figure 8.6 also includes the 1200 \( \mu \text{m}^2 \) round top-hat post and beam support for comparison. For the maximum displacement, the second rail structure (\( \gamma \)) (Figure 8.6(b)) appears to result in greater maximum displacement. In the case of the snap-down voltage versus temperature, the rail \( \gamma \) is observed to require much greater voltages to produce snap-down, while the rail \( \delta \) structure reduces the voltage requirement slightly.

![Figure 8.6: (a) pull-in and (b) maximum displacement versus temperature for the two rail designs and the round top-hat fixed-fixed beam version \( \alpha_2 \).]
8.2.2 Residual stress and snap-down

Figure 8.7 show (a) snap-down voltage and (b) maximum displacement versus residual stress for the round and square configurations, respectively. As the residual stress increases from 0 MPa to 300 MPa the voltage requirement for snap down increases from roughly 15 V to 50 V. If the residual stress in the support material is increased there is a significant change in the actuation voltage required to reach maximum displacement for all top-hat style fixed-fixed beams. The snap down voltage versus residual stress are very similar for square and round top-hat configurations, with the disparity between the two increasing at large residual

![Figure 8.7: Pull-in voltage and maximum displacement versus residual stress for the three round featured fixed-fixed beam actuations structures. Pull-in voltage and maximum displacement versus residual stress for the three square featured fixed-fixed beam actuations structures.](image)
stress. A trend towards increased maximum displacement with increases residual stress is also observed for both configurations. No strong correlation between top-hat configuration and maximum displacement versus residual stress is evident as the scatter in the data is significant.

Figure 8.8(a) shows the maximum displacement versus residual stress for rail δ, rail γ, and fixed-fixed beam α2. In this case the rail structures exhibit, on average, 100 nm greater maximum displacement than the post and beam. The snap-down voltage versus residual stress is shown in Figure 8.8(b). The rail supports show very stable actuation behavior, with no significant change in snap-down voltage with increasing residual stress. This steady behavior is in strong contrast to the behavior of the post and beam structure.

Figure 8.8: (a) Maximum displacement and (b) pull-in versus residual stress for the two rail designs and the round top-hat fixed-fixed beam version α2.
8.2.3 Direct membrane strain and snap-down

The third analysis for the support structures is the addition of stress from a suspended membrane. The membrane stress is simulated by applying a static load normal to the face of the tether as shown in Figure 8.9 for rail δ and fix-fixed beam α2. Figure 8.10 presents the snap-down voltage and maximum displacement versus membrane stress for the square and round post-and-beam configurations, respectively. The snap-down voltage shows a slight increase with increased membrane strain for both configurations. There is also a trend towards reduced maximum displacement with increased membrane strain for both the square and round post and beam configurations.

Figure 8.9: The twisting effect observed in fixed-fixed beam actuators and the rail δ actuator due to perpendicular loading as indicated by the arrow.
Figure 8.10: Pull-in voltage and maximum displacement versus membrane strain for the three round featured fixed-fixed beam actuations structures. Pull-in voltage and maximum displacement versus membrane strain for the three square featured fixed-fixed beam actuations structures.

Comparing the square and round configurations for each top hat size we see that the data for each top-hat size is nearly identical. In each case the behavior of the square and round posts are satisfactorily similar. In each case we also note a slight trend towards increased snap-down voltage and a decrease in maximum displacement with increasing membrane strain.

Looking now to the rail structure we see a different story. Figure 8.11(a) shows the displacement versus membrane strain and Figure 8.11(b) shows the snap-down voltage versus membrane strain for the rail style support structures. Figure 8.11(a) we see that for the second rail structure the maximum displacement
Figure 8.11: (a) Maximum displacement and (b) pull-in versus membrane strain for the two rail designs and the round top-hat fixed-fixed beam version α2.

actually increases with increasing membrane strain. Additionally, for the case of snap-down voltage, Figure 8.11(b) shows a decrease in voltage requirements with increased membrane strain for the second rail configuration. In both displacement and snap-down the first rail configuration behaves very similarly to the post-and-beam structures.

8.2.4 Summary

When comparing the round to square structures, in the context of this work, there are great benefits to approximating rounded features with square ones. The
use of square features resulted in 5 to 20 times less computation time as their round counter part, with no significant difference in the model behavior across any test condition. For large scale devices, exchanging curves for squares, could reduce simulation time down from weeks to just hours without the need for additional computer capabilities. It is important to recognise that exchanging a circle for a square is not the same as exchanging a circle for a high number polygon structure, as this exchange increases the required mesh points and would actually increase the computation time.

When scaling up micron size suspended membranes to millimeter scaled membranes, alternative support structures are required to cope with the additional strain. The proposed structure, a rail design, gives increased anchor area and improved stability across all test conditions. When exposed to large temperature changes the Rail $\gamma$ structure maintained consistent displacement and actuation voltage, while other structures showed a decrease in maximum displacement and an increase in voltage requirements at low temperatures.

When applying a large tensile stress both rail designs maintained consistent displacement and actuation voltage requirements, whereas the post-and-beam structures reported significant changes in voltage requirements. If we reflect on the structures, however, one would anticipate that in a complete device configuration the voltage requirements of the rail structure would also increase with increasing residual stress. The stand-alone rail is akin to a large cantilever. However, with the addition of a membrane and rail on the opposing side, it would behave under stress similarly to a fixed-fixed beam structure.

The application of a membrane strain also demonstrates the improved stability of the rail structure. With increasing strain from an attached membrane the maximum displacement actually increases in Rail $\gamma$, where it decreases in all other cases. It would also appear that this structure can withstand much higher strain, with the simulation not failing until much higher applied load values than in all other cases.

It is worth noting the reduced voltage requirements of Rail $\delta$ and the increased voltage requirements of the Rail $\gamma$ structure observed in Figures 8.6(a), 8.8(a), and
8.11(a). The increased voltage requirement for Rail $\gamma$ results from the support much more closely approximating a very short wide cantilever. Two ways to reduce voltage demand in actual device fabrication would be to thin the support or to add etch holes between the electrode and the sidewall. The addition of etch holes is effectively finding a balance between the reduced voltage requirements of Rail $\delta$ and the superior structural and actuation performance of Rail $\gamma$.

8.3 FEM of large suspended membranes

A 1 mm by 1 mm membrane suspended by each support class demonstrates the impact of each structure on the flatness of the membrane. Figure 8.12 shows the 1200 $\mu$m$^2$ round top-hat post-and-beam and both rail structures. A 100 MPa tensile stress has been added to the a-Si material. We observe reduced membrane buckling with the second rail device as compared to all other structures. We observe that the displacement in the membrane for the second rail structure is greater than the total achievable displacement of the electrode (as also noted in Figure 8.15), where as the membranes for all other designs are only able to deflect as much as the actuator deflects. This unique behavior is due to the cantilever motion of the actuator arm being perpendicular to the membrane edge as opposed to running parallel with it, as is the case with fixed-fixed beam actuators.

Typical electrostatic actuators employed to move suspended membranes take the form of fixed-fixed beams, or doubly supported beams. The basics regarding the electrostatic actuation of a fixed-fixed beam were discussed in section 4.3 and section 4.1.2. Figure 8.13 shows how a fixed-fixed beam actuator can be applied to a suspended membrane filter for actuation. Figure 8.13(a) shows the $\cos^2$ beam profile created during actuation. Tethers attached at the center point of each beam attach to the filter to lower it. This form of actuator reaches a limit at 1/3 actuation, at which point snap down occurs.

A proposed method to extend the actuation range of a fixed-fixed beam is to split the electrodes as was demonstrated by Hung et al. [271] and shown schematically in Figure 8.14. This design employs a leveraged bending method, where by
Figure 8.12: The deformation occurring in a membrane supported by the different support and actuation structures with a 100 MPa applied tensile stress. (top to bottom) Round fixed-fixed beam, square fixed-fixed beam, rail $\delta$, and rail $\gamma$ actuation supports with a suspended membrane.
electrostatic force is only applied to a portion of a cantilever or fixed-fixed beam, with the remaining non electrostatically active portion able to move through the snap-down displacement. Through this technique, Hung et al. were able to achieved a travel range of 1.75 \( \mu \text{m} \) from an initial gap of 2 \( \mu \text{m} \), or 87.5\% of the gap.

The proposed new design utilises the same idea behind the extended tuning range fixed-fixed beam demonstrated by Hung et al., but adapting it in a new way. Moving away from the beam and filter as two separate entities, we have
merged them into one. As pictured in Figure 8.15, our filter can be viewed as a very wide beam with split electrodes. The design utilises the leverage bending method via the electrodes about the perimeter to deflect the interior region of the membrane beyond the snap-down displacement.

The placement of the electrodes is important to achieve the actuation profiles pictured in Figure 8.15 (a) and (b) insets. If the electrodes are too close to the sidewall, there will be an increase in voltage requirements to actuate the filter. If the electrodes are placed too far from the sidewall, bending of the upper membrane will occur between the electrode and the side wall allowing for snap down without full range of motion. We have found, through fabrication and simulation, that approximately 100 µm from sidewall to top electrode works well for filters in the 1 mm x 1 mm size range made of a-Si:H of 250 nm to 1 µm in thickness.

As Figure 8.15 (c) shows, the anchor footing can effectively be the size of the entire substrate, addressing stress-induced film delamination, and producing a very robust device. This method of actuation does require increased voltages. However, with the additional of release holes patterned along the edge (between the upper electrode and side wall as shown) the voltage requirement drops significantly.
Figure 8.15: (a) Cross-section of the new filter design, highlighting the doubly supported beam with split electrode style configuration. The inset shows a cross-section of a finite element model simulation of a full filter being actuated. (b) and (c) show how this is extended to create a filter. The cut outs in the corner of (b) and (c) are used to relieve stress build up and allow the filter region of the device to remain flat. The inset in (b) shows how this design results in a very flat membrane across the full length and width of the device.
Figure 8.16 shows the FEM actuation displacement profile of the suspended membrane for the bench structure at room temperature and cryo. Actuation of the full initial gap height to 1 \( \mu m \) is demonstrated. The effect of cooling the device appears to result in strain stiffening which increases the voltage requirement for actuation from 5 V to 10 V. Since the curves in Figure 8.16 follow the membrane displacement which is enhance via the leverage bending mechanism, they do not follow the smooth curve trajectory typically shown for fixed-fixed beam actuation.

This new structure has many advantages over previous iterations:

1. Tuning range capable of reaching 100% of the gap without snap down;

2. The placement of upper and lower electrodes pulls the filter region of the device flat, eliminating any bowing effects at large tuning (Figure 8.15 (a) inset for reference);

3. Moving away from a post and beam design also has allowed us to extend the size limits of the device with much more rigid anchor points;

4. Structural stability allows for increased tensile stress in films without delamination of anchors, which leads to increased stability across large temperature ranges (i.e. no buckling at cryogenic temperatures).
8.4 Realisation of large area, actutable filter

Prototype actuators have shown the newly proposed structure to provide the added stability predicted by the FEM simulations described previously. Figure 8.17 shows optical profilometry micrographs of a prototype filter showing very flat central membranes on the multi-millimeter scale. Precise dimensions of the filter have been removed as this is proprietary information.

![Optical profilometry image of a released membrane suspended using the rail support structure (rail $\gamma$) and profile of the bisecting lines showing an extremely flat membrane of a film under approximately 50 MPa tension.](image)

**Figure 8.17:** Optical profilometry image of a released membrane suspended using the rail support structure (rail $\gamma$) and profile of the bisecting lines showing an extremely flat membrane of a film under approximately 50 MPa tension.
Actuation of the filter also showed movement beyond the typical 1/3 snap-down displacement with full recovery of the device after actuation. Figure 8.18 shows normalised transmission spectrum for various voltages. The spectrum has been normalised such that the spectral wavelength is given as a fraction of the initial gap height due to proprietary restrictions on the tuning range for this particular device. The substrate that the filter was deposited on had a cut off coating at approximately \( \lambda / h_o = 1 \) and \( \lambda / h_o = 1.7 \). We can see that at 5V the peak is somewhere below \( \lambda / h_o = 1 \) indicating actuation beyond 50% of the initial gap height. This was followed by full recovery of the filter indicating no snap down at extended actuation ranges.

![Figure 8.18: Spectrum for rail γ support type filter showing actuation beyond 50% of initial gap \( h_o \) followed by full recovery of the filter.](image)

Tuning range as a factor of initial gap height, \( h_o \), is shown in Figure 8.19 for state of the art tunable filters, as well as the filter developed here. Compared to state of the art Fabry-Pérot type tunable filters, the device prototype presented here out performs devices in terms of total optical area and tuning range. Further testing and optimization of this prototype device is underway with the basic design features being implemented across the research group [272, 273].
Figure 8.19: Normalised tuning range as a factor of initial gap, $h_o$. Data shown for this work, other works within the UWA research group [272–274], and other works in literature [275–279]. Devices with an area of zero indicate the filter was designed for single point detection.
Conclusions and Future Work

This work accomplished the three main objectives of:

1. characterisation and testing of materials for the proposed devices:
   
   This thesis has presented a full array of mechanical and material properties for both ICPCVD a-Si:H and BTIBD a-Si$_x$Ge$_{1-x}$ thin films for use in MEMS and MOEMS.

2. designing and modeling of MEMS actuation structures capable of driving the specified (and unprecedented) large-area filter and operating at very low temperatures:
   
   This thesis reported on various geometries of support structures through FEM analysis with the most promising structures selected for prototype development.

3. characterisation and optimisation of prototype systems fabricated based on the designs for proof of concept:
   
   The leverage bending actuation method and a new support structure were combined in a prototype device demonstrated to have large range tuning for the large area (millimeter scale) microspectrometer.
9.1 Development and Verification of Micromechanical Testing Techniques

Several techniques have been used to determine the Young’s modulus \( (E) \), Shear modulus \( (G) \), Hardness \( (H) \), Poisson’s ratio \( (\nu) \), density \( (\rho) \), coefficient of thermal expansion (CTE, \( \alpha \)), residual stress \( (\sigma_0) \), and tensile strength \( (\sigma_T) \) of thin film a-Si:H and a-Si\(_x\)Ge\(_{1-x}\) materials. These techniques include, thin film induced substrate bending, nanoindentation, microcantilever resonance, quartz crystal microbalance, and double necked microbridge fracturing. Specifically, the individual material parameters were measured as follows:

\( E \) : Young’s modulus was determined through the traditional nanoindentation as well as through microcantilever vibration, and substrate bending. All methods showed good agreement with each other. ICPCVD a-Si:H had a significantly reduced Young’s modulus compared to BTIBD Si, and BTIBD a-Si\(_x\)Ge\(_{1-x}\) exhibited the expected compositional variation of increasing Young’s modulus with increasing Si content.

\( G \) : Shear modulus was determined through microcantilever resonance showing ICPCVD a-Si:H to have much lower shear modulus than BTIBD Si, indicating a process dependence of shear modulus. Shear modulus of BTIBD a-Si\(_x\)Ge\(_{1-x}\) was determined to be compositionally dependent, increasing with increasing Si content.

\( H \) : Hardness was determined through traditional nanoindentation techniques showing ICPCVD a-Si:H to have a similar hardness to BTIBD Si and hardness of a-Si\(_x\)Ge\(_{1-x}\) to be compositionally dependent.

\( \nu \) : Poisson’s ratio was determined through microcantilever resonance resulting in a similar value of 0.2 for both ICPCVD a-Si:H and BTIBD a-Si\(_x\)Ge\(_{1-x}\) (independent of composition).

\( \rho \) : Density of the films was determined through the quartz crystal microbalance technique showing increased density of films in BTIBD deposition compared to ICPCVD depositions.
α: Temperature dependent coefficient of thermal expansion was determined for ICPCVD a-Si:H and BTIBD a-Si$_x$Ge$_{1-x}$ through low temperature measurements of thin film induced substrate bending. Tests showed that processing conditions contribute greatly to the observed CTE of the thin films with latent processing gas species potentially playing a significant role in the thermal expansion of the film.

σ$_o$: Residual stress in the as-deposited films and heat treated films was determined through thin film induced substrate bending. For BTIBD a-Si$_x$Ge$_{1-x}$ the as-deposited film stress is compressive, becoming more compressive with increasing Si content. The single-event as-deposited stress of a-Si:H had a normal distribution centered on 27 MPa compressive stress.

σ$_T$: Tensile strength was determined for the thin films through fracturing of a double necked suspended microbridge. The microbridge was designed to exploit the stress concentration effect at the necked areas, forcing failure of the microbridge to a region in true tension. This test showed a potential improvement in strength of BTIBD films over ICPCVD films.

The values for mechanical properties determined for ICPCVD a-Si:H and BTIBD a-Si$_x$Ge$_{1-x}$ are given in Table 9.1
### Table 9.1: Mechanical Properties of ICPCVD a-Si:H and BTIBD a-Si$_x$Ge$_{1-x}$

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Technique</th>
<th>Section</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E$</td>
<td>$125 \pm 5 \text{ (GPa)}$</td>
<td>Nanoindentation</td>
<td>6.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>substrate bending</td>
<td>6.3.2</td>
</tr>
<tr>
<td>$G$</td>
<td>$49.7 \pm 2.3 \text{ (GPa)}$</td>
<td>Cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td>$H$</td>
<td>$9.9 \pm 0.9 \text{ (GPa)}$</td>
<td>Nanoindentation</td>
<td>6.2</td>
</tr>
<tr>
<td>$\nu$</td>
<td>$0.20 \pm 0.04$</td>
<td>Cantilever vibration</td>
<td>6.5</td>
</tr>
<tr>
<td>$\sigma_T$($\text{strength}$)</td>
<td>$1.3 \pm 0.1 \text{ (GPa)}$</td>
<td>Microbridge fracturing</td>
<td>6.6</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>$-3.231 \times 10^{-6}$</td>
<td>Substrate bending</td>
<td>6.3.2</td>
</tr>
<tr>
<td></td>
<td>$+(7.20 \times 10^{-8})T$</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$-(6.29 \times 10^{-10})T^2$</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$+(3.66 \times 10^{-12})T^3$</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$-(1.12 \times 10^{-14})T^4$</td>
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<tr>
<td></td>
<td>$+(1.31 \times 10^{-17})T^5$</td>
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<tr>
<td>$\rho$</td>
<td>$2.08 \pm 0.25 \text{ (g/cm}^3\text{)}$</td>
<td>QCM</td>
<td>6.5</td>
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<table>
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<tr>
<th>Property</th>
<th>Value</th>
<th>Technique</th>
<th>Section</th>
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</thead>
<tbody>
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<td>$E$</td>
<td>$110$ to $160 \text{ (GPa)}$</td>
<td>Nanoindentation</td>
<td>7.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>cantilever vibration</td>
<td>7.5</td>
</tr>
<tr>
<td>$G$</td>
<td>$45$ to $65 \text{ (GPa)}$</td>
<td>Cantilever vibration</td>
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<tr>
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<tr>
<td>$\nu$</td>
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<td>Cantilever vibration</td>
<td>7.5</td>
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<tr>
<td>$\sigma_T$($\text{strength}$)</td>
<td>$1.6$ to $1.1 \text{ (GPa)}$</td>
<td>Microbridge fracturing</td>
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<tr>
<td>$\alpha(\text{T=300K})$</td>
<td>$7.1 \times 10^{-6}$ to $6.4 \times 10^{-6} \text{ K}^{-1}$</td>
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<td>$\rho$</td>
<td>$4.68$ to $2.63 \text{ (g/cm}^3\text{)}$</td>
<td>QCM</td>
<td>7.2</td>
</tr>
</tbody>
</table>

## 9.2 Suspension and actuation of large area structures

This study developed a new actuation structure aimed at maximising the tuning range of a large area filter. Figure 9.1 summarises the key features of the new filter design developed in this thesis.

## 9.3 Future Work

This work aimed at determining mechanical properties of thin film material to enable finite element modeling and subsequent fabrication of a large area filter.
A successful design featuring an a-Si:H based filter was achieved demonstrating large tuning range and flat millimeter-scale membranes. Initial material properties of a-Si$_x$Ge$_{1-x}$ were determined highlighting important areas for filter design considerations with this material. Moving forward with the successful implementation of a-Si$_x$Ge$_{1-x}$ thin films in to a stable flat filter would require:
1. Development of post processing or in situ heat treatments to create tensile BTIBD films. The compressivity of the BTIBD a-Si$_x$Ge$_{1-x}$ thin films is due to the highly energetic deposition process as well as the Ar contained within the films. Current processing and post processing heat treatments to eliminate Ar result in, at best, neutral to slightly compressive films. Further heating times result in the films being destroyed. Tensile films are required to form flat suspended membranes and this poses a great challenge with BTIBD depositions.

2. Mechanical properties change with heat treatment. As such, once a suitable process is developed for creating tensile BTIBD a-Si$_x$Ge$_{1-x}$ films, material properties of the treat films will need to be determined.

3. The effect of crystallisation on mechanical properties of a-Si$_x$Ge$_{1-x}$ was not explored as the films presented were all amorphous. However, with extensive heat treatments crystallisation of the films is sure to occur resulting in a change in the mechanical properties.

4. Processing gas species remaining in the films after deposition and post process annealing may have a significant impact on CTE. The 4wave BTIBD system has capabilities to utilise alternative sputtering gasses to evaluate the effect the latent gas species have on the CTE of the thin films. This is critically important in application where the films must be cooled to cryogenic temperatures and only low tensile stress is achievable in the room temperature devices.
Appendix A

This appendix gives a full report of the FEM pull-in data, for which a summary was presented in Chapter 8. The support actuation structures modeled are:

- $\alpha_1$ round featured post with no top-hat,
- $\alpha_2$ round featured post with $1200 \, \mu m^2$ top-hat
- $\alpha_3$ round featured post with $3200 \, \mu m^2$ top-hat
- $\beta_1$ square featured post with no top-hat,
- $\beta_2$ square featured post with $1200 \, \mu m^2$ top-hat
- $\beta_3$ square featured post with $3200 \, \mu m^2$ top-hat
- $\delta$ open style rail
- $\gamma$ solid style rail

Figure A.1 shows pull-in versus temperature. In this study, temperature of the FEM model was varied between 77 K and 300 K for the various support configurations. The actuation displacement curves were modeled at each temperature.

Figure A.2 shows pull-in versus changing pre-release film stress. For this study residual film stress was varied from 0 MPa to 300 MPa. Voltage displacement behavior at each residual stress level was simulated for the different actuation structures.

Figure A.3 shows pull-in versus a localized mirror stress. For this simulation a load was applied normal to the tether face to simulate the localized load due to an attached membrane. The load was varied from 0 MPa to 15 MPa and Voltage displacement behavior was simulated for the various actuation structures.
Figure A.1: Pull-in versus temperature for $\alpha$ fixed-fixed beams with round anchors, $\beta$ fixed-fixed beams with square anchors, $\delta$ open rail, and $\gamma$ closed rail.
Figure A.2: Pull-in versus changing pre-release residual stress for $\alpha$ fixed-fixed beams with round anchors, $\beta$ fixed-fixed beams with square anchors, $\delta$ open rail, and $\gamma$ closed rail.
Figure A.3: Pull-in versus an orthogonal pulling strain to simulate a tensile suspended mirror for \( \alpha \) fixed-fixed beams with round anchors, \( \beta \) fixed-fixed beams with square anchors, \( \delta \) open rail, and \( \gamma \) closed rail.
Appendix B

B.1 Strain

The application of stress to a sample results in a dimensional change in the material. This change is given as the material strain:

\[ \varepsilon = \frac{\Delta l}{l_o} \]  

(9.3.1)

where \( \Delta l \) is the change in length of the sample and \( l_o \) is the original length.

For a sample of irregular profile, like that in Figure B.1 (a), an effective strain can be calculated as the sum of the strains from each segment:

\[ \varepsilon_o = \varepsilon_1 \frac{l_1}{L_T} + \varepsilon_2 \frac{l_2}{L_T} + \ldots \varepsilon_n \frac{l_n}{L_T} \]  

(9.3.2)

where \( \varepsilon_o \) is given as the overall change in length of the non-uniform structure as per Equation 9.3.1, \( l_n \) is the length of a given portion of the beam, \( \varepsilon_n \) is the corresponding strain for the given section, and \( L_T \) is the total length of the structure.

For a constant load, \( F \), on the structure, the strain in one region will be proportional to the strain in another region by the cross-sectional area. That is to say \( \varepsilon_n = \varepsilon_1 \times \left( \frac{A_1}{A_n} \right) \). Given this, \( \varepsilon_o \), can be re-written in terms of the strain in a single region, \( \varepsilon_1 \):

\[ \varepsilon_o = \varepsilon_1 \frac{l_1}{L_T} + \varepsilon_1 \frac{A_1}{A_2} \frac{l_2}{L_T} + \ldots + \varepsilon_1 \frac{A_1}{A_n} \frac{l_n}{L_T} \]  

(9.3.3)

or simply
B.1 Drawing of a test structure with non-uniform cross-section: (a) test structure with discrete segmentation and (b) test structure with non-discrete (continuous) segmentation.
\[ \varepsilon_o = \sum_{n=1}^{n} \varepsilon_1 \frac{A_1}{A_n} \frac{l_n}{L_T} \] (9.3.4)

For a non-uniform object of non-discrete segments like that shown in Figure B.1 (b), as \( l_n \to 0 \), Equation 9.3.4 becomes:

\[ \varepsilon_o = \frac{\varepsilon_1 A_1}{L_T} \int_{0}^{l} \frac{\delta l}{A(l)} \] (9.3.5)

where \( l \) is the position along the length of the structure, \( A_1 \) is the cross-sectional area of the region to be evaluated with corresponding strain \( \varepsilon_1 \), and \( A(l) \) is the position dependent area function for the test structure. To evaluate the strain in a particular section of the test structure we simply rearrange Equation 9.3.5:

\[ \varepsilon_1 = \frac{\varepsilon_o L_T A_1^{-1}}{\int_{0}^{l} 1/A(l) \delta l} \] (9.3.6)

Venant’s Theorem states that stress and strain produced at points in a body sufficiently removed from the region of load application will be the same as the stress and strain produced by any applied loadings that have the same statically equivalent resultant, and are applied to the body within the same region [228, 229]. The converse of this being that any sudden change in geometry (namely width in the case of double-necked microbridges) will result in non uniform stress across the width of the structure. Compromises regarding Venant’s Theorem were made with the microbridge structures to ensure that fracture was achieved in less than 5 \( \mu \)m of vertical displacement (as is the limit of the Hysitron 950 Triboscan).
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