Non-contact and Non-destructive Ultrasonic Method for Material Characterisation

by

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Declarations

I declare that the research presented in this thesis carried out at the Department of Physics, University of Warwick, during the period between February 2015 and February 2019, under the supervision of Dr Rachel Edwards. All work composed by myself and no part of it has been previously submitted in this or any other academic institution for the purposes of obtaining a higher degree. Parts of this thesis have appeared or is being prepared for publication in journal papers and conference proceedings.

Publications


Abstract

This thesis covers interdisciplinary research, using vibrations in the ultrasonic frequency range to analyse the behaviour of suspended structures made from Ge, 3C-SiC, and SiN. These structures can be used in micro-electromechanical Systems (MEMS) for applications such as pressure sensors. A laser interferometer was used to measure the vibration resonance patterns, along with a self-developed system which can do two-dimensional (2D) scanning of the samples at reduced pressures, and over a range of temperatures, in order to probe the behaviour of the materials. This work presents the feasibility of this non-contact and non-destructive method for material characterisation of suspended structures through the information obtained from the deflection and the structures resonant frequencies.

Vibration testing is typically done in the linear regime, and linear vibrations of the structures are initially investigated with the 2D scanning system. Larger amplitude vibrations lead to non-linear behaviour. The non-linear dynamics experimental results show the amplitude-frequency response curve has a position dependence for where the laser probe is on the sample, and is related to the strength of excitation force. A critical displacement of the oscillation amplitude is identified for the samples in these experimental measurements. The experimental results from the non-linear dynamic measurements are used to consider the applicability of several theoretical studies from the literature to this type of sample.

The laser probe was shown to have a small thermal effect on the measurements. The linear and non-linear 2D scan studies used low laser power to minimise heating. A calibration measurement was then done on the thermal effects which were induced
by the laser probe for different detection laser powers. The dynamic thermal field generated by the moving laser during a 2D mapping process causes different thermal gradients across the sample when the laser is placed at different positions on the suspended thin film. The dynamic thermal field could be used as a novel tool for material characterisation following further investigation.

Finally, the onset of damage followed by self-healing behaviour was observed on a Ge thin film with sub-micron and nano meter cracks. The damage was analysed by comparing the percentage change of the residual stress between theoretical predictions from considering the thermal expansion of the thin film and substrate materials, with experimental measurements done under a varying temperature. Additionally, the quality-factors of the resonance were also monitored during the measurement and showed the onset of damage. This work when taken as a whole offers a new novel analysis technique for suspended structures used in MEMS applications, with the potential to characterise the material properties, and to inspect them for quality assessment or damage detection. The method could be made to be fully non-contact through using activation using e.g. air-coupled transducers, leading to a potential on-line analysis technique.
Chapter 1

Introduction

Over the last century, technology has developed rapidly, even faster than at any other time in the last millennium, especially since semiconductor materials and silicon-based devices have been introduced into our daily life. The well-known statement known as Moore’s law describes that the number of transistors on an integrated circuit will double every two years, until it reaches the critical physical scale. However, the real development speed of the silicon (Si) industry is faster than Moores’s prediction, thanks to complementary metal oxide semiconductor (CMOS) scaling. Thus, mechanical devices are being made smaller and smaller as more transistors can be packed in a smaller area.

Since 1960, the complexity and performance of microelectronic devices has increased by many orders of magnitude, whereas size, power consumption, and price have been reduced significantly. The first known electromechanical micro-component of a transistor with a vibrating beam resonator was proposed by Bell Laboratories in 1967. Monolithic structures in three dimensions on a silicon substrate were processed by anisotropic chemical wet etching in 1978 by K.E. Beam. The wet etching process was later applied to a localised etching on a thin layer of soft silica (as a sacrificial layer) to produce suspended structures for use as mechanical mobile parts of micro-electromechanical systems (MEMS), by several laboratories during the 1980s. This wet etching concept was generalized for silicon as the best material for micromechanics devices in 1982 by Professor K.E. Petersen. This process paved the way to numerous electromechanical applications of microsystems in the field of sensors and micro-actuators; the traditional watch industry was certainly improved by the miniaturization of mechanical and electromechanical components in view of their precision of manufacturing and assembly. An all-silicon micro-engine with a diameter of 150 µm was manufactured in 1989 at the University of
The microfabrication techniques developed have provided an opportunity for researchers to develop versatile systems on the micro-scale such as MEMS. Such microsystems are small-dimensional, and combine electrical and mechanical components, with a total size ranging from centimeters down to micrometers. To fabricate a MEMS one has to use several different technologies of manufacturing in parallel, as with integrated circuit batch-processing. The main evolution of MEMS functions, beyond electronics, came from the introduction of micromechanics using mobile parts (eg. suspended structures), optics, and intelligence (software embedded into the microsystems) by using the technologies of microelectronics. Various MEMS structures, such as linear comb capacitors, micro-mirrors, microchannels, cantilevers, and diaphragms have been successfully fabricated, and are widely used in accelerometers, pressure sensors, optical switches, and lab-on-chip platforms \[7, 8, 9, 10, 11, 12, 13\].

In the last few decades, MEMS have been extended to the nanoscale through the development of materials and processing techniques. This has enabled a new class of devices, known as nano-electro-mechanical systems (NEMS). These can have the ability to detect extremely small quantities, on the order of femtometers, zeptograms, attonewtons and attojoules \[14, 15, 16\].

The MEMS and NEMS sensors are often constructed from simple mechanical structures such as cantilevers and beams, partly because of their very high mechanical responsivities, ease for modelling, fabrication simplicity, and availability of materials. Nanotechnologies also enable creating local functions, using sculpture by manipulating atoms, molecules, or molecular assembly. They are achievable as nano-micro-meso-macro systems, ensuring a coherent and additional set of useful functions from the nano scale to the real world. Key benefits of MEMS and NEMS devices include their miniature size, lightweight nature, high resonant frequencies, the short thermal time constant, and the capability to integrate with nano or microelectronics\[14\].

As the field of MEMS grows considerably, the advantages of smaller, less costly devices and systems have given impetus to develop MEMS devices for more widespread applications, such as micro-spacecraft, miniature communication satellites, and meteorological instrumentation based on utilization of MEMS technology. However, one of the key challenges facing the design of these devices is reliability in the working environment such as robustness to vibration. Thus, understanding the relationship between microstructure and mechanical properties has always been one of the primary goals during development. The mechanical properties of structural materials have always been of paramount importance to material scientists. A sim-
and accurate non-destructive method for mechanical property characterisation for such devices is still lacking [17].

1.1 Materials

In 1833, Michael Faraday first studied semiconductor behaviour by measuring the temperature dependence of the electrical conductivity of silver sulphide [18]. Another interesting property, photoconductivity behaviour, was discovered in selenium by Willoughby Smith in 1873. The first patent of a semiconductor based rectifier was filed by Sir J C Bose in 1901, who invented a semiconductor device capable of receiving wireless signals for radio waves [19].

The semiconductor industry was based on the invention of the transistor by John Bardeen, Walter Brattain and William Shockley at Bell labs in 1947, after much researchers’ work had led to greater understanding of semiconductors. The first junction transistor was fabricated from germanium (Ge) because its mechanical and physical properties made it easier to work with, and more efficient than Si in some applications [20]. Since 1952, high quality Si crystals have begun to take over as the predominant material in the semiconductor industry. As Si still dominates the market in complementary metal oxide semiconductor (CMOS) technology, compatibility with Si technology, such as being able to grow particular semiconductors on a Si substrate wafer, is essential nowadays for the mass implementation of other materials such as Ge and silicon carbide (SiC) [21, 22, 23].

Ge, SiC and silicon nitride (Si₃N₄) have been the focus of much research in the last few decades, because of their potential for use as MEMS. The tensile-strained Ge membrane can be applied to infrared photonic applications, in near-infrared lasers, or used as the mirror for Fabry-Perot filters [24, 25, 26]. SiC has hardness close to diamond and is much more suitable for use as sensors in hostile environments, such as where there is high voltage, high power, or high temperature, and high frequency applications. A variety of practical applications, such as biomedical membranes, substrates for GaN epitaxial growth, electro-osmotic pumps, and nanopattern templates have been suggested [27]. Si₃N₄ has excellent resistance to thermal shock and is stable up to a temperature of 1500 °C with a large energy gap [28, 29].

1.1.1 Germanium

In recent years, Ge has regained attention because there are some advantages over Si through both its electrical and mechanical properties. Ge can be used for a wide range of applications as CMOS based devices. Ge has much higher mobility for
electrons and holes than Si, making it ideal for MOSFETS due to the lower transport mass for both electrons and holes. Ge is known as a pseudo-direct bandgap material. This means that it only has a small difference between its direct and indirect gap (0.136 eV). The band-gap of Ge makes it highly suitable for photonics purposes, hence the recent demonstration of a germanium laser [30, 31].

Due to its low natural abundance, the use of bulk Ge wafers is not economically viable for mass production. Ge also has a higher atomic mass than Si, and when this is combined with an increased substrate thickness, Ge substrates could become prohibitively heavy, especially for the larger 500 mm and 450 mm wafers now standard for Si. Meanwhile, because the semiconductor industry has invested heavily in the fabrication of silicon based devices, for any new technology to be economically viable, it should be compatible with the existing silicon fabrication process. The adoption of Ge by industry requires Ge to be easily integrated with the industry-standard (001) silicon platform. If high quality layers of Ge can be grown on silicon (001) wafers this integration can be achieved [18].

Ge is typically incorporated onto Si substrates using epitaxy. Unfortunately, Ge on Si (001) epitaxy normally gives rise to high surface roughness. This roughness is due to islanding from Stranski-Krastanow (S-K) growth (described later). The 4.2% lattice mismatch between Si and Ge causes S-K growth after an initial period of 2D growth. Also, the lattice mismatch can create high densities of both misfit-dislocations and threading-dislocations in the interface section. Many researchers are devoted to inventing new fabrication processes which can be used to improve and achieve high quality layers of germanium growth on silicon substrates [32, 33].

1.1.2 Silicon Carbide

Another very interesting semiconductor material is silicon carbide (SiC). SiC is one of several compound semiconductors that can be formed from group IV elements (C, Si, Ge, Sn) as it possesses a defined crystal bonding structure consisting entirely of Si-C bonds. SiC is effectively a cross between pure Si and diamond and possesses many properties similar to the latter. It is referred to as a wide bandgap semiconductor, a term typically given to semiconductors with a bandgap significantly greater than that of Si (1.1 eV). All SiC polytypes (3C-SiC, 4H-SiC, and 6H-SiC) have indirect bandgaps, however, the magnitude of this bandgap varies between polytypes. SiC is also well known for being an extremely hard substance with Mohs hardness of 9.2 to 9.3, which is close to that of diamond (Mohs hardness of 10). It is also inert in almost all chemicals at room temperature. Thus, its wide bandgap, extreme hardness, high Youngs modulus, high chemical inertness, and strong radiation toler-
ance make SiC extremely well suited for the extreme conditions found in combustion systems and space applications, when compared to Si, GaAs and other semiconductors. These beneficial properties, coupled with its high sublimation temperature, facilitate continuous and reliable operation of SiC MEMS devices at temperatures above 500 °C, and make it ideal for a range of applications operating under harsh environmental conditions, such as high power and high temperature devices [34].

The first synthesis of Si-C bonds was done by Jons Berzelius in 1824. Edward Acheson was able to synthesise SiC by mixing silica and coke and used a smelting furnace to produce abrasive materials to replace diamond in 1892. Acheson discovered a series of hexagonal crystals in his SiC samples which led to the discovery of polytypism, that SiC can exist in a number of different crystalline forms, as shown in figure 1.1.

![SiC Polytypes](image)

Figure 1.1: SiC polytypes; unit cells of 3C, 2H, 4H and 6H, from left to right. Picture taken from [21].

Of all the SiC polytypes there is only one with a cubic structure. Cubic silicon carbide, commonly referred to as 3C-SiC or β-SiC, has a stacking sequence of AB-CABC... which results in a purely zinc-blend structure. 3C-SiC has various advantages over other polytypes, such as having the lowest bandgap and highest potential electron mobility and saturation drift velocity, a result of reduced phonon scattering from its higher crystal symmetry. A key advantage of 3C-SiC is that its crystalline structure is more stable at lower temperatures. A further advantage is that it has a purely isotropic crystal structure. Although 3C-SiC has the smallest bandgap of all the SiC polytypes, this can be seen as an advantage as it reduces the electric field required for channel inversion within metal oxide semiconductor field effect transis-
tors (MOSFETs), and reduces the density of traps at SiO$_2$/3C-SiC interfaces, which can help increase the drift mobility inside the channel [21].

The first 3C-SiC growth on a Si substrate was done by W. G. Spitzer et al. in 1958 [35], but it was not highly regarded and little research on SiC followed. In recent years, the main process for growing SiC crystals has still mainly used the seeded sublimation process which was invented by Tairov and Tsvetov in 1978 [36]. Later on, the step-controlled epitaxy process was invented and used to grow high quality SiC films on off-axis SiC substrates. While SiC devices have still not penetrated the mass market, wafers are sold as the most important substrate for GaN, which is used for light-emitting diodes (LEDs), radio frequency (RF) and power applications [37, 38, 39, 40, 41, 42].

Although SiC substrates have been in production for many years they are still limited to smaller wafer sizes, and are significantly more expensive than equivalent Si wafers. One of the most attractive solutions to the issue of wafer size and price is to grow 3C-SiC on Si substrates, which combines the properties of SiC with the cost point and scale of Si. However, one of the most detrimental issues to this concept is the thermal behaviour mismatch between SiC and Si, which leads to high levels of thermal stress in the heterostructure. This leads to an effect known as wafer bow, which can distort 3C-SiC/Si wafers, making them unsuitable for device fabrication. Also, the downside to performing heteroepitaxy is that the lattice constant of 3C-SiC is 19.7% mismatched from the lattice constant of Si, which leads to a high number of defects at the interface between the two materials that propagates throughout the 3C-SiC epilayer [43]. These defects can cause problems in devices or on structures.

1.2 Thin Film Fabrication

The most common methods to grow films being deposited onto substrates are physical vapour deposition (PVD) techniques such as molecular beam epitaxy (MBE), and chemical vapour deposition (CVD). The deposition is normally carried out in a vacuum chamber to enable control of the vapour composition. PVD and CVD are classified as methods where a vapour is created by physical means with or without a chemical reaction. The choice of technique depends on the requirements, such as material purity, structure quality, the rate of growth, temperature constraints and other factors, and many variations of the basic methods of vapour deposition have been developed. The final result of film growth is to condense the vapour onto the substrate surface to produce the film [44, 45, 46].

MBE growth occurs through the deposition of material on a crystalline substrate
using a thermal beam of atoms or molecules emitted from one or more targets under ultra-high vacuum \[47\]. MBE is an excellent research tool as it allows fine control over growth parameters and can be used to grow complicated structures and novel compounds, but does not offer the throughput necessary for mass volume manufacturing. Substrate temperatures are often limited to \(< 1100 \, ^\circ C\) for Si and even lower for certain III-V semiconductors. A schematic diagram of MBE is shown in figure 1.2.

![Schematic diagram of MBE. Picture taken from [44].](image)

The condensation process of film formation involves complex processes which require knowledge of thermodynamics. The start of the growth of a thin film on a substrate is described in atomistic terms. To form a single crystal film, atoms of the film material have to adhere to the substrate and settle into equilibrium positions before structural defects are left behind the growth front. For producing an amorphous film, atoms must be prevented from seeking a stable equilibrium position once they arrive at the growth surface. The atoms deposited onto the surface of a substrate are referred to as adatoms.

The migration of atoms depends on a range of parameters of the growth system including the temperature, chemical species, and the strain in the system. If the migration length is sufficiently long, the free energy of the system is reduced most effectively by the adatoms bonding into lattice sites at the edges of steps on the substrate surface. This leads to 2-dimensional (2D) film growth with surface roughness and is referred to as Frank van der Mewre growth, see figure 1.3 (a). However, if the adatoms lack the energy to reach a stepped terrace, or the surface interactions are too great, then the adatoms will instead bond to the surface of a terrace which will lead to subsequent adatoms nucleating on the same site. This results in
3-dimensional island growth which increases the surface roughness of the film and is referred to as Volmer-Weber growth, see figure 1.3(b). This type of growth is more likely to occur in highly mismatched systems such as growing Ge or 3C-SiC on Si, as the surface energy is greater due to the presence of lattice mismatch and strain. It is possible for the growth of a film to begin under the Frank van der Mewre regime, however, as surface effects can become more severe as the film thickness increases, and then the system can transition to Volmer-Weber growth. This is referred to as Stranski-Krastinov growth, see figure 1.3(c) [48].

The process of depositing atoms, and alignment of crystallographic atom positions forming a crystalline layer on top of another crystalline layer or substrate, is referred to as epitaxial growth. The film material does not necessarily have to be the same as the substrate material. When the thin film material is the same as the substrate material, crystallographic registry between the thin film and the substrate is commonly referred to as homoepitaxy. If the two materials are different, the epitaxial deposition is known as heteroepitaxy. An epilayer which continues the crystalline structure and orientation of the substrate can range in thickness from single atomic layers to tens or even hundreds of microns. Epitaxy offers high quality monocrystalline thin films, as well as fine control over layer thickness and doping profiles of either single or multiple stacked epilayers, which provides major advantages in material fabrication for microelectronic and optoelectronic applications.

Heteroepitaxial film growth is extremely useful for the growth of materials that either do not have native substrates, such as alloys, or where substrates are either limited in quality, wafer size, or are extremely expensive, such as Ge or GaN substrates. It is also crucial for the heterogeneous integration of other semiconductor materials into the silicon industry to be able to grow epilayers on Si substrates. The substrate crystal structure offers a template for the positioning of film material atoms. However, there is almost always some mismatch between the crystal structure of the substrate and the thin film because of the difference in the lattice parameter of these materials. When the mismatch in lattice is below 0.5%, the
growth tends to be planar. If the mismatch is large, the material tends to gather into islands on the surface but remains epitaxial. All these mismatches lead to either compressive or tensile strain, with (mismatch strain = (lattice size of substrate - lattice size of film) / lattice size of film), and ultimately crystal defects in the epilayer, once the thickness exceeds some critical value [44].

1.3 Defects

Any lattice mismatch between the film and the substrate must be accommodated by a uniform strain in the film (together with slight bending of the substrate), and this typically leads to very large biaxial stresses in the film [44]. Growing films onto a dissimilar substrate with a large difference of lattice size can lead to a huge number of issues, such as an increased number of crystal defects and high levels of stress in the heterostructures, which can lead to cracks, as shown in figure 1.4. However, materials scientists are still interested in trying to form dislocation-free epitaxial thin films on single crystal substrates, or reducing the density and manipulating the type of defects during the epitaxial thin film growth, by controlling the growing parameters.

![Figure 1.4: Cracks in a GaN film grown on sapphire. Picture taken from [49].](image)

There are several different types of defects which can be generated in heteroepitaxial film growth, including misfit dislocations, stacking faults, microtwins, inversion domain boundaries, voids, vacancies and polytype inclusions. Each of these defects can exhibit different features and effects in the films, even after they are fabricated into microelectronic and optoelectronic devices.

The misfit dislocation is well known as being due to a large mismatch between the lattice constants of the film material and substrate material. The dominant
defects at the interface section of the two materials are from these misfit dislocations. One example is the 3C-SiC on Si substrate with an about 19.7% mismatch, which leads to a large strain field between the two crystals at the interface. Thus, partial dislocations form at a lattice plane in order to fully relax the epilayer. These defects combine to form a pure edge misfit dislocation at the interface between the 3C-SiC and the Si substrate.

The strain relaxation of the lattice mismatch at the interface can also introduce additional monolayers in the 3C-SiC crystal. This type of defect is referred to as a stacking fault as they disrupt the stacking sequence of the lattice. The stacking sequence for a purely 3C-SiC crystal is ABCABC....but the insertion of additional monolayer can alter this sequence to ABCBABCAB, as shown in figure 1.5. The reason for formation of this type of defect is not well understood. The assumption is that the stacking faults result through two 60 (degree) partial dislocations at the interface which do not join to form a perfect misfit dislocation and instead each form a pair of 30° and 90° partial dislocations [50]. Stacking faults are considered device killers for power electronic device applications, in that they induce significant leakage current in vertical rectifying devices. A transmission electron microscopy (TEM) picture (in part (a)) and a schematic diagram (in part (b)) of stacking faults is shown in figure 1.5.

![Figure 1.5](image.png)

Figure 1.5: Cross sectional TEM image (a) and schematic diagram (b) through the 3C-SiC crystal structure showing the formation of a stacking fault (SF). Picture taken from [21].

A similar defect to stacking faults is microtwins. These are also planar defects that
result from disruptions in the crystal structure, however, while stacking faults involve only one atomic bilayer, microtwins involve several atomic planes by changing the stacking sequence of 3C-SiC to e.g. ABCBACBCA..., as shown in figure 1.6. However, microtwins can also annihilate when they intercept each other in the same way that stacking faults do, and as such the density of both planar defects decreases with increasing epilayer thickness [51]. A TEM picture (part (a)) and a schematic diagram of microtwins is shown in figure 1.6.

Another common defect in compound semiconductors with heteroepitaxial growth is inversion domain boundaries (IDBs), also commonly referred to as anti-phase domains (APDs) [52, 53]. An IDB formed in 3C-SiC is the exchange of C and Si lattice site positions at an interface between two domains of the crystal. The origin of the domains is due to the initial island nucleation of 3C-SiC on the Si lattice. On-axis substrates typically have mono-atomic steps which lead to the formation of these non-equivalent domains. When the islands spread to form an epilayer, the domains meet and form IDBs. This type of defect has been shown to be a more significant killer of device performance than stacking faults, causing power devices such as Schottky diodes to short, losing all forward and reverse bias characteristics. However, by using an off-axis Si wafer for growth the heterostructure can simply avoid the IDB defects at diatomic steps. These diatomic steps can be promoted in Si substrates through hydrogen based thermal annealing prior to epitaxial growth, which was studied by number of researchers.

With high temperature epitaxy growth, void defects can be formed by the out-
diffusion of Si atoms from the substrate during growth, and are typically shaped like inverted pyramids. The formation of voids can be reduced by growing at a lower temperature, between 900-1300 °C. The easiest way to prevent void formation is by proper optimisation of a buffer layer between the film and substrate. Voids at the interface are a significant problem for vertical device applications such as heterojunction bipolar transistors, where a high quality interface is essential [54, 55]. A cross section TEM picture with defect of void is shown in figure 1.7. There is also an uncommon type of defect, called polytype inclusion, where different hexagonal polytypes can be grown in the same bilayers [27]. This is mainly an issue with the homoepitaxy of 4H-SiC or 6H-SiC, which can be plagued with 3C-SiC polytype inclusions, affecting the electrical performance and lifetime of devices by trapping electrons.

1.4 Techniques for Characterisation

Semiconductor manufacturers and materials scientists require knowledge of epitaxy layer characteristics in order to choose an appropriate characterisation method for
application in different processes, and further require device characterisation. Understanding these characteristics will help to determine optimal film casting conditions, control growth quality, and develop thin film transport. Epitaxy layer characteristics of interest include surface morphology, mechanical properties and various chemical and physical properties. Characterisation methods can be classified into two categories: destructive and non-destructive. An ideal method should be non-destructive, accurate, repeatable, and fast, and should maximize the data obtained. However, not all information can be obtained from non-destructive method. Thus, in the following sections destructive and non-destructive characterisation methods will be introduced.

Knowledge of mechanical properties is key for device fabrication. It is well known that mechanical properties of thin films are different from those of bulk materials \[56\]. This difference can be explained by the large surface-to-volume ratio, since the microstructure of the surface will have a significant influence on the mechanical properties of thin films. Furthermore, defects and unique microstructures from the fabrication process for thin films also contributes to the variation of mechanical properties. Thus, mechanical properties of thin films cannot be simply deduced from those of their bulk counterparts. Therefore, it is necessary to experimentally investigate the length scale effects and microstructure effects on mechanical behaviour in thin films, and several specialized testing techniques have been developed \[57\] \[58\] \[59\] \[60\] \[61\].

Researchers have tried to determine mechanical properties of thin film materials experimentally. The challenges include the ability to measure the geometrical features of test structures and relative displacements (strain) accurately, to apply a small force (\(\mu\)N or mN range) to test structures without damaging them, and to make a good alignment between applied forces and test structures.

1.4.1 Direct Tension Test

The concept of the direct tension test is rather simple: a test structure is loaded in axial tension until it breaks and tension is recorded. The direct tension test is an effective method to measure the mechanical properties and the measured data can be easily interpreted. However, the requirements for strain measurement and sample alignment are stringent. Atomic-force microscopes (AFM), image-correlation methods, and optical interferometry have been used to measure the small displacements. The loading system is usually incorporated in the specimen preparation step to solve any alignment problem \[62\] \[63\] \[64\].
1.4.2 Wafer Curvature Method

The wafer curvature method was developed for residual stress measurement during a temperature change, when film and substrate have different thermal expansion coefficients. The radius of curvature of the substrate is used to calculate the stress in the film. If the temperature change is cyclically repeated, the thermal fatigue behaviour of a thin film can be studied [65, 66].

The Stoney equation for determining thin film stress given wafer curvature before and after film deposition, as well as the curvature for a simple biaxial bending analysis, is [44]

\[
\frac{1}{R} = \frac{1}{M_s} \frac{6\sigma_f h_f}{h_s^2},
\]

(1.1)

where \( M_s = \frac{E}{1-\nu} \) is the biaxial elastic modulus of the substrate, \( E \) is the Young’s modulus of the substrate, \( \nu \) is the Poisson’s ratio of the substrate, \( \sigma_f \) is the residual stress of the deposited film, and \( h_f \) and \( h_s \) are the thickness of thin film and substrate, respectively. \( R \) is the radius of wafer curvature after thin film deposition (assuming the substrate was flat initially). It is assumed that the elastic bending of the substrate does not depend on the elastic properties or any other mechanical properties of the film.

1.4.3 Bulge Test

Bulge test is a method used to determine elastic, plastic, and time-dependent deformation behaviours when a sample is formed as a freestanding thin film. The freestanding film is pressurized with a uniform pressure from one side and the resultant bulge height is measured as the film deflects outward, as shown in figure 1.8.

The relation of stress and strain in the film can be determined from measurements of pressure (P) and the window’s deflection (d). By measuring the lateral deflection and by incorporating subsequent finite element (FE) analysis, one can extract the Young’s modulus, Poisson’s ratio, and the residual stress from the test data [67, 68].

![Figure 1.8: Schematics of bulge test.](image-url)
The load-deflection technique was initially developed for measuring stress in low modulus tensile materials such as metal and polyimides. Nowadays, this technique is widely used for films that are compatible with silicon micromachining and its derivatives such as silicon carbide. The relation between the pressure (P) and deflection (d) as the film is elastically deformed can be expressed as

$$ P = C_1 \frac{\sigma_f h}{a} d + C_2 \left( \frac{E}{1 - \nu} \frac{h}{a^3} d^3 \right), $$

where $C_1$ and $C_2$ are geometry dependent shape factors, and $a$ is the characteristic length of the membrane. For example, for a circular membrane, $a$ is the radius and $C_1$ and $C_2$ are 4 and $8(1.015 - 0.247\nu)/3$, respectively. For non-circular thin films, such as square or rectangular shape diaphragms, the result is more complicated. For a square film with residual stress $C_1$ and $C_2$ are about 3.393 and $(1.996 - 0.613\nu)$, respectively. For a very long rectangular window $C_1$ and $C_2$ are about 2 and $4/3$, respectively [69].

An accurate determination of Youngs modulus from the bulge test may not be possible because the onset of discernable non-linear elastic behaviour may occur under practical loading conditions. Under these conditions, the load deflection data may overstate the Youngs modulus. For submicron-thick diaphragms with a large area, bulge testing has proven to be challenging because of the difficulty of loading sufficiently to determine Youngs modulus.

### 1.4.4 Indentation Test

Indentation is also a widely used method to determine the mechanical properties for thin films, such as Youngs modulus and micro-hardness, fracture strength and toughness. In a typical test, a diamond tip with well-defined geometry (i.e., spherical, Vickers, Berkvitch, or cone type) is contacted onto the test structure, and the depth of penetration ($d$) is measured as a function of the applied load ($F$). In general, the material hardness can be enhanced by a compressive residual stress, or become softened by a tensile stress. By examining the $F$-$d$ curves obtained from experimental data and fitting with a computer simulation to have a systematic dimensional analysis, it is possible to extract the residual stress of films [56, 57, 60].

The deviations in shape of the material pile-up occurring at the edge of the contact circle can also give information about the level and sign of residual stress (compressive or tensile) within the specimen. Deviations in shape of the load-displacement response from the ideal shape could also be used as an indication of residual stresses, but experiments show that the effect is too small to be measured accurately. Re-
recently, Taljat and Pharr derived a formula based on spherical indentation. The residual stress can be expressed as

$$\sigma_f = Y - \left( \frac{4E^*}{3.3\pi} \right) \frac{a}{R},$$

(1.3)

where $E^*$ is the so-called reduced modulus, $Y$ the yield strength, and $(a/R)$ is the indentation strain.

The indentation method leads to very inhomogeneous stresses and strains, and hence is difficult to use for quantitative determination of fatigue behaviour. A more detailed review of mechanical testing of thin films is given in [57, 60, 61].

1.4.5 M-test

The M-test (see figure 1.9) is a widely used method to determine the Youngs modulus and residual stress of microelectromechanical suspended structures above a fixed ground plane with a gap, such as cantilever beams, fixed end beams, and clamped diaphragms [70]. The stiffness of the material within the suspended structure can be measured by applying a voltage difference (as pull-in voltage) between the upper movable structure and the fixed ground (substrate), causing a downward deflection due to the electrostatic attraction. At a critical pull-in voltage, $V_{PI}$, the upper conductor becomes unstable and spontaneously collapses (or pulls in) to the ground plate. The pull-in voltage is related to the test structures geometry and intrinsic material properties.

![Figure 1.9: Schematic cross-section of the M-test](image)

In comparison with other methods, this method has the advantage of rapidly chang-
ing the loading state, with the potential for testing a batch of specimens simulta-
neously. Also, the measurement can be made entirely using electrical probing, in
a manner similar to that used to check microelectronic circuits. This provides the
opportunity for process monitoring and quality control during device production.
However, the modelling is far more complicated and indirect than the traditional
methods, and only electrically conductive materials are suitable for this technique.
There is also the strict requirement of a flat beam before testing.

1.4.6 Acoustic Test

Standard ultrasonic techniques for the measurement of stress are based on the vari-
ation of the wave speed (i.e., the acoustoelastic effect), which can be conceptually
described by the relation

\[ V = V_0 + K\sigma, \]  \hspace{1cm} (1.4)

where \( V_0 \) is the velocity of a wave in an unstressed medium, \( \sigma \) is the stress and \( K \)
is a material dependent parameter known as the acoustoelastic constant. Detailed
derivation of the acoustoelastic constant and its complete crystallography orienta-
tion dependence can be found elsewhere. In general, waves are launched by a trans-
mittting transducer, propagate through a region of the material, and are detected
by a receiving transducer. The technique is often called pulse-echo (monostatic) if
the same transducer is used for excitation and detection, and pitch-catch (bistatic)
if different transducers are used. In MEMS, this configuration can be realized by
designing piezoelectric surface acoustic wave (SAW) devices that are attached to the
device [71].

1.4.7 Resonance Test

The resonant structure concept was introduced in 1979, and in the first works arrays
of thin, narrow cantilever beams of various lengths were fabricated, with composite
structures extending over an anisotropically etched pit in the substrate. The beams
were excited by variable frequency electrostatic attraction between the substrates
and the beams, and elastic properties of the composite structure were determined
from the measurement of the resonant frequencies. The test structures used in
resonant tests can be very small, which makes this approach suitable for on-chip
testing with appropriate actuation designs. Resonant frequencies are typically in
the ultrasonic range.

For constrained structures, the resonant frequencies shift from the non-stressed val-
ues if residual stresses exist. This is because the natural frequency is proportional
to the square root of the equivalent stiffness of the structure and the stiffness can be changed by the presence of residual stress. The existence of tensile residual stress will stiffen the structure and increase the natural frequency. For a beam simply supported at both ends, the natural frequency can be expressed as

\[ f = \frac{\pi}{2\sqrt{12}} \frac{h}{L^2} \sqrt{\frac{Eh}{\rho}} \sqrt{1 + \frac{12\sigma_b}{\pi^2 Eh}}. \]  

(1.5)

where \( E, \rho, h, L, \) and \( \sigma_b \) are the Youngs modulus, density, thickness, length, and residual stress of the beam, respectively.

An accurate expression obtained by Wylde using Rayleighs quotient for a beam with both ends clamped, describes the natural resonant frequency as

\[ \omega_n^2 = \frac{(2\pi)^4}{3} \frac{EI}{\rho AL^4} + \frac{(2\pi)^2}{3} \frac{\sigma_b}{\rho L^2}. \]  

(1.6)

If the beam is very flexible (i.e., a string), the resonant frequency is simply

\[ f = \frac{1}{2L} \sqrt{\frac{\sigma_b}{\rho}}. \]  

(1.7)

Zhang, et al. [72] reported on the simultaneous measurement of the Youngs modulus and residual stress in single-crystal silicon beams with clamped boundaries at both ends. Kim and Allen used the simple expression above to evaluate the tensile residual stress in microfabricated polyimide structures. It is important to note that air damping and squeezed-film damping can also lead to significant shifts in the resonant frequencies of microfabricated structures, as described later. Therefore, the structure’s frequencies are usually measured under vacuum conditions in order to obtain accurate mechanical properties. Manceau et al. used plate vibration techniques to measure the residual stress of electrolytic nickel coating.

Resonance testing of bulk micromachined diaphragms is potentially a suitable, low risk alternative to static bulge testing, in large part because the amount of diaphragm loading is extremely small. The conventional approach for estimating the frequency of a resonant diaphragm is to treat the structure as a pure membrane with no bending rigidity.

The resonant frequencies of elastic bodies can be modelled using several methods. The most common method involves first calculating the increase in potential and kinetic energies of the body under vibration, and using these values and the corresponding boundary conditions to calculate the modes of vibration. In general, vibrating bodies can be classified into two groups; membranes and plates. The ideal
membrane is assumed to be perfectly flexible, infinitely thin, and of uniform material composition and thickness. Moreover, it is assumed that this membrane is under isotropic tension of sufficient magnitude that small vibrations of the membrane do not affect the overall tension. These assumptions allow for the increase in potential energy to be calculated by multiplying the increased surface area of the membrane by the tension. Equation describes the resonance behaviour of an ideal rectangular membrane:

\[ f_{mn} = \frac{1}{2} \sqrt{\frac{\sigma}{\rho}} \sqrt{\left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2}, \]  

where \( m \) and \( n \) are mode numbers, \( f_{mn} \) is the frequency for a mode described by \( m \) and \( n \), \( a \) and \( b \) are side lengths of the membrane, \( \sigma \) is the stress and \( \rho \) is the mass density.

For an ideal plate it is assumed that the body is perfectly elastic, homogeneous in composition, isotropic in mechanical properties, uniform in thickness and that the thickness is less than 10% of the diaphragm area, but not infinitely thin in comparison to the other dimensions. For an ideal plate, deformation induces a stress in the structure that is proportional to the radius of curvature of the displacement. The increased potential energy that is accumulated in the body under vibration can then be calculated. This, in combination with the boundary conditions, allows for the calculation of the resonant modes of the plate, which is given by

\[ f_{mn} = \frac{1}{2} \sqrt{\frac{Eh^2}{12\rho(1-\nu^2)}} \left[ \left(\frac{m}{a}\right)^2 + \left(\frac{n}{b}\right)^2 \right], \]  

where all terms are as described above.

Measurement and calculation of a realistic resonant frequency of an elastical thin film under isotropic tension is complicated, and more details will be discussed in chapters 2 and 3.

1.4.8 Microscopic Method

There are three main microscopic techniques used to study the morphology of thin film surfaces are atomic force microscopy (AFM), scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

**AFM**

Binnig et al. invented the AFM in 1986 [73]. It is a technique for surface characterisation that is used to map the surface details of materials and measure the surface
roughness on a microscopic scale. The scanning probe of the AFM consists of a sharp tip attached at one end of a cantilever. A typical cantilever can be made from e.g. Si or SiN with the tip radius of curvature on the order of nanometers. When the sharp tip is brought into proximity of a sample surface, the interaction of both attractive (chemical forces, van der Waals and electrostatic) and repulsive (ionic repulsion) forces between the tip and the sample surface, lead to a deflection of the cantilever according to Hooke’s law. Commonly, the deflection of the cantilever is measured by an array of photodiode sensors with a laser which is reflected from the top surface of the cantilever. There are also other methods that are used, including optical interferometry, capacitive sensing, or piezoresistive cantilevers [74]. The schematic diagram of the AFM is shown in figure 1.10.

![Figure 1.10: The schematic diagram of the AFM, which copied from [21].](image)

When imaging soft or fragile samples such as polymeric thin films, there is some plastic deformation that can be introduced by the cantilever tip. Thus, different strategies have been devised in order to minimize the interaction between the tip and the surface, such as contact mode and non-contact mode (or tapping mode), with the optical technique chose for different materials, sample structures or measurement conditions.

The most common and simple mode used in the AFM is the contact mode. For this, the tip is lowered down onto the sample surface until it receives a repulsive force with a mean value of $10^9$ N. There is an excessive tracking force which can be introduced on the sample surface through the probe during the measurement and scanning.

The non-contact mode raises the tip and hovers the probe above the sample surface at a distance of 50 to 150 Angstroms, to create an attractive field of van der
Waals force between the tip and surface. The attractive forces from the sample are substantially weaker than the repulsive forces used by the contact mode, thus it is required to obtain more information for data analysis such as the change in amplitude, phase, or frequency of the oscillating cantilever in response to force gradients from the sample. This non-contact method of doing AFM was suggested and invented by Martin et al. in 1987. Another non-contact method, tapping mode, was developed by Zhong et al. in 1993 for overcoming the loss of lateral resolution because of the increased tip-surface distance of the non-contact method. This method was applied to study suspended graphene membranes.

**SEM and TEM**

Another widely used and important microscopy method for thin film inspection is electron microscopy (EM). There are two types of EM which can be used; scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Both techniques are well developed and used for characterising epitaxy films and MEMS.

SEM scans a focused beam of electrons over a surface to image it. A high speed electron can have a short wavelength, as described by the de Broglie equation \( \lambda = \frac{h}{p} \), where \( h \) is Planck’s constant and \( p \) the momentum of the electrons. The resolution of SEM can be down to about 1 nm, which is significantly smaller than a traditional optical microscope as the wavelengths of white light are from 400 to 700 nm.

The main components of the SEM include a source of electrons, electromagnetic lenses, scanning coils, and an electron detector, and are all in a vacuum chamber, as shown in figure 1.11. When primary electrons in the beam interact with sample surfaces, they produce various signals such as backscattered electrons, secondary electrons, Auger electrons and X-rays, that can be used to obtain information about the surface topography and composition.

TEM is different from SEM as it mainly uses the secondary electrons emitted from the sample surface due to excitations by the primary electron beam as a surface reflection method. TEM requires that electrons pass through the specimen before being focused onto an image capturing device, thus, the samples normally are very thin. The images produced by TEM can have higher magnification and greater resolution than SEM. TEM images can obtain the crystalline structure directly, and analyse the elemental composition of samples. The schematic of a typical TEM is shown in figure 1.12.
1.4.9 X-ray Diffraction

The crystalline morphology is essential knowledge for materials science. The information about crystalline structure includes dimensions of the unit cell, the percentage of crystallinity, crystallite size, and orientation. X-ray diffraction (XRD) is a very common technique to study materials’ crystallinity. The measurement data of XRD can also offer more information on many important factors, including crystal quality, composition, tilt and strain state. The working principle of XRD specifically refers to Bragg’s Law, $\lambda = 2\sin(\theta)$, where $n$ is an integer value, $\lambda$ is the incident X-ray wavelength, $d$ the distance between crystal planes and $\theta$ is the incident angle of the beam. The details of XRD experimental setup is shown in figure 1.13.

1.4.10 Spectroscopic Methods

**Fourier Transform Infrared (FTIR) Spectroscopy**

Commonly, spectroscopic techniques are used to identify and study chemicals. However, FTIR spectroscopy is also a powerful tool that can be used for epitaxy film characterisation. There are two modes of FTIR spectroscopy: transmittance and reflectance. The measurement data from transmittance mode can offer information
on the material bandgap and certain defects and impurities in the epitaxy layers. The thickness of the layer can be measured by the reflectance mode which uses the interference of light. For transmittance mode, the infrared light beam is split into two separate beams. One of the beams passes through the sample, and the other one is used as a reference. Both beams are then re-directed towards and enter a detector. The re-combined light creates a spectrum by light interference with useful information which can be obtained using Fourier transform analysis.

**Raman Spectroscopy**

Raman spectroscopy is another powerful technique for material characterisation. It is widely applied in different fields such as physics, chemistry, biology, security and inspecting fraudulent artwork. For material studies, it can provide information about the distribution and orientation of the structure, the quality and strain state of crystals. There are three different Raman spectroscopic methods which can be used for surface studies: microprobe, internal reflection and surface-enhanced Raman spectroscopy. However, there are some problems that can influence the measurement results of Raman spectroscopy, such as the optical clarity, fluorescence, chemically complexity and scramble in the heterogeneous system.
1.5 Outline of Thesis

In this thesis the method of resonance testing is used to investigate the properties of materials forming suspended structures. In particular there is a focus on measuring mechanical properties for varying materials and structure sizes. In chapter 2, the classical theories of plate and membrane vibrations are introduced. The details of the developed laser scanning system and how it can be used to measure the dynamic vibration behaviour is described in chapter 3. In chapter 4, several different suspended thin films were investigated through linear resonance testing with small amplitude vibrations. Using the simplest model, the residual stress of each sample was found. In chapter 5, thin films were vibrated into the non-linear regime, and the non-linear vibration behaviour of thin films with fully clamped boundaries was studied. In chapter 6, the thermal effect induced by the probe laser was investigated. A dynamic thermal field created by the probe laser could potentially be used as a new tool for material characterisation. In chapter 7, the thermal cycling test was used as a tool for NDT studies where thin films had submicron or nanometer size cracks. Overall conclusions and suggestion for further work are presented in chapter 8.
Chapter 2

Theory of plates and membranes

This chapter discusses the classical theory of free vibration of plates and an ideal membrane. Further theoretical background of relevant topics is discussed at the start of each experimental chapter.

Stress and strain commonly exist in epitaxially grown thin films as a result of constraints imposed by the substrate. Stress in thin films is composed of three primary components: intrinsic stresses, thermal stresses and mechanical stresses [44]. The residual stress in a film/substrate structure at an arbitrary temperature is mainly governed by two parts: the intrinsic stress generated by the lattice mismatch between film and substrate during growth, and the thermal stress created by the different coefficients of thermal expansion (CTE) between thin film and substrate as the structure is cooled from the growth temperature. Stress in thin films is known to cause stiffness (yield) and reliability problems in microelectronic devices as deformation and fracture occurs and measurement of stress is useful [75, 76, 77].

The importance of thin films for modern technologies such as MEMS was presented in the first chapter. Their reliability is one of the most critical factors for applications of MEMS products. The mechanical properties of thin films or structures can partially influence the reliability of devices through e.g. the fatigue behaviours. Failure mechanisms due to fatigue can be classified into three types; process related failure mechanisms, in-use failure mechanisms, and packaging related failure mechanisms. Understanding the mechanical properties and structure dependability is the first step before applying materials and structures in devices [57, 78].

Researchers have used different types of equipment and various techniques to characterise film mechanical properties such as the residual stresses, as described in the
previous chapter. Each of the techniques has their own limitations and cannot be used as a universal tool to measure all types of materials or structures. For example, the wafer curvature measurement can introduce significant errors in the results where there is similarity of the rigidity of the film and the substrate, or a large deformation in the structure. The method of bulge testing often gives an over- or under-estimated residual stress because of the difficulty for positioning for measuring the deflection at the exact centre. XRD and Raman spectroscopy are highly localized measurements and can only be applied to a limited class of materials, for example the XRD is difficult to use to analyse noncrystalline materials [17, 74].

In this chapter, the method developed and investigated in this work, that of acoustic oscillation and measuring free-vibration resonance frequencies for investigating thin films mechanical properties, is presented in detail with the basic theoretical description. Depending on the material properties and range of resonant frequencies, a thin film can be classified as plate-like or membrane-like [79, 80]. The resonant frequency of a solid is often considered to be an absolute quantity, which depends only on the mass density, elastic constants, residual stress and boundary conditions. However, in reality, thin film mechanical behaviour is more complex than the simple classification as plate- or membrane-like [79].

2.1 Elastic thin films

The elastic behaviour observed during vibration measurements is described by Hooke’s law. Within the elastic regime in a solid, a small displacement is linearly proportional to a force, and the displacement disappears when the force is removed. The relationship between the deformation and internal restoration forces of the solid is given by stress and strain within the material. Expressed in Cartesian coordinates, the stress $\sigma$ and strain $\varepsilon$ are given by [75, 81, 82, 83, 84]

$$\sigma = \begin{bmatrix} \sigma_{xx} & \tau_{xy} & \tau_{xz} \\ \tau_{yx} & \sigma_{yy} & \tau_{yz} \\ \tau_{zx} & \tau_{zy} & \sigma_{zz} \end{bmatrix}$$ (2.1)

$$\varepsilon = \begin{bmatrix} \varepsilon_{xx} & \gamma_{xy} & \gamma_{xz} \\ \gamma_{yx} & \varepsilon_{yy} & \gamma_{yz} \\ \gamma_{zx} & \gamma_{zy} & \varepsilon_{zz} \end{bmatrix}$$ (2.2)

where $\sigma$ and $\varepsilon$ are the normal stress and normal strain (direction perpendicular to the plane), $\tau$ and $\gamma$ are the shear stress and shear strain (direction in plane). Stress
is the force per unit area, $\sigma = \frac{F}{A}$, and strain describes changes in stretching (normal) as $\varepsilon = \frac{dL}{L}$, or angled stretching (non-normal).

In general, for a linearly elastic solid, the relationship between the stress and strain is $\sigma_{ij} = C_{ijkl}\varepsilon_{kl}$, where the $i, j, k, l$ represent any of the three spatial directions of $x, y, z$. $C_{ijkl}$ is the fourth-order stiffness tensor. For an homogenous and isotropic material, the stiffness tensor is \[ \begin{bmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{bmatrix} \tag{2.3} \]

where $C_{12} = \lambda$ and $C_{44} = \mu$ are called the Lamé constants, and $C_{11} = \lambda + 2\mu$. These elastic moduli are material microstructure dependent, and grain size, defects, and also external factors such as temperature or applied load can lead to variations.

Using Hooke’s law to describe the stress and strain, simplified to give the basic equations for a three-dimensional isotropic system, a set of equations for strain and stress are produced,

$$
\varepsilon_{xx} = \frac{1}{E}[\sigma_{xx} - \nu(\sigma_{yy} + \sigma_{zz})] \quad \gamma_{xy} = \frac{2(1 + \nu)}{E} \tau_{xy} \quad \gamma_{xy} = \gamma_{yx} \tag{2.4}
$$

$$
\varepsilon_{yy} = \frac{1}{E}[\sigma_{yy} - \nu(\sigma_{xx} + \sigma_{zz})] \quad \gamma_{xz} = \frac{2(1 + \nu)}{E} \tau_{xz} \quad \gamma_{xz} = \gamma_{zx} \tag{2.5}
$$

$$
\varepsilon_{zz} = \frac{1}{E}[\sigma_{zz} - \nu(\sigma_{xx} + \sigma_{yy})] \quad \gamma_{yz} = \frac{2(1 + \nu)}{E} \tau_{yz} \quad \gamma_{yz} = \gamma_{zy} \tag{2.6}
$$

where $E$ is Youngs modulus and $\nu$ is Poissons ratio.

Deposition of a thin film on a substrate leads to biaxial stress in the film, where $\sigma_{zz} = 0$ and $\sigma_{xx} = \sigma_{yy}$. Therefore,

$$
\sigma = \sigma_{xx} = \sigma_{yy} = M\varepsilon, \tag{2.7}
$$

$$
\varepsilon_{zz} \approx -\frac{2\nu}{1 - \nu} \sigma, \tag{2.8}
$$

27
\[ M = \frac{E}{1 - \mu}, \quad (2.9) \]

where \( M \) is the biaxial elastic modulus of the film \[75\].

For thin films made from a single crystal or polycrystalline material with strong crystallographic texture, the biaxial elastic modulus has to consider the effects of anisotropic properties when the stress is applied in different planes. Some results for a cubic single crystal film are given below as \[57\]:

\[
M(001) = C_{11} + C_{12} - \frac{2C_{12}^2}{C_{11}}, \quad (2.10)
\]

\[
M(111) = \frac{6C_{44}(C_{11} + 2C_{12})}{C_{11} + 2C_{12} + 4C_{44}}, \quad (2.11)
\]

\[
M(100) = C_{11} + C_{12} - \frac{C_{12}(C_{11} + 3C_{12} - 2C_{44})}{C_{11} + 2C_{12} + 4C_{44}}. \quad (2.12)
\]

The residual stresses in thin films can arise from three mechanisms; epitaxial stresses, thermal stresses and intrinsic (or growth) stresses. When films are perfectly coherent with the substrate at an interface, where the lattice dimensions are equal, the crystal lattices in the film and the substrate would perfectly line up. The elastic accommodation strain of lattice misfit between film and substrate can simply be given by

\[
\varepsilon_{mf} = \frac{a_s - a_f}{a_f},
\]

where \( a_s \) and \( a_f \) are the lattice sizes of the substrate and the film. However, this expression for the misfit strain only holds when the film is sufficiently thin. When the film thickness increases, the perfect coherence at the interface will be broken and misfit dislocations will be formed. These dislocations would release part of the misfit stress, and the epitaxial strains would become smaller \[44\].

The thermal stress results from a temperature change following growth and different thermal expansion in the film and the substrate. The elastic stress created to ensure the film and substrate remain together is

\[
\sigma = \int_{T_0}^{T_f} \Delta \alpha M dT,
\]

where \( \Delta \alpha \) is the difference between the thermal expansion coefficients of the film and substrate. The thermal effects on vibration measurements are discussed in detail in chapters 6 and 7.
The intrinsic stress results from defects such as dislocations in the films. It is developed in films during the epitaxial growth because of non-equilibrium growth conditions. In addition, the intrinsic stresses may be affected by the surface stresses, crystalline coalescence, grain size and boundaries, vacancy annihilation, impurities and phase transformations.

2.2 Stress Determination Using Resonance Testing

In order to have reliable implementation for thin films in practical devices, a good understanding of the residual stress in the film is very important. The residual stress investigation methods can fall into two groups; one covers static methods such as load-deflection, while the other includes dynamic methods such as the flexural-vibration method which is based on vibration theory and related to the resonant frequencies of the structure.

There are two classical descriptions for wave motion in a two-dimensional system which can be used to determine the mechanical properties of thin films; one assumes the thin film behaves as an elastic plate, with the dominant restoring forces due to the bending of the material. The other considers the thin film behaving as a flexible membrane under tension (tensile stress) along its boundary.

2.2.1 Vibrations of Plates

Plate theory development has used a blend of experiments and theories. The most comprehensive source for plate vibration studies is a compendium produced by Leissa and published in 1969 [86]. There have been numerous mathematicians, scientists and engineers who contributed their knowledge to the theory development. In 1776 Leonard Euler started to investigate the free vibration behaviour of plates. The description of the partial differential equation of plates vibration was developed by mathematician Joseph-Louis Lagrange in 1811. More historical details of the development of plate theory are in the book Stresses in Beams, Plates, and Shells by Ansel C. Ugural [75].

During vibration, the plate will be bent into a curvature, and the lateral deflection (assuming no variation in plate thickness) can be defined as \( w(x, y) \) when using Cartesian coordinate. Strain-stretch relations in a body vary from point to point along an axis, hence the normal strain in 2D is defined by \( \varepsilon_x = \frac{\partial u}{\partial x} \) and \( \varepsilon_y = \frac{\partial v}{\partial y} \) (\( \varepsilon_{ii} \) simplified to \( \varepsilon_i \)) and a shear strain \( \gamma_{xy} = \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} \) exists, where \( u \) and \( v \) are the stretches under the axial load. For the curvature deformation, \( u \) and \( v \) are related
to the lateral deflection via
\[ u = -z \frac{\partial w}{\partial x}, \quad u = -z \frac{\partial w}{\partial y} \] (2.13)

Therefore, the strain in the bent plate can be expressed as
\[ \varepsilon_x = -z \frac{\partial^2 w}{\partial x^2}, \quad \varepsilon_y = -z \frac{\partial^2 w}{\partial y^2}, \quad \gamma_{xy} = -2z \frac{\partial^2 w}{\partial x \partial y}. \] (2.14)

The stress-strain relationship in an isotropic and homogeneous material can be obtained by substituting equation 2.14 into equations 2.4 and 2.5, yielding
\[ \sigma_x = \frac{E}{1 - \nu^2} (\varepsilon_x + \nu \varepsilon_y) = -\frac{Ez}{1 - \nu^2} \left( \frac{\partial^2 w}{\partial x^2} + \nu \frac{\partial^2 w}{\partial y^2} \right), \] (2.15)
\[ \sigma_y = \frac{E}{1 - \nu^2} (\varepsilon_y + \nu \varepsilon_x) = -\frac{Ez}{1 - \nu^2} \left( \frac{\partial^2 w}{\partial y^2} + \nu \frac{\partial^2 w}{\partial x^2} \right), \] (2.16)
\[ \tau_{xy} = \frac{E}{2(1 + \nu)} \gamma_{xy} = -\frac{Ez}{1 + \nu} \frac{\partial^2 w}{\partial x \partial y}. \] (2.17)

The stresses distributed over the thickness of the plate produce bending moments, twisting moments, and vertical shear forces. The resultant bending and twisting moments can be written in terms of curvatures and deflection, giving formula such as
\[
\begin{bmatrix}
M_x \\
M_y \\
M_{xy}
\end{bmatrix} = \int_{-h/2}^{h/2} \begin{bmatrix}
\sigma_x \\
\sigma_y \\
\tau_{xy}
\end{bmatrix} z dz, \] (2.18)

then,
\[ M_x = -D \left( \frac{\partial^2 w}{\partial x^2} + \nu \frac{\partial^2 w}{\partial y^2} \right), \] (2.19)
\[ M_y = -D \left( \frac{\partial^2 w}{\partial y^2} + \nu \frac{\partial^2 w}{\partial x^2} \right), \] (2.20)
\[ M_{xy} = -D(1 - \nu) \frac{\partial^2 w}{\partial x \partial y}. \] (2.21)

where \( D = \frac{Eh^3}{12(1 - \nu^2)} \) is the flexural rigidity, \( h \) is the thickness, and \( M_{ij} \) are the moments, distinguishable from the biaxial elastic modulus by the subscripts.

There are also two vertical shear forces \( Q_x \) and \( Q_y \) from the strain components \( \gamma_{xz} \) and \( \gamma_{yz} \) produced by the plate bending. In fact, they have the same magnitude
as the surface loading and moments to balance forces and achieve the equilibrium state. The total vertical momentum per unit area is then given by

$$\frac{\partial Q_x}{\partial x} + \frac{\partial Q_y}{\partial y} = \frac{\partial^2 M_x}{\partial x^2} + 2 \frac{\partial^2 M_{xy}}{\partial x \partial y} + \frac{\partial^2 M_y}{\partial y^2},$$

(2.22)

thus,

$$\frac{\partial Q_x}{\partial x} + \frac{\partial Q_y}{\partial y} = D\left( \frac{\partial^4 w}{\partial x^4} + 2 \frac{\partial^4 w}{\partial x^2 \partial y^2} + \frac{\partial^4 w}{\partial y^4} \right).$$

(2.23)

Using Newtons law, \( F = ma \), therefore, the free vibration of a homogenous and isotropic plate can be described as

$$D\left( \frac{\partial^4 w}{\partial x^4} + 2 \frac{\partial^4 w}{\partial x^2 \partial y^2} + \frac{\partial^4 w}{\partial y^4} \right) = -\rho \frac{\partial^2 w}{\partial t^2},$$

(2.24)

where \( \rho \) is mass density per unit area of the plate, \( t \) is time, and the sign come from direction.

To solve equation 2.24 for a plate with a fully clamped boundary, there are several methods that can be used, such as the Rayleigh-Ritz method, Weinsten’s method, integral equations, and the variational approach. More details of each of these are beyond the scope of this section but are given in the book Vibration of plates by Leissa [86].

A general formula for resonant frequency for a square shaped plate can be expressed as

$$f_{mn} = \frac{K_{mn}}{2\pi a^2} \sqrt{\frac{D}{\rho}},$$

(2.25)

where \( K_{mn} \) is the mode coefficient (tables of mode coefficients are available in the literature [86]), and \( a \) is the lateral dimension size.

### 2.2.2 Vibrations of Membranes

Membranes can be described as a very thin plate with no flexural rigidity \( (D = 0) \). Therefore, ideal membranes have no stiffness of their own, and the restoring force of oscillation is supplied by an externally applied tension \( (T) \) such as the residual stresses [87].

As with plate theory, membrane vibration has also been studied for a long time [88]. The study of vibrating membranes can be traced back about three centuries. Since thin film structures are now being applied to more practical applications and are becoming more important for modern technologies, membrane studies have received
more attention. A large amount of literature presents membrane studies for both the theoretical and experimental side, and covers linear and non-linear vibrations. Here, only the classical membrane vibrating in the linear regime will be presented, and the non-linear behaviours are discussed in chapter 5.

Considering the restoring force on a membrane arising only from a constant biaxial stress \( \sigma_0(\sigma_x = \sigma_y = \sigma) \) and ignoring the thickness variations during oscillation, when a point on the membrane is deflected by a small distance \( dw \), the restoring force is

\[
F_x = \sigma dy \left[ \frac{\partial^2 w}{\partial x^2} \right]_{x+dx} - \left( \frac{\partial w}{\partial x} \right)_x = \sigma dy \frac{\partial^2 w}{\partial x^2} dx, \tag{2.26}
\]

\[
F_y = \sigma dx \left[ \frac{\partial^2 w}{\partial y^2} \right]_{y+dy} - \left( \frac{\partial w}{\partial y} \right)_y = \sigma dx \frac{\partial^2 w}{\partial y^2} dy. \tag{2.27}
\]

The total force on the element of \( dx \, dy \) is \( F = F_x + F_y \), so the equation of vibration obtained from Newton’s law \( F = ma \) gives

\[
\sigma dx dy \left( \frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} \right) = \rho dx dy \frac{\partial^2 w}{\partial t^2}, \tag{2.28}
\]

for an element of mass \( m = \rho dx dy \). Then, the equation of motion is

\[
\sigma \left( \frac{\partial^2 w}{\partial x^2} + \frac{\partial^2 w}{\partial y^2} \right) = \rho \frac{\partial^2 w}{\partial t^2}. \tag{2.29}
\]

Solving the oscillation for a stretched membrane having rectangular fixed boundaries, the deflection can be expressed as

\[
w(x, y, t) = Ae^{i(\omega t - k_x x - k_y y)} = X(x)Y(y)T(t), \tag{2.30}
\]

where \( \omega \) is the angular frequency, and \( k \) is the wave number with \( k_x \) and \( k_y \) on each axis and satisfied by

\[
k = \frac{\omega}{c} = \sqrt{k_x^2 + k_y^2}, \tag{2.31}
\]

where \( c \) is the wave velocity. \( X, Y, \) and \( T \) are separable functions of \( x, y, \) and \( t \) respectively. From substituting equation [2.30] into equation [2.29] and rearranging, one obtains

\[
1 \frac{\partial^2 T}{\partial t^2} = \frac{c^2}{X} \frac{\partial^2 X}{\partial x^2} + \frac{c^2}{Y} \frac{\partial^2 Y}{\partial y^2}, \tag{2.32}
\]

which can be solved to give the separable functions as

\[
T(t) = Asin(\omega t) + Bcos(\omega t), \tag{2.33}
\]

32
\[ Y(y) = C \sin(k_y y) + D \cos(k_y y), \] (2.34)\

\[ X(x) = E \sin(k_x x) + F \cos(k_x x), \] (2.35)

where \( A, B, C, D, E, F \) are constants which can be obtained from boundary conditions.

For fully clamped boundaries, at positions along the lines \( x = 0 \) to \( l_x \) for \( y = 0, l_y \), and \( y = 0 \) to \( l_y \) for \( x = 0, l_x \), where \( l \) is the lateral dimension of the membrane, the deflection is zero. Applying these boundary conditions to the equations above and substituting into 2.30 leads to

\[ w = W \sin(k_x x) \sin(k_y y) e^{i \omega t}, \] (2.36)

where \( W \) is the maximum amplitude of the transverse displacement, and

\[ k_x = \frac{n_x \pi}{l_x}, \quad n_x = 1, 2, 3, \ldots \] (2.37)

\[ k_y = \frac{n_y \pi}{l_y}, \quad n_y = 1, 2, 3, \ldots \] (2.38)

Therefore, from

\[ f = \frac{\omega}{2\pi} = \frac{c}{2\pi} \sqrt{k_x^2 + k_y^2} \] (2.39)

the resonant frequencies for the allowed modes of free vibration of an ideal membrane are given by

\[ f_{n_x n_y} = \frac{1}{2} \sqrt{\frac{\sigma}{\rho}} \sqrt{\left( \frac{n_x}{l_x} \right)^2 + \left( \frac{n_y}{l_y} \right)^2}. \] (2.40)

When the membrane shape becomes a square, the allowed frequencies from pairs of modes for an ideal membrane will have the same frequency, i.e. \( f_{ij} = f_{ji} \) even when \( i \neq j \). This is called a degeneracy. In fact, each of the vibration modes are different, and have different wave functions \( \psi_{ij} \psi_{ji} \), but can still have the same frequency. The vibrations can be standing waves, with different partial values \( a \) and \( b \) satisfying

\[ w = (a \psi_{ij} + b \psi_{ji}) \cos(\omega t), \quad \text{with} \quad a^2 + b^2 = 1. \] (2.41)

A splitting of normal modes of vibration (i.e. \( f_{ij} \neq f_{ji} \) for a square membrane) is related to the material properties, for example any anisotropy in the material or the boundary conditions, giving separate frequencies for modes \( ij \) and \( ji \). This is
discussed further in chapter 4.

2.2.3 Thin Films

Given both classical theories described above, identifying the regime of operation of a thin film (i.e. whether it between as a plate or membrane) can be done by comparing the flexural rigidity and the values of the resonant frequencies. For a typical material these frequencies can be an order of magnitude different \[80\], and the thin film can be designated as plate or membrane. However, in the real world, the elastic force in a bending system cannot be ignored. Thus, a free vibration thin film is most fully considered as a thin plate under in-plane tension. Blevins developed a formula which included both residual stress and flexural rigidity to describe the resonant behaviour of fully clamped rectangular diaphragms \[89\], where

\[
f_{mn} = \sqrt{\left(\frac{\pi}{2} \sqrt{\frac{(m+0.5)^4}{a^4} + \frac{(n+0.5)^4}{b^4} + \frac{2J_m J_n}{a^2 b^2} \sqrt{\frac{Eh^2}{12 \rho (1/\nu^2)}}} \right)^2 + \frac{\sigma_m J_m}{4 \rho a^2} + \frac{\sigma_n J_n}{4 \rho b^2}},
\]

where

\[
J_n = \left(n + \frac{1}{2}\right)^2 \left[1 - \frac{2}{\pi(n + \frac{1}{2})}\right],
\]

\[
J_m = \left(m + \frac{1}{2}\right)^2 \left[1 - \frac{2}{\pi(m + \frac{1}{2})}\right],
\]

where \(m\) and \(n\) are mode numbers, \(J_m\) and \(J_n\) are mode dependent numerical values, \(a\) and \(b\) are side lengths, and \(\sigma_m\) and \(\sigma_n\) represent the biaxial stress.

Waitz et al. also reconstructed the formula for free vibration thin films, giving an angular frequency of \[90\]

\[
\omega = \sqrt{\frac{Eh^2}{12 \rho} k^2 + \frac{\sigma}{\rho}}.
\]

To simplify the data analysis for a small deflection amplitude, the main restoring force on the thin film vibration can be approximated by only considering the constant residual stress, given by the static pre-stress, and therefore, the motion is approximately linear, and close to that of an ideal membrane. The resonant frequencies of thin films in this regime are given by equation 2.40 without considering air damping \[80\] (see next section). Therefore, the resonant frequencies in these measurements are simply calculated using equation 2.40 when the thin film is in the linear vibration regime under small oscillations, measured under vacuum.
2.2.4 Damping and Quality factors (Q-factors)

When thin films vibrate in air, there is a downward frequency shift compared to the natural resonant frequency in a vacuum. This is due to the energy dissipation is a viscous damping mechanism, and is vibration-condition dependent. The resonant frequencies obtained in air with damping, rather than in vacuum can be corrected by using the non-dimensional added virtual mass incremental (NAVMI) factors \([91, 92]\),

\[
f_{\text{air}} = \frac{f_{\text{nm}}}{\sqrt{1 + \Gamma \beta}},
\]

where \(\beta = \rho_{\text{air}} l / \rho h\) is the thickness correction factor, \(\rho_{\text{air}}\) is the density of air, \(h\) is the thickness of the membrane, and \(\Gamma\) is the non-dimensional added virtual mass incremental (NAVMI) factor, which is determined by boundary conditions and mode shape.

There are two parameters typically used to characterize a damped oscillator; \(\omega_0\) and \(\gamma\). \(\omega_0\) is the undamped oscillation angular frequency as discussed above, and \(\gamma\) is the reciprocal of the time required for the energy to decrease to \(1/e\) of its initial magnitude. These two parameters together define a new parameter called the quality factor (Q-factor), given by

\[
Q = \frac{\omega_0}{\gamma}.
\]

The larger the value of \(Q\), the slower the energy is dissipated, giving a greater number of cycles of free oscillation during amplitude decay and suggesting a higher quality resonator.

A common method to measure the Q-factor for oscillators in experiments is the bandwidth method, as known as the -3 dB bandwidth method or the half maximum bandwidth method. The formula of this method is expressed as

\[
Q = \frac{f_{\text{res}}}{\Delta f},
\]

where \(f_{\text{res}}\) is the resonant frequency measured at the position of the maximum amplitude of displacement (\(A_{\text{max}}\)), \(\Delta f = f_2 - f_1\) is the frequency bandwidth, and \(f_1\) and \(f_2\) are the frequencies where the amplitude is \(A_{\text{max}}/\sqrt{2}\).

For MEMS or NEMS resonators, there exist multiple energy dissipation mechanisms, many of which are not well understood. The three most significant energy loss mechanisms other than air damping are anchor loss, thermoelastic damping, and intrinsic material loss. The air damping is generally neglected in scientific mea-
surements because most of the resonators are tested in a vacuum system. However, air damping is significant for real world applications in devices which are operated at non-vacuum pressures. More details are given in reference [93, 94, 95, 96].

2.3 Summary

- This chapter has discussed simple, classical theories for plate and membrane vibrations.

- Thin films can be approximated as either plate or membrane. The real picture is generally a combination of the two, but is generally close to one of the two, and can be determined by the magnitude of the vibration frequencies in many situations.

- This theory has assumed linear vibrations at a constant temperature.

- Theories of non-linear behaviour and the effect of thermal expansion are given in the relevant experimental chapters, 6 and 7.
Chapter 3

Experimental Details

The following chapter describes the experimental details for this study, including the experimental apparatus, sample preparation, measurement procedures, and simple modelling of vibration patterns. The sample fabrication uses techniques developed in the literature and by the Nano-Silicon group at Warwick. The experimental setup at the start of research used a simple 1D scan system. 2D scanning, vacuum methods and thermal analysis have been developed as part of this PhD.

3.1 Sample Fabrication

Ge and 3C-SiC thin films have been grown onto Si substrates, and suspended thin film structures produced by etching part of the Si base. Some of the structured samples have been provided by the Nano-Silicon Group, while others have been produced by the author using layered material provided by the Nano-Silicon group. Details of the sample preparation are given here, starting with production of the layered materials.

3.1.1 3C-SiC

A typical growth temperature for 3C-SiC is very close to the melting point of Si (about 1410 °C) in order to gain high quality growth and maintain high crystallinity and growth rate for a micron scale thickness epitaxial layer [21 97]. Two types of monocrystalline 3C-SiC epitaxy thin film samples were used in this study: one was grown at high temperature, at 1350 °C at a pressure of 100 Torr in the LPE ACIS-M8 system using reduced pressure chemical vapor deposition (RP-CVD), the other thin films were grown within a cold-wall CVD reactor at a lower temperature of approximately 1200 °C and at a reduced pressure (smaller than 100 Torr) in the
ASM Epsilon 2000 system by RP-CVD. Both epitaxial growths were performed onto 100 mm diameter Si (001) substrates with standard 525 µm thickness on-axis. By controlling the growth time, thin film samples with various thickness were achieved. Sample thicknesses can be achieved from tens of nanometers to a few microns. Before the high temperature epitaxial growth, the Si substrate was carbonised with ethylene at a lower temperature. For the low temperature method to achieve high quality mono-crystalline 3C-SiC epitaxy layers, a buffer layer was used before 3C-SiC growth. Two types of buffer layer were used: a single layer or double layers. The single buffer layer was made from a silicon carbon alloy (Si$_{1-y}$C$_y$, y < 0.015) with tens of nanometers thickness between the Si substrate and 3C-SiC. Double buffer layers added another tens of nanometers thickness layer of silicon boron (Si$_{1-x}$B$_x$, x < 0.01) before the Si$_{1-y}$C$_y$ layer in order to reduce the edge effects when thin films are fabricated. All the thin films were grown by Gerard Colston who was a PhD student in the Nano-Silicon Group in the Physics Department at the University of Warwick, and more growth details are given in references [21, 97].

3.1.2 Ge

The Ge films were also grown by a PhD student, Vishal Ajit Shah, from the Nano-Silicon Group. The two temperature method was used to grow the Ge epitaxy layers, which has been refined to give high quality layers. The same type of Si wafer was used and polished before being used in the Ge epitaxy growth. The Ge layers were grown in the ASM Epsilon 2000E system by RP-CVD. The first deposition temperature was 400 $^\circ$C for the first 100 nm thick Ge layer, then, the following layer was grown at 670 $^\circ$C, and a final anneal was done at a temperature of 830 $^\circ$C for 10 minutes. More growth details are available in references [98, 18, 99].

3.1.3 Si$_3$N$_4$

The Si$_3$N$_4$ thin film was provided by AG Scheer research group is at Konstanz University. The thin film growth was done on a 500 µm thick commercial silicon wafer (100) on both sides. A layer of about 500 nm thickness was produced using Low Pressure Chemical Vapor Deposition [100].

3.1.4 Lithography

After achieving high quality films, the next step for sample fabrication is lithography. This process is a technique to transfer copies of a master pattern onto the surface of the epitaxy layer, with the pattern giving the geometry for etching. The most
commonly used type of lithography is photolithography, which transfers the patterns from a designed mask \[101, 102\].

A simplified process for photolithography and pattern transfer is summarized in figure 3.1. ProTEK photoresist has been used in the process \[21\]. It is sensitive to a UV light wavelength. Several methods can be used to deposit the liquid photosensitive materials as a coating onto the surface, including spin coating, spray coating, dip coating, meniscus coating, electrodeposition, and curtain coating. The spin coating method was used to produce these samples, using a resist spinner as shown in figure 3.2. The wafer was placed on the vacuum chuck and held by the vacuum. The spin speed used is commonly in a range from 1500-6000 rpm, depending on the viscosity of the photoresist \[101\].

The photoresist can be distinguished into two classes: positive or negative photoresist. The positive type can be removed by a developer (a chemical solvent) after exposure in UV radiation, while the negative type would remain after exposure and developer. Negative and positive photoresists are used in different circumstances.

### 3.1.5 Etching

Etching is the process to remove the exposed part without protection by the hardened photoresist to achieve the designed structure. Etching can be classified as wet etching and dry etching. Wet etching uses appropriate chemicals to etch and remove
materials. Dry etching uses a plasma system, such as reactive ion etching (RIE). Dry etching is a directional process and highly anisotropic [102]. To fabricate the suspended structures used here, part of the Si substrate was removed using a deep anisotropic wet etching process using 30% potassium hydroxide (KOH) at a temperature range between 70 and 90 °C for a few hours [21, 103, 104]. The etching time depends on the temperature and concentration of chemicals. A dry etch was later used to etch the 3C-SiC layer to give different thickness and patterns. The process of sample structure fabrication is shown schematically in figures 3.3 and 3.4. A few sample pictures were taken using an optical microscope and are shown in figure 3.5.

### 3.2 Equipment Construction for Vibration Testing

Preliminary vibrational methods were developed for a feasibility study reported in [80]. These used a 1D scanning stage and a laser interferometer to measure vibrations at a few points on the sample. During this PhD a full 2D scanning system and related analysis software and methods have been produced, along with equipment to do measurements at different pressures and temperature [105, 106]. The experiments have required development of a heating-cooling stage to control sample temperature, and adaption of vacuum equipment. This section describes
Figure 3.3: Schematic diagram for Si removal to fabricate a suspended structure.

1. Thin film grown on Si (001) grown by HP-CVD.
2. ProTEK photoresist is coated and patterned into squares on the underside Si (001) substrate by UV photolithography.
3. The structure is etched in TMAH solution 50°C for 16 hours to etch through the Si substrate.
4. Upon removal the membrane is rinsed in deionised water.
5. The membrane is placed in a piranha etch for ~5 mins to remove the ProTEK photoresist.
6. Finished and cleaned Ge or 3C-SiC-suspended membrane.

Figure 3.4: Schematic diagram of the process to fabricate patterns on 3C-SiC thin films.

1. 3C-SiC epilayer grown on Si (001) grown by RP-CVD
2. Surface of the 3C-SiC is patterned with standard photoresist.
3. Ni is evaporated on the 3C-SiC before lift-off leaving a highly resistant mask for dry etching.
4. 3C-SiC left exposed by Ni mask is dry etched using a plasma enhanced RIE-ICP etch.
5. The Ni is etched away in a solution of nitric, acetic and sulphuric acid.
6. The sample is placed within a TMAH bath for ~90 mins in order to etch under the 3C-SiC microwires.
7. Final suspended 3C-SiC microwires
Figure 3.5: Different sample images measured using an optical microscope.

Figure 3.6: Schematic diagram of simplified vibration experiment set-up.

Figure 3.6 illustrates a schematic of the simplified experimental setup, which includes three main components: an interferometer (produced by Intelligent Optical Systems, IOS), a sample holder with a temperature controller system, and a vacuum chamber. The sample holder includes a ring shape transducer, a temperature sensor (Pt1000 thermistor), a thermally insulated fire brick with a hole in the middle and small gaps on its edge, a Peltier device, and a hot finger. Each part of the...
sample holder is individually moveable (see figure 3.7), but they can be fixed in the vacuum chamber using two thick metal plates clamped together. The sample and holder were placed inside a small vacuum chamber and pressure down to about 1 microbar were achievable using a rotary pump.

![Figure 3.7: Schematic diagram of the sample holder.](image)

3.3 Actuation Schemes

There are four different ways to excite a vibration on a resonator: magnetomotive, capacitive (electrostatic), thermoelastic and piezoelectric. To simplify the experimental set-up, and give well controlled vibration for all materials, the actuation of vibration used a piezoelectric transducer which made was from lead zirconate titanate (PZT) to generate vibrations. In order to keep the pressure the same on both sides of the thin film after it was mounted onto the transducer, the transducer was fabricated to a ring shape. The hole through the transducer also helped to remove the noise signal caused by the reflection of the detector laser beam from the transducer surface, when the sample is near-transparent for the detection laser. To remove all possible noise from unexpected reflections, a graphite disc was placed as the background (in the fire brick) to absorb the infrared laser. The transducer drive used an AC voltage from a function generator with an available frequency range from 100 Hz to 10 MHz, and a peak to peak voltage from 10 mV to 10 V. The input AC signal was controlled by a LabVIEW program.

The ring-shaped PZT had a 1 mm thickness, with 8 mm diameter of outer ring and 3 mm diameter of inner ring. Figure 3.8 shows the frequency response of the
transducer which was measured using an impedance meter and the interferometer. According to the measurement result from the impedance meter (figure 3.8 red and green lines), the main frequency of this transducer is around 780 kHz, with some small features for frequencies below 130 kHz. The transducer vibration strength with different driving frequencies is shown in figure 3.8 by the black line, which shows that the vibration strength depends on driving frequency, as well as the driving voltage.

This transducer was also used for the thin film non-linear vibration study described in chapter 5. The linearity of this transducer was checked over the range of driving frequency and voltage used. The results are showing in figure 5.8, and show that the transducer behaviour is linear in the region of interest.

3.4 Detection Schemes

Detection of small motion, i.e. nano or micron scale mechanical oscillation, needs a highly sensitive measurement system [107][108]. There are again several different techniques which can be used. The most common three ways are capacitive, AFM, and optical (using an interferometer) [109]. The advantage of the capacitive method is it can be used as a signal generator and detector at the same time, but it cannot give the details of the vibration at each position on the sample and hence is unable to map the shape of the resonant modes. Using AFM to measure the deflection has a risk of breaking the sample, and also might introduce an external force to affect
the vibration, thus influencing the accuracy of measurement results [110].
Recent enhancements in interferometric techniques have proven that laser interferometry can be an accurate tool for dynamic displacement measurement, and it is a non-contact and can be a non-destructive process. A commercial two wave mixer laser interferometer (homodyne interferometer) produced by Intelligent Optical System (IOS-AIR-1550-TWM) was used in this study [111, 112, 113, 114, 115, 116, 117]. This system is sensitive to the transverse vibration displacement with a sensitivity of \(4 \times 10^{-7} \text{ nm} (\text{W/Hz})^{1/2}\) and detects it with a continuous wave infrared laser operating at a wavelength of 1550 nm, with variable power from 50 mW to 2 W. The detection bandwidth is 125 MHz with sensitivity from 100 Hz upwards. The system offers two different laser spot size diameters: 30 \(\mu\text{m}\) and 200 \(\mu\text{m}\), but the size can be varied by using an adaptor (lens system). The working principle and equipment details are given in references [118, 119].

3.5 Temperature Control

For thermal cycling tests, a self-made temperature control system was combined with the sample holder. This implemented a Peltier device combined with a copper sheet (used as a hot finger) to transfer the heat from Peltier to the thin film directly. The maximum temperature on the sample obtained was 105 °C at microbar pressure. The temperature was measured using a platinum wire sensor (Pt1000 thermocouple) combined with a multimeter Keithley 2100 which used the four wire method to measure its resistance. The sensitivity and accuracy of this thermocouple is 0.3 °C over a temperature range from -50 °C to 600 °C. It was placed close to the edge of suspended boundary on samples, and measurements were made once thermal equilibrium was reached.

3.6 Data Acquisition and Analysis

A 2D scanning system was produced using two linear stages which had minimum step sizes of 6.45 \(\mu\text{m}\) and 4.25\(\mu\text{m}\). These were controlled by a LabVIEW program which controlled a raster-scan over the sample. Data from the IOS interferometer was recorded by an oscilloscope and the LabVIEW program at each position in the scan. This measured the DC reflected signal and used this calibrate the AC vibration signal and give the exact displacement during the oscillation. As noted from the theoretical formule in chapter 2, the most important information for thin film characterisation through the resonance method is the resonant
frequencies and their related mechanical mode numbers.

Obtaining these two bits of information by interferometric techniques involves two main steps: (i) scanning in frequency, with the laser interferometer spot at a fixed position (at the centre, a quarter of mid-line, or a quarter of diagonal) on the suspended thin film in order to be sensitive to most resonant frequencies; (ii) full two-dimensional scanning to image the resonant modes with a driving frequency found from the frequency spectrum which was obtained in the first step for each mode.

Vibrations of the thin films were studied over a frequency range from 30 kHz to a few MHz, depending on the residual stress and thin film lateral dimension sizes. Vibrations were generated by the ring-shaped PZT transducer which was excited using a continuous sinusoidal AC voltage from a function generator, with vibrations coupled through the Si substrate into the thin film using solvent-free glue. Measurements were repeatable after pressure cycling showing that the coupling remained constant once each sample had been placed on the transducer. The amplitude of vibration depended on the quality of this coupling, as well as actuation voltage, frequency used, and mode shape. The pressure value was varied from atmospheric pressure to about one microbar. The driving voltage for the transducer was between 1 mV (when using an attenuator) and 10 V. A schematic diagram of the measurements is shown in figure 3.9.

To identify the resonant frequencies a frequency sweeping measurement was used, which had to consider the required accuracy (or resolution) when setting the scanning step size ($df$). The influence of scan step size is shown in figure 3.10 for a 3C-SiC thin film at microbar pressure. A step size of 1 kHz was too large to identify all resonance and obtain an accurate Q-factor, while for this sample 0.01 kHz steps
gave an excellent measurement. After achieving the frequency spectrum, each peak was fitted with a Lorentzian function.

Figure 3.10: Frequency scan results with different scan step size for a 3C-SiC thin film at microbar pressure. The inset shows the measurements zoomed in to the different resonances.

3.7 Measurement in the Linear Regime

For thin film characterisation in the linear vibration regime, the measurements steps to collect the data and data analysis are as described above. However, the vibration displacements were small and hence the signal noise could be quite strong in comparison. Figure 3.11 (a) shows example data obtained from a Ge sample at atmospheric pressure, which is noise dominated. Therefore, a narrow bandpass digital filter with ±1.5 kHz bandwidth around the driving frequency was applied to the signal processing done using LabVIEW, and both raw data and filtered data are
saved separately, giving the results shown in figure 3.11 (b) for the filtered signal. A clear vibration matching the frequency applied was found.

Figure 3.11: The detected data using interferometer; (a) raw data, (b) filtered data using digital filter from LabVIEW.

3.8 Measurement in the Non-Linear Regime

Non-linear oscillations are observed when a large amplitude vibration (see chapter 5) is generated at low pressure. The oscillations observed exhibited a superposition of multiple frequencies rather than a simple sinusoidal wave, and thus the bandpass filter was removed for all the non-linear measurements during the frequency scan. As the non-linear resonant amplitude curve is asymmetric, in which the resonant frequencies are pulled either to higher frequency for material hardening, or to the lower frequency for softening (more details in chapter 5), the frequency sweep was measured for both increasing and decreasing the driving frequency, to confirm the asymmetry.
The non-linear oscillations are generated by a large amplitude vibration, thus a measurement to investigate the critical displacement was done by increasing the excitation voltage at a natural resonant frequency, driving the vibration from the linear regime until non-linear oscillations occurred. The non-linear oscillation mode shapes and their amplitude-time traces were measured by 1D and 2D scanning.

### 3.9 Measurements for Thermal Effects

Two types of thermal effects were examined individually. One was a localized thermal influence induced by the probe laser which must be considered when analysing sample behavior. Another thermal effect is uniform heating induced by the Peltier device, or by heating transfer from the local heating due to the laser after reaching thermal equilibrium.

As some of the residual stress is formed by the difference of thermal expansion between the substrate and thin film, resonant frequency shifts can be observed with a temperature variation. The Q-factor also can be impacted by the thermal influence at the boundaries. For the local heating effects, the sample temperature was monitored using a thermal imaging camera (CEDIP infrared systems-Titanium Series) and a Pt1000 thermal sensor (thermocouple) placed close to the edge of a boundary on the sample, when the sample was at atmospheric pressure outside of the vacuum chamber to enable operation of the camera. The incident laser power was varied from 20 mW to 2 W, depending on the thin film material and thickness. After the sample was put into the vacuum chamber, the sample temperature could only be measured by the Pt1000 because the window on the chamber affected the measurement done by the thermal camera.

For the uniform thermal heating, the first three resonant responses were studied in a temperature range from 23 °C to 97 °C at the reduced pressure of 1.34 microbar. To simplify the measurement, the temperature dependence of the Q-factor and the residual stress change were mainly studied for the fundamental resonance once initial experiments had been performed to support this simplification, with the frequency scan (f-scan) done at the samples’ mid-positions with a fixed excitation voltage (10mV). The fundamental mode was chosen as it has a cone shape which is symmetric and does not have mode splitting or mixing. The temperature control was set using the thermal cycling system which was controlled and monitored by the LabVIEW program, written by the author. Each measurement at a different temperature was done once the sample reached thermal equilibrium.
3.10 Air Damping

It is important to consider air damping, especially during heating and cooling cycles which can affect the pressure. For vacuum measurements, the pump was left on the whole time to stabilise the pressure. If the pump was not left on, a small leak led to a gradual repressurisation. The effect of this is shown in figure 3.12 for the pump switched off over a period of time.

Figure 3.12: Frequency shift and Q-factor reduction because of repressurisation due to a leak.

3.11 Modelling

Finite element simulation of thin film resonance and uniform thermal effects were modelled using the commercial software COMSOL. There are two modes in the software which can be used to simulate the thin film resonance; one is done directly using the membrane mode to do the simulation, the other is to simulate the thin film as a plate under tension using the solid mechanics mode. The results from both simulation models and calculation based on the theoretical formula of ideal membranes are plotted in figure 3.13 showing the relation between the side length and the fundamental resonant frequency change. Some of the simulated resonant modes are presented in figure 3.14.

The thermal effect on the suspended thin film with a thick substrate cannot be simulated using the membrane mode because this mode does not include the substrate in the modelling. Therefore, the best option to simulate the thermal effects on this type of sample is through the mode of solid mechanics. There are also two options which can be chosen for the modelling design: 2D model and 3D model. The 2D model is suitable for these samples as they have symmetric geometry. The 2D modelling can save lots of memory and time for the simulation.
Figure 3.13: Ratio of frequency change with different lateral dimensions, results from simulation and calculation.

Figure 3.14: Resonant mode amplitudes from simulation results.
3.12 Summary

- Fabrication details of the thin film samples have been given.

- Details of the equipment developed, with data collection and data processing, during this PhD are given in detail.

- For low pressure measurements, be given to enable a stable pressure, acheived here by leaving a pump running.

- There is a small difference between MATLAB calculations and COMSOL simulations of the resonant frequencies for different membrane dimensions, but the results are close. Simulations show the expected magnitude of the displacement at each position for several modes.
Chapter 4

Thin Film Resonance in the Linear Regime

As described by the simple model and the classical mechanical dynamic theory outlined in chapter 2, some of the mechanical properties of thin films can be determined from the internal residual stress and measured using the eigenfrequencies of their normal modes of vibration. The frequency range of these modes can indicate whether the thin film material behaves more as a plate or membrane. The techniques which are used to measure the resonant frequencies are detailed in chapter 3.

In this chapter, experimental measurements, supported by the modelled vibrational response (presented in chapter 2), are presented to demonstrate and verify the principles of this technique, using improved methods compared to reference [80]. The discussion of the experimental measurements in this chapter is dedicated to the linear vibration regime measured at atmospheric pressure, in a light vacuum (mbar), or a medium vacuum ($10^{-3}$ mbar). The eigenfrequencies are pressure dependent due to the air damping impact, as discussed in chapter 3. The measurements are compared to the theory presented in chapter 2, and the average residual stresses calculated.

The investigation was applied to two different types of suspended thin film structures on a silicon (Si) substrate: a single material layer suspended structure, and a thin film composed of three layers of different materials, 3C-SiC, $\text{Si}_{1-y}\text{C}_y$ and $\text{Si}_{1-x}\text{B}_x$, where the value of $x$ is smaller than 0.01 and $y$ is smaller than 0.015. The single material layer suspended structure was produced for three different materials: germanium (Ge), cubic silicon carbide (3C-SiC), and silicon nitride (Si$_3$N$_4$). The size of these thin films had lateral dimensions ranging from 2.85 mm down to 310 $\mu$m, and thickness from 4 $\mu$m down to 100 nm. The sample fabrication details,
material properties and pictures of the structures are given in chapter 3. Because of
the range of sample sizes and geometries, splitting or mixing of the resonant modes
are observed on different samples.

4.1 Ge Thin Film with 700 nm Thickness, 860 × 865 µm Dimensions

In this section, the experimental results obtained from a suspended thin film which
was fabricated from a single crystalline Ge layer will be discussed. The Ge layer was
heteroepitaxially grown on a 100 mm diameter and 300 µm thick Si (001) substrate.
The suspended thin film had 700 nm thickness and was approximately square, with
860 (±20) µm × 865 (±20) µm lateral dimensions. The resonance response at
atmospheric pressure of this Ge thin film was first measured using a frequency scan,
by sweeping the driving frequency for the piezoelectric transducer with 1 kHz scan
step size from 100 kHz to 1 MHz with a continuous sinusoidal AC voltage of peak
to peak amplitude 1 V. The resonance spectra result, which was obtained for the
laser detector at a position close to the middle point on the thin film, is illustrated
in figure 4.1 and shows several peaks (black line). To eliminate the stack effect of
the actuation from the piezoelectric transducer and any transducer resonance, the
displacement was also assessed at a random position on the substrate of the sample
with 10 times stronger excitation voltage, shown by the red colour line in figure 4.1.
In figure 4.1, the first two peaks around 100 kHz show consistency between the fre-
quency sweep measurements on both the centre of the thin film and on the substrate.
Thus, the first two peaks have a high potential of being due to the stack effect of the
actuation from the piezoelectric transducer, and cannot be directly classified as thin
film resonances. However, where the thin film response shows a peak in the absence
of a peak measured on the substrate, the potential thin film resonant frequencies
can be identified. In figure 4.1, these are the peaks at 156 kHz, 248 kHz, 350 kHz,
410 kHz and at higher frequencies.
To confirm that the peaks are coupled with a mechanical resonance and identify each
resonance, the vibration response at each identified frequency was fully mapped
using two-dimensional (2D) scanning to classify the resonant modes. The mode
images were produced by measuring the peak to peak displacement of the vibration
at each position during the 2D-scan. The interferometric mapping image can then
be compared with the simulated model image shown in figure 3.13 in chapter 3,
to classify the resonant modes. The resonant frequencies were discerned from the
spectral peaks united with the 2D-scan modes map matched with the resonant mode
Figure 4.1: The response of the Ge thin film with 700 nm thickness and 860 x 865 µm lateral dimensions to excitation at different frequencies at atmospheric pressure, measured at a position around the centre of the thin film. A measurement was also done on the substrate at a random position, to deduce the impact from the transducer itself.

pattern from modelling. Several of the mode images produced from the 2D-scans are shown in figure 4.2 including the fundamental mode 1:1 (at 164 kHz), a set of modes which appear to be variants of 1:2 (at 248 kHz, 251 Hz, 254 kHz and 256 kHz), 2:2 (325 kHz), variants of mode 1:3 (at 350 kHz and 355 kHz), variants of mode 1:4 (at 458 kHz and 468 kHz), and the mixture modes of 2:3 + 3:2 (at 410 kHz), and 1:5 + 5:1 (at 698 kHz). There are lines which appear as a box surrounding the resonant pattern in some 2D mapping images in figure 4.2 this simply shows the edge between the suspended thin film and substrate, as discussed in chapter 3, and whether or not it is observed depends on thin film and substrate geometry and the laser beam orientation. The limitation of the laser spot size used in these experiments (about 200 µm diameter) means that it is not possible to map a clear image for higher level resonant modes with a complex bending pattern, because of the spatial averaging effect in the measurement and signal processing due to the mode dimensions being smaller than the spot size.

From the resonant frequencies observed, this suspended thin film behaved as a membrane rather than a plate as the measured resonant frequencies are in the 100 kHz
Figure 4.2: The resonant modes from 2D-scan measurement for Ge thin film with 700 nm thickness and 860 × 865 µm dimensions at atmospheric pressure. (a) Fundamental mode at 164 kHz, (b) 1:2 mode at 248 kHz, (c) 1:2 ± 2:1 with (70/30%) at 251 kHz, (d) 1:2 ± 2:1 with (50/50%) at 254 kHz, (e) 2:1 mode at 256 kHz, (f) 2:2 mode at 325 kHz, (g) 1:3 mode at 350 kHz, (h) 3:1 mode at 355 kHz, (i) 3:2 ± 2:3 mode with (50/50%) at 410 kHz, (j) 1:4 mode at 458 kHz, (k) 4:1 mode at 468 kHz, (l) 5:1 ± 1:5 mode with (30/70%) at 325 kHz.
plus range. The restoring force is therefore mainly dominated by the residual tensile stress, and the additional restoring force from the elastic bending is neglected.

Figure 4.3: Displacement response to excitation frequency in a smaller region for three resonant frequency ranges for the Ge thin film with 700 nm thickness and $860 \times 865 \mu m$ dimensions. (a) contained up to four different resonant mode in one peak which included 1:2, 1:2 $\pm$ 2:1, and 2:1 mode. (b) contained two resonant frequencies in one peak, the 1:3 and 3:1 modes. (c) has two peaks for mode 1:4 and 4:1.

To understand the set of multiple variants of a mode such as the resonances around 250 kHz which are identified as the 1:2 mode, the peak spectra around these three resonant modes were scanned in more detail, shown in figure 4.3. The scans cover the frequency ranges of 200 to 300 kHz, 340 to 370 kHz and 420 to 490 kHz, corresponding to the mode variants for modes of 1:2, 1:3 and 1:4. Figure 4.3 (c) shows a clear main resonance at 468 kHz, but also a potential further smaller peak at 458 kHz. The relatively smaller magnitude of the amplitude displacement in the peak at 458 kHz could be caused by the frequency scan measurement position being close to the node point of the resonant mode, but it also could be caused by the vibration strength of the piezoelectric transducer which has an amplitude corresponding with the driving frequency. According to figure 3.8, there is a small drop in the transducer vibration strength for driving frequency around 450 kHz. The assumption of a reduced driving force for 458 kHz mat therefore be accurate. Figures 4.2 (j) and (k) add weight to the assumption of transducer driving frequency response; note that, whilst the peak displacement shown in the scan at 458 kHz is smaller than that at 468 kHz, the displacements are a more similar order of magnitude to those shown in the frequency scan in figure 4.3 (c) for a lower-order mode. The behavior shown here offers some evidence of mode splitting observed on this thin film. The frequency difference between the two peaks for the 1:4 and 4:1 mode is about 10 kHz. The FWHM of the main resonance peak is 5.91 kHz, which is smaller than the difference between the peak values.

By contrast, in figures 4.3 (a) and (b), a visible peak splitting cannot be observed.
However, the peaks are not a clear single peak and there are many features which could potentially be due to the overlapping resonances. Fitting was done considering the resonances as a single peak in each frequency range, giving a FWHM of 35.98 kHz for (a) and 14.39 kHz for (b) respectively, which are far larger than the value of 5.91 kHz found in (c). Thus, it is possible that the peaks in part (a) and (b) actually contain several overlapped and hard to distinguish modes, where each extra feature in the peak could be considered as a potential resonant frequency.

To identify each of the potential resonances, 2D-scans were done at each peak frequency, within each resonance are shown in figure 4.2. The resonance around 250 kHz shows variable behaviour, with the behaviour changing from predominantly 1:2 at 248 kHz to 2:1 at 256 kHz, and demonstrating mode mixing in between these frequencies. This is potentially evidence of mode splitting for this mode, with a small frequency difference of 2 to 3 kHz between frequencies. Similarly, figures 4.2 (g) and (h) show the 2D-scans for two frequencies determined from figure 4.3 (b), with some evidence of mode splitting for this mode also.

Earlier research on a similar thin film sample gave a residual stress of 0.22 GPa from vibrational measurement and 0.182 GPa from X-ray diffraction strain measurement [80, 98] with details discussed in [21, 98]. Table 4.1 gives the predicted resonant frequencies for this current membrane based on a residual stress of 0.22 GPa at atmospheric pressure, using equation 2.46. Similarly, the equation can also be used to calculate the average tensile stress from each measured resonant frequency. The air damping effect index 0.35–0.45 for the full range of materials and geometries considered in this thesis, used in equation 2.46 is vibration mode and pressure dependent, as discussed in chapter 2, and values are given in the table 4.1. The average value of the residual tensile stress from this measurement is 0.218 ± 0.007 GPa, which is similar to the previous result in [80] on a similar thin film. These detected frequencies are in the range expected if the thin film was acting in the membrane regime, which is described by equations 2.40 and 2.46, in agreement with earlier measurements.

According to previous research [80, 98, 120], mode splitting, where modes such as 1:2 and 2:1 occur at different frequencies, or mixing, where modes 1:2 & 2:1 occur at the same frequency, can be caused by several different conditions. These include, in order of importance; different (or equal) lateral dimensions, boundary conditions, or anisotropy (isotropy) in the mechanical properties. There can also be some influence from the mounting condition of sample on transducer and environmental factors such as air damping. Indeed, a real thin film will not, in practice, be perfectly described as a ideal membrane by the idealized equations, and mode separation or mixing will
<table>
<thead>
<tr>
<th>Modes</th>
<th>Predicted rf (kHz)</th>
<th>Measured rf (kHz)</th>
<th>Measured Stress (GPa)</th>
<th>Γ</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>159.227</td>
<td>164</td>
<td>0.233</td>
<td>0.35</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>250.885</td>
<td>248</td>
<td>0.215</td>
<td>0.37</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>252.631</td>
<td>256</td>
<td>0.226</td>
<td>0.37</td>
</tr>
<tr>
<td>2:2 mode</td>
<td>318.453</td>
<td>325</td>
<td>0.229</td>
<td></td>
</tr>
<tr>
<td>1:3 mode</td>
<td>354.391</td>
<td>350</td>
<td>0.215</td>
<td>0.36</td>
</tr>
<tr>
<td>3:1 mode</td>
<td>357.684</td>
<td>355</td>
<td>0.217</td>
<td>0.36</td>
</tr>
<tr>
<td>1:4 mode</td>
<td>461.847</td>
<td>458</td>
<td>0.202</td>
<td>0.36</td>
</tr>
<tr>
<td>4:1 mode</td>
<td>466.583</td>
<td>468</td>
<td>0.206</td>
<td>0.36</td>
</tr>
</tbody>
</table>

Table 4.1: Predicted resonant frequencies of selected vibrational modes for a predicted stress of 0.22 GPa, and the measured frequencies at atmospheric pressure. The calculated residual stress for the Ge thin film of 700 nm thickness and 860 × 865 µm dimensions are also given for each measured mode (rf is Resonant Frequency).

not be influenced by a single factor. The predicted resonant frequencies shown in table 4.1 consider only the differences in lateral dimension when calculating mode splitting, without including the effect of errors in measuring the dimensions. The frequency difference predicted between the 1:2 and 2:1 modes, and the 1:3 and 3:1 modes, are about 1.75 kHz and 3.29 kHz respectively, for the approximately 5 µm difference in lateral dimension of each side of the thin film. This is the same order as the potential splitting observed in figures 4.2 and 4.3, lending weight to the suggestion that mode splitting is observed, in this case dominated by variation in the lateral dimensions.

Greater clarity could be obtained by measuring at a reduced pressure to enhance the resonance Q-factors. These measurements were therefore repeated at a pressure of 10⁻³ mbar, using a smaller voltage of 100 mV to drive the transducer. The full frequency sweep from 40 kHz to 600 kHz is shown in figure 4.4. Because the air damping effect is reduced, the resonant frequency peaks become more sharp with a smaller FWHM. The peak splitting becomes more distinct, with smaller frequency step size scans shown in figure 4.5. In this figure, the frequency range was zoomed into a similar region to the potential mode splitting found in figure 4.3. The predicted resonant frequencies based on membrane behaviour at low pressure and measured values are presented in table 4.2. An average residual stress of 0.215 ± 0.006 GPa was found.

Figures 4.5 (b) and (c) show a much clearer peak splitting with peak fitting performed for the frequencies around modes 1:3 and 3:1, and modes 1:4 and 4:1. In part (a), there is only a single peak clearly observed, however the peak fitting pro-
Figure 4.4: The response of the Ge thin film with 700 nm thickness and 860 × 865 µm lateral dimensions to excitation at different frequencies at 10⁻³ mbar pressure, measured at a position around the centre of the thin film and a random position on the substrate.

Figure 4.5: The amplitude response with a smaller frequency scan step size of the Ge thin film with 700 nm thickness and 860 × 865 µm lateral dimensions to excitation at different frequencies at 10⁻³ mbar pressure, measured at a position around the centre of the thin film.
procedure with a single peak does not give a suitable fit. The difference between the fitted and measured data could indicate a second smaller resonance. The FWHM measured for this mode is 1.368 kHz, and the shoulder seen on the right hand side of the resonance could be due to a second resonance with a peak splitting close to that predicted in table 4.1. Note that, as shown in figure 4.2 (b) and (c), the central position on the membrane is close to a node for this resonance, and it is therefore possible that the frequency scan has not detected the full resonance curve. The best way to prove this assumption is using the 2D vibrational pattern image to identify the resonant modes in this frequency range. Unfortunately, the 2D vibration pattern was hard to obtain at this low pressure (10$^{-3}$ mbar) for this sample because of the heating effect from the laser detector (more details in Chapter 6). However, the FWHM values found in the data shown in figure 4.4 (and summarised in table 4.2) can be used as indirect evidence to suggest there is mode splitting for modes 1:2 and 2:1 in the frequency range of 250 kHz to 280 kHz at low pressure.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Predicted rf (kHz)</th>
<th>Measured rf (kHz)</th>
<th>Stress (GPa)</th>
<th>FWHM (kHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>166.197</td>
<td>164.75</td>
<td>0.233</td>
<td>1.142</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>261.868</td>
<td>260.76</td>
<td>0.215</td>
<td>1.368</td>
</tr>
<tr>
<td>1:3 mode</td>
<td>369.906</td>
<td>362.52</td>
<td>0.215</td>
<td>1.315</td>
</tr>
<tr>
<td>3:1 mode</td>
<td>373.343</td>
<td>369.25</td>
<td>0.217</td>
<td>0.758</td>
</tr>
<tr>
<td>1:4 mode</td>
<td>482.066</td>
<td>473.51</td>
<td>0.202</td>
<td>3.241</td>
</tr>
<tr>
<td>4:1 mode</td>
<td>487.009</td>
<td>479.56</td>
<td>0.206</td>
<td>1.494</td>
</tr>
</tbody>
</table>

Table 4.2: Predicted resonant frequencies of selected vibrational modes for a predicted stress of 0.22 GPa, and the measured frequencies at low pressure. The calculated residual stress for the Ge thin film of 700 nm thickness and 860 × 865 µm dimensions are given for each mode.

4.2 Ge Thin Film with 4 µm Thickness, 2850 × 2855 µm Dimensions

In the first section of this chapter, the air damping at atmospheric pressure and mode splitting was discussed. In order to understand more the influence caused by the air damping and highlight any potential reasons for mode splitting other than lateral dimensions, a second single crystalline Ge suspended thin film, which was grown under the same conditions as the first Ge thin film sample presented in section 4.1, but with 4 µm thickness and 2850 × 2855 µm lateral dimensions, was studied. The same measurements were done at a light vacuum (medium pressure of around 1 mbar), and at a medium vacuum (low pressure of around 10$^{-3}$ mbar).
In the size comparison between this Ge sample and the first thin film studied, the ratio of the difference of the side lengths is 0.175% and 0.580%, respectively. Thus, the second Ge thin film was closer to a square shape compared to the first sample, which means mode mixing or indistinguishable spitting is predicted, with any larger splitting predominantly caused by the boundary conditions or mechanical properties of the thin film such as anisotropy.

Following the procedures of thin film property characterisation outlined previously, the resonant frequency behaviour of the thin film in a light vacuum and in a medium vacuum was measured over a wide frequency range, from 40 kHz to 200 kHz. Some modes are clearly detected, as shown in figure 4.6. The scanning frequency range was selected based on the predicted resonant frequencies for membrane-like behaviour using the residual stress found in section 4.1 as an initial assumption. The predicted and measured resonant frequencies at both pressures are given in table 4.3. This table also contains the calculated stress using equation 2.40 from each of the identified resonant frequencies in the peak spectra and the calculated Q-factors for the modes.
with the measured FWHM, at the two different pressures. The values in table 4.3a are obtained from the light vacuum (mbar) measurement and table 4.3b contains the values measured in the medium vacuum ($10^{-3}$ mbar). The average residual stresses from these two measurements are 0.236 ± 0.008 GPa and 0.220 ± 0.007 GPa, respectively.

(a) Light vacuum with a millibar pressure.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Predicted rf (kHz)</th>
<th>Measured rf (kHz)</th>
<th>FWHM (kHz)</th>
<th>Q-factor</th>
<th>Stress (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>50.09</td>
<td>50.74</td>
<td>1.744</td>
<td>29.090</td>
<td>0.235</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>78.87</td>
<td>77.80</td>
<td>-</td>
<td>-</td>
<td>0.223</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>79.53</td>
<td>85.12</td>
<td>1.972</td>
<td>43.157</td>
<td>0.264</td>
</tr>
<tr>
<td>1:3 mode</td>
<td>111.38</td>
<td>103.10</td>
<td>1.594</td>
<td>64.682</td>
<td>0.195</td>
</tr>
<tr>
<td>3:1 mode</td>
<td>112.63</td>
<td>110.65</td>
<td>0.992</td>
<td>111.579</td>
<td>0.223</td>
</tr>
<tr>
<td>1:5 mode</td>
<td>179.44</td>
<td>185.99</td>
<td>1.078</td>
<td>172.526</td>
<td>0.245</td>
</tr>
<tr>
<td>5:1 mode</td>
<td>181.77</td>
<td>196.45</td>
<td>0.735</td>
<td>267.354</td>
<td>0.270</td>
</tr>
</tbody>
</table>

(b) Medium vacuum with a $10^{-3}$ mbar pressure.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Predicted rf (kHz)</th>
<th>Measured rf (kHz)</th>
<th>FWHM (kHz)</th>
<th>Q-factor</th>
<th>Stress (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>50.31</td>
<td>50.69</td>
<td>0.079</td>
<td>640.635</td>
<td>0.222</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>79.42</td>
<td>79.39</td>
<td>0.058</td>
<td>1362.241</td>
<td>0.218</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>79.67</td>
<td>86.31</td>
<td>0.072</td>
<td>1194.053</td>
<td>0.258</td>
</tr>
<tr>
<td>1:3 mode</td>
<td>112.26</td>
<td>104.11</td>
<td>0.093</td>
<td>1121.834</td>
<td>0.187</td>
</tr>
<tr>
<td>3:1 mode</td>
<td>112.73</td>
<td>112.05</td>
<td>0.145</td>
<td>770.410</td>
<td>0.217</td>
</tr>
</tbody>
</table>

Table 4.3: Predicted resonant frequencies of selected vibrational modes for a predicted stress of 0.218 GPa, and the measured frequencies with the FWHM at different low pressure. The calculated residual stress and Q-factor for the Ge thin film of 4 µm thickness and 2850 × 2855 µm lateral dimensions are given for each mode.

Figure 4.6 shows the peak spectra measured at a position around the centre of the thin film. The stack effect of the actuation on the sample substrate is not shown here. At the lower pressures when the air is partially removed, the damping effect is reduced significantly. Therefore the Q-factors of the resonances increase considerably as expected, as demonstrated by the sharp peaks with narrower FWHM. This means that resonances are clearer, as is any potential mode splitting.

At the same time as the Q-factor increases, the resonant frequencies also would have an upward shift in frequency because of the pressure dependence discussed in chapter 2. In figure 4.6, the resonances show a general shift to higher frequencies as the pressure is reduced. The increase shown in table 1.3 is smaller than could be expected, indicating the influence of another effect on the resonant frequency.
More details about this behaviour at low pressure will be discussed in chapter 6, considering the heating effect from the probe laser which can become significant at a lower pressure, as mentioned in section 4.1.

The 2D-scan mapping images which correspond to these resonant modes are shown in figure 4.7 (d) to (l), in the light vacuum with millibar pressure. Figures 4.7 (a) to (c) show the frequency scans zoomed into the region of several identified modes with splitting. Each peak has been fit using a Lorentzian fitting to yield the FWHM and ascertain the Q-factor at exact frequencies of the mixed modes, and identify any split modes. This fitting does offer some evidence of mode splitting for higher order modes.

The 2D-scan maps of the resonant modes in figure 4.7 show the mode splitting along with some partial mixing, where mixed modes are observed at two slightly different frequencies, but with a different vibrational strength at each frequency. For example, figure 4.7 presents the 2D-scan image of the mixed mode of 1:2 - 2:1 in part (e) with a maximum displacement of 0.077 nm, and 2:1 + 1:2 mode in part (f) with a maximum displacement of 0.582 nm at the millibar pressure. The displacement amplitude of the frequency response in figure 4.7 (a) shows a potential overlap between two resonant frequencies, as shown by the fitting lines. Thus, the mixed mode is caused by a superposition of the classical theory predicted for the mixed modes, similar to the earlier study in section 4.1 on the first Ge thin film sample (figure 4.3 and 4.5 respectively). The maximum displacement in the 2D mode images shows which of the mixing modes is intensified (+) or is subdued (−).

At a lower pressure, the vibrational displacement will become larger under the same excitation force with the damping reduced, as displayed by the differences between the amplitudes for the frequency scans at these pressures in figure 4.6. The change in the maximum displacement measured for the fundamental mode as the pressure is reduced from millibar to microbar is more than 10 times. These larger amplitude vibrations can lead to non-linear behaviour, which is discussed in detail in chapter 5 and can lead to the alteration of the resonant mode shapes, as shown in the 2D-scans at 10^{-3} mbar presented in figure 4.8. To observe more details of the resonant mode pattern from the 2D scanning measurement, the displacement scale was zoomed in, giving the images in figure 4.8 (c) to (h). The area of the thin film with a larger vibration displacement shows as a white colour in these mode mapping images. The peak spectra shown in figure 4.8 (a) are the resonant peaks for the mixed mode of 1:2 − 2:1 (2D scan image in part (d)) and 2:1 + 1:2 mode (2D-scan image in part (e)) found in the microbar pressure measurement, which are now clearly split.
Figure 4.7: The Ge thin film with 4 µm thickness and 2414 µm × 2520 µm lateral dimensions at millibar pressure. (a) to (c) are the frequency scans zoomed in on the region of mode splitting. (d) Fundamental mode at 50.74 kHz, (e) 1:2 + 2:1 at 77.80 kHz, (f) 2:1 − 1:2 at 85.12 kHz. (g) and (h) are an unknown mode pattern which might be a mixture between the 2:2 mode and 1:3 mode or 3:1 mode at 103.10 kHz and 110.65 kHz. (i) 5:1 − 1:5 at 185.99 kHz, (j) 5:1 + 1:5 at 196.45 kHz, (k) 5:2 at 201.12 kHz, (l) 4:4 at 210.23 kHz.
Figure 4.8: The Ge thin film with 4 \( \mu m \) thickness and 2414 \( \mu m \times 2520 \mu m \) lateral dimensions at microbar pressure. (a) to (b) are the frequency scans zoomed in on the region of mode splitting. (c) Fundamental at 49.57 kHz, (d) 1:2 + 2:1 at 78.73 kHz, (e) 1:2 − 2:1 at 85.64 kHz. (f) and (g) are an unknown mode pattern which might be a mixture between the 2:2 and 1:3 or 3:1 at 104.11 kHz and 112.05 kHz. (h) 3:1 − 1:3 at 119.88 kHz.
without overlapping. The frequency difference between the two peaks is 6.91 kHz and the FWHM of the peaks are 0.058 kHz and 0.072 kHz (table 4.3b). The mode patterns show these to both be mixed mode, rather than a splitting of the pure 1:2 and 2:1 mode; Table 4.3b shows the predicted frequency difference of the splitting which would be caused by the difference of lateral dimensions, which is smaller than 1 kHz. The measured behaviour is contrary to what would be expected from the theory and the earlier measurement results. Similar behaviour is observed in the unknown resonant modes in figure 4.7 (g), (h) and figure 4.8 (f), (g), and the frequency scans in part (b) of figures 4.7 and 4.8 with a splitting observed between mode mixed, rather than pure modes.

The unknown resonant modes are found at 103.10 kHz and 110.65 kHz at millibar pressure and 104.11 kHz and 112.05 kHz at microbar pressure. The measured frequency of the undefined modes are close to the predicted resonant frequency range for mode 1:3 or 3:1 (table 4.3), but these measured frequencies are also very close to mode 2:2, which should be observed around 95.60 kHz at millibar pressure or 98.84 kHz at microbar pressure based on the theoretical prediction. It is, therefore, possible that these are a mode mixture between 2:2 and 1:3 or 3:1. This type of mode has not been considered in our modelling. Although the 2:2 mode resonant frequency was not found from the frequency sweep, this is expected as the measurement was made with the laser probe at the centre position of the thin film, which is the node position of the 2:2 mode pattern. From the medium vacuum (10−3 mbar) measurement, the typical mixed behaviour for the 1:3 − 3:1 mode was found and shown in figure 4.8(h). This result confirmed the prediction that the resonances of 1:3 or 3:1 modes are around the frequency region of 110 ± 10 kHz.

There is still potential for debate regarding the mode assignment at 110 ± 10 kHz. There is a large frequency difference between the three modes at of 104.11 kHz, 112.05 kHz and 119.88 kHz, and disagreement with the theoretical prediction. Looking more carefully at the shapes in figures 4.8 (f) and (g), the mode shape in part (f) looks more like a 2:2 mode, whereas (g) looks more close to a 1:3 mode, with each of these frequencies close to its theoretical calculation. Also, there is a 6.91 kHz frequency difference for the mode mixing between 1:2 and 2:1, with no observable overlapping between peaks.

These unexpected behaviours, such as the unknown resonant modes or the mode mixing behaviour without the superposition of resonant peaks, cannot be described using the simple model and classical theory. Mechanical anisotropy could explain the splitting. The elastic constants are orientation dependent for a crystalline cubic lattice structure such as these Ge thin film samples, which would lead to mode
splitting rather than mixing. The splitting with modes mixing might be affected by the mechanical property changes due to the high number of non-uniform defects at the interface between the thin film and substrate. This can be tested by measuring a thin film with a different level of defects [21].

4.3 Two 3C-SiC Thin Films with 600 nm Thickness, 1654 × 1726 µm and 1902 × 1923 µm Dimensions

3C-SiC has a high melting point and the suspended thin films produced are translucent at optical wavelengths. The compound of 3C-SiC is formed from group IV elements and offers a good contrast with the non-transparent Ge thin film which use an element from the same group. The lattice difference between 3C-SiC and Si is larger than that between Ge and Si, thus the 3C-SiC thin film on Si substrate contains lots of defects generated at the interface layer, which means the 3C-SiC thin film can have a wide range of Young’s modulus or residual stress, dependent on the density of defects contained in the thin film [21]. In addition, these 3C-SiC thin film materials were grown using a new growth process [21, 97], whose properties have not yet been fully characterised. Only the strain relaxation has thus far been investigated through the use of micro X-ray diffraction (µ-XRD) [121, 122]. The aim of this section of the chapter was to measure the residual stress of the suspended 3C-SiC thin films using the same procedure which has been previously applied to crystalline Ge thin films in sections 4.1 and 4.2. More material property details of 3C-SiC are described in chapter 1.

A comparison measurement was done on two 600 nm thickness 3C-SiC suspended structure thin films with different lateral dimensions. One was a rectangle rather than a square, with side lengths of 1654 µm and 1726 µm (a difference of 4.353%), while the second 3C-SiC thin film sample was close to a square with side lengths of 1902 µm and 1923 µm (the difference is 1.052%, which was similar to the error of the optical microscope which is used to measure the lateral dimensions). Both thin film samples should have similar mechanical properties because they were made on the same wafer and hence have identical growth conditions.

Figure 4.9 shows the frequency peak spectra obtained from the two 600 nm thickness 3C-SiC thin films. Part (a) was measured on the rectangular thin film for the detection laser at the position around the centre of the thin film, at atmospheric pressure (RP) and microbar pressure (LP), and a random position on the sample substrate at RP, in the frequency sweeping range from 50 kHz to 450 kHz. Part (b) was achieved in the frequency sweeping range between 50 kHz to 600 kHz on the
Figure 4.9: The frequency spectra of the 3C-SiC thin film with 600 nm thickness. (a) is from the rectangular thin film with 1654 $\times$ 1726 $\mu$m lateral dimensions at atmospheric pressure (RP) and $10^{-3}$ mbar pressure (LP), measured in a position around the centre of the thin film and a random position on the substrate. (b) is from the square thin film with 1902 $\times$ 1923 $\mu$m lateral dimensions at $10^{-3}$ mbar pressure, measured at three different positions on the thin film.
square thin film at microbar pressure, measured at three different positions: the first frequency scan was done at a position around the thin film centre, the second position was selected at the quarter of a middle line, and the third position was at the quarter position of the diagonal, as shown in the schematic.

Figure 4.10: 2D-scan images of resonant modes for 3C-SiC 600 nm thickness with 1654 × 1726 µm lateral dimensions at atmospheric pressure. (a) is the 1:2 mode at 197.2 kHz. (b) is the 3:1 mode at 234.2 kHz.

As with the previous study on Ge thin films, the peaks in the substrate response indicate the transducer resonances. Measuring the response at the different positions allows one to identify more resonant modes because the modes have different maximum deflection and node lines at different positions. In this way, modes such as 2:2 should be visible on certain frequency scans.

The measurement done at RP gave a messy peak spectrum (black colour in figure 4.9 (a)), caused by the air damping and a very strong stack effect of the actuation from the transducer. Because of the uncertainties for 3C-SiC properties, it is harder to predict the resonant frequencies. However, pressure reduction will still give a clearer indication of mode. Although the low pressure would intensify the heating effect from the probe laser, a clear spectrum (red colour) shows the resonant peaks more clearly.

By using 2D scanning at each of the resonant frequencies, some of the 2D pattern images of the resonant modes were found. Figure 4.10 shows a 1:2 mode at 197.2 kHz in part (a) and 3:1 mode 234.2 kHz in part (b) at atmospheric pressure as an example. Figures 4.11 (b) to (d) show the fundamental mode (1:1) at 126.38 kHz, 1:3 mode at 284 kHz and 3:1 mode at 294.2 kHz at microbar pressure, both for the rectangular thin film. The average residual stress calculated from these resonances is 0.285 ± 0.008 GPa. The average tensile strain from the µ-XRD measurement is about 0.099 ± 0.03 %. A rough approximation of the average Youngs modulus from the resonance measurements is 287.88 ± 8.73 GPa. The most common Youngs moduli measured for 3C-SiC are in a range from 100 to 500 GPa, giving residual
Figure 4.11: Mode splitting peak spectra and the 2D-scan images of resonant mode for 3C-SiC 600 nm thickness with 1654 × 1726 µm lateral dimensions at microbar pressure. (a) is the resonance peaks for mode 1:3 and 3:1. (b) is the fundamental mode at 126.38 kHz. (c) is the 1:3 mode at 284 kHz. (d) is the 3:1 mode at 294.2 kHz.

Stresses ranging from 0.098 to 0.486 GPa \cite{123, 124, 125, 126}, and are strongly dependent on the deposition conditions. The experimental result is in the value range from the literature \cite{123, 127}, and the residual stress agrees with the strain measurements reported in the literatures \cite{121, 123, 124, 125, 126, 127}.

In microbar pressure, the frequency sweep was done on the square 3C-SiC thin film at different positions, showing a different vibration strength for each resonant frequency in figure 4.9 (b). The vibration strength observed is related to the detection position, and whether it is placed close to the node line or antinode line of the resonant modes. In figure 4.12, the peak spectra are shown zoomed into the frequency ranges of 130 kHz to 170 kHz and 170 kHz to 230 kHz. The frequency scan step size was 0.1 kHz, larger than in other experiments. The predicted resonant frequencies shown in table 4.4 which used the residual stress found from the earlier rectangular sample measurement, suggests these peaks are 1:2 or 2:1 modes and 1:3 or 3:1 modes respectively. The smaller peak around a frequency of 185 kHz might be the resonant mode 2:2 because it will only be clearly observed when the detector is placed on the position of the quarter of the diagonal.

The resonant modes images in the zoomed in frequency ranges were identified by using 2D scanning. Two sets of mixed modes with splitting are illustrated in fig-
Figure 4.12: The peak spectra, with a 1 kHz scanning step size, in a zoomed in frequency range from 130 kHz to 230 kHz shown in two separate sections for 3C-SiC of 600 nm thickness with 1902 $\times$ 1923 $\mu$m lateral dimensions at microbar pressure.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Predicted rf (kHz)</th>
<th>Measured rf (kHz)</th>
<th>Q-factor (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>110.17</td>
<td>91.75</td>
<td>0.193</td>
</tr>
<tr>
<td>1:2 + 2:1 mode</td>
<td>173.62</td>
<td>145.33</td>
<td>0.192</td>
</tr>
<tr>
<td>2:1 − 1:2 mode</td>
<td>174.77</td>
<td>147.10</td>
<td>0.198</td>
</tr>
<tr>
<td>1:3 ± 3:1 mode</td>
<td>245.27</td>
<td>205.05</td>
<td>0.193</td>
</tr>
<tr>
<td>3:1 − 1:3 mode</td>
<td>247.43</td>
<td>207.14</td>
<td>0.197</td>
</tr>
<tr>
<td>2:2 mode</td>
<td>220.34</td>
<td>185.49</td>
<td>0.197</td>
</tr>
</tbody>
</table>

Table 4.4: Predicted resonant frequencies of selected vibrational modes for a predicted stress of 0.285 GPa, and the measured frequencies at microbar pressure. The calculated residual stress for the 3C-SiC thin film of 600 nm thickness and 1902 $\times$ 1923 $\mu$m dimensions are given for each mode.

Figure 4.13: Parts (c) and (d) show the mode splitting with a partial mixing between 1:2 and 2:1, with 1:2 + 2:1 at 144.94 kHz and 2:1 − 1:2 at 147.10 kHz. Note that the displacement at 144.94 kHz is large, and the thin film behaviour is affected, as discussed in chapters 5 and 6. However, the smaller displacement observed at 147.10 kHz gives a measurement closer to the classical, linear prediction. Parts (e) and (f) show similar behaviour for the 1:3 modes, at 205.05 kHz with the plus effect and at 207.14 kHz with the minus effect between 1:3 and 3:1 modes.

The average residual stress on the second 3C-SiC thin film is about 0.195 $\pm$ 0.004 GPa, which is smaller than the residual stress (about 0.285 $\pm$ 0.008 GPa) which was found from the first 3C-SiC thin film sample. Because both of the samples were fabricated from the same wafer, it could be assumed that they would have the same residual stress. However, the difference in residual stress between the two samples is about
Figure 4.13: The mode splitting peak spectra and the 2D-scan images of resonant mode for 3C-SiC 600 nm thickness with 1902 × 1923 µm lateral dimensions at microbar pressure. (a) and (b) are the resonance peaks for mode splitting. (c) to (f) are the 2D mode pattern images at 144.94 kHz, 147.10 kHz, 205.05 kHz, and 207.14 kHz for resonant modes 1:2 + 2:1, 2:1 − 1:2, 1:3 + 3:1 and 3:1 − 1:3.

37.5%, which could be due to the difference in the density of stacking faults from the thin film growth on different locations on the wafer [21].

To compare the mode splitting between these two 3C-SiC samples; the first sample with a rectangular shape found a pure mode splitting between the 1:3 mode and 3:1 mode, with the resonant peaks having a frequency difference of 10.2 kHz and without peak overlapping (figure 4.11) which is slightly larger than the value of 9.5 kHz from the theoretical calculation (1:3 mode at 275.79 kHz and 3:1 mode at 285.30 kHz). For the square sample, the mode splitting instead showed a partial mode mixing (figure 4.13) at different frequencies, rather than a splitting of pure modes. This again suggests that geometrical differences are dominating, but that the stacking faults have an effect on the overall stress.
Figure 4.14: The frequency spectra of the multilayer thin film in frequency range from 30 kHz to 230 kHz at microbar pressure, measured at the position close to the thin film center.
4.4 Multilayer Structure of 3C-SiC on Si$_{1-y}$C$_y$/Si$_{1-x}$B$_x$

Thin Film with Total Thickness of 570 nm and 2267 $\times$ 2564 $\mu$m Dimensions

To reduce the formation of defects in the 3C-SiC epilayer, around a 50 nm thickness Si$_{1-x}$B$_x$ and around 20 nm of Si$_{1-y}$C$_y$ ($x < 0.01$, $y < 0.015$) were grown on the Si substrate as epilayers in this order, before the 3C-SiC was grown upon the epi-wafer. In this case, the 3C-SiC film was approximately 500 nm thick. The total thickness of this multilayer thin film is therefore about 570 nm, with a rectangular shape with lateral dimensions 2267 $\mu$m $\times$ 2564 $\mu$m.

Figure 4.15: The mode splitting peak spectra and the 2D-scan images of resonant modes for the multilayer thin film at microbar pressure. (a) and (b) are the resonance peaks for mode splitting. (c) Fundamental mode at 47.5 kHz, (d) 1:2 at 72 kHz, (e) 2:1 at 77.7 kHz, (f) 1:3 at 100.4 kHz, (g) 3:1 at 111.2 kHz and (h) 2:3 at 117.3 kHz.

The frequency sweep measurement was done in the frequency range from 30 kHz to 230 kHz at microbar pressure. The measurement position was close to the center.
of the thin film. There are 21 peaks observed from this measurement, as shown in figure [4.14]. Only the first six resonant peaks were investigated using the 2D scanning procedure as higher modes would not give a clear image with the 200 µm laser spot size. The mode pattern images measured for the six resonances are plotted in figures [4.15] (c) to (h). There are two sets of resonances showing mode splitting, classified as the 1:2 mode at 72 kHz and 2:1 mode at 77.7 kHz, and the 1:3 mode at 100.4 kHz and 3:1 mode at 111.2 kHz. This splitting is expected from the rectangular shape of the thin film. The measurement results and calculated residual stress are given in table [4.5]. Note that, the mode shapes are also affected by the laser heating for the low pressure measurement (discussed in detail in chapter 6).

To calculate the residual stress using the single layer membrane equation 2.40 (from chapter 2), the multilayer thin film density was approximated as 3025 kg/m³. The pure 3C-SiC density is 3166 kg/m³ (at 293 K). The densities of the Si_{1-x}B_x and Si_{1-y}C_y epilayers were considered as the density of pure Si, as the values of x and y are smaller than 1%. The percentage of the total thickness comprised of the Si_{1-x}B_x and Si_{1-y}C_y epilayers is about one sixth. By using the approximate thin film density and these identified resonant frequencies, the average residual stress of the multilayer thin film is 72.41 ± 7.24 MPa. This residual stress is significantly smaller than the results found in section [4.3] for single layer structure 3C-SiC, which might provide evidence that this novel preparation route can reduce the number of defects in heteroepitaxially crystal growth and hence reduce the residual stress. This agrees with the assumption made in [21] when using this new growth method.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Measured rf (kHz)</th>
<th>Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>47.5</td>
<td>73.42</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>72</td>
<td>67.48</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>77.7</td>
<td>78.59</td>
</tr>
<tr>
<td>1:3 mode</td>
<td>100.4</td>
<td>65.61</td>
</tr>
<tr>
<td>3:1 mode</td>
<td>111.2</td>
<td>80.48</td>
</tr>
<tr>
<td>2:3 mode</td>
<td>117.3</td>
<td>68.89</td>
</tr>
</tbody>
</table>

Table 4.5: The calculated residual stress from each measured resonant mode frequency for the multilayer thin film of 570 nm thickness and 2267 × 2564 µm lateral dimensions.
4.5 Two Square Si$_3$N$_4$ Thin Films with 310 µm and 315 µm Dimensions and 300 nm Thickness

This section presents measurement results on two Si$_3$N$_4$ thin film samples in the linear regime, which were provided by the research group of AG Scheer from Konstanz University [100]. These two thin film samples were fabricated from the same wafer and each has a square shape, with side lengths of about 310 µm (sample SiN$_1$) or 315 µm (sample SiN$_2$) respectively, and with 300 nm thickness. Note that these thin films are significantly smaller than earlier samples and are also used for the non-linear vibration study presented in chapter 5.

There are two different spot sizes available for the probe laser, which are 200 µm and 30 µm in diameter. Whilst the large size was used for the earlier tests, both were used to investigate the Si$_3$N$_4$ thin films. The probe laser spot size of 200 µm in diameter covers most of the area of these thin films, leading to a different type of measurement looking at average displacement, so the smaller laser spot was also applied and measurements repeated. The resonant mode images from the 2D-scans were produced using the smaller spot size to reduce the spatial averaging effect in the interferometer signal processing. All these measurements were done at microbar pressure.

The frequency sweep result for the Si$_3$N$_4$ thin film with 315 µm lateral dimensions is shown in figure 4.16 and the 2D modes pattern images for each mechanical resonance found from the peak spectra are presented in figure 4.17. The fundamental resonant frequency was found to be 1362.05 kHz (2D image in figure 4.17 (a)), with the significantly higher value due to the small size of the thin film. A mode splitting of modes 1:2 (figure 4.17 (b)) and 2:1 (figure 4.17 (c)) was identified at 2148.12 kHz and 2157.28 kHz. The average residual stress is found to be $1.166 \pm 0.015$ GPa from these measurements. This error is caused by the lateral dimension error of ±10 µm and the resonance shift due to laser heating (see chapter 6). Also, only three resonance values were used to estimate the average residual stress due to the high frequencies required, and hence the accuracy is likely to be lower than earlier. Each of the calculated residual stresses and Q-factors from the resonant frequencies are presented in table 4.6.

The measurement results of peak spectra and 2D mode images for the second Si$_3$N$_4$ thin film with 310 µm lateral dimensions are shown in figures 4.18 and 4.19. This thin film was characterised by using the same procedure as previously, under the same experimental conditions, with the small laser spot size. There are five mechanical resonances which are found in the frequency range from 1 to 3 MHz. The identified
Figure 4.16: The frequency spectra measured at the position close to the thin film center for the Si$_3$N$_4$ sample with 315 µm lateral dimensions.

Figure 4.17: 2D scan images of resonant modes for the Si$_3$N$_4$ sample with 315 µm lateral dimensions.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Measured rf (kHz)</th>
<th>Q-factor</th>
<th>Stress (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>1362.12</td>
<td>13250.17</td>
<td>1.167</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>2148.12</td>
<td>9269.51</td>
<td>1.161</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>2157.28</td>
<td>13430.92</td>
<td>1.171</td>
</tr>
</tbody>
</table>

Table 4.6: Experiment results for the Si$_3$N$_4$ sample with 315 µm lateral dimensions.
modes included the fundamental 1:1 mode, 1:2 mode, 2:1 mode, 2:2 mode, and 1:3 mode, with the 2:2 mode detected due to experimental difficulties in locating the exact centre of these small thin films. The average residual stress was about 0.953 ± 0.012 GPa. Each of the calculated residual stresses and Q-factors from the resonant frequencies are presented in table 4.7.

![Frequency spectra](image)

Figure 4.18: The frequency spectra measured in the position close to the thin film center for the Si$_3$N$_4$ sample with 310 µm lateral dimensions.

<table>
<thead>
<tr>
<th>Modes</th>
<th>Measured rf (kHz)</th>
<th>Q-factor</th>
<th>Stress (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:1 mode</td>
<td>1256.72</td>
<td>12161.45</td>
<td>0.962</td>
</tr>
<tr>
<td>1:2 mode</td>
<td>1968.61</td>
<td>17010.38</td>
<td>0.944</td>
</tr>
<tr>
<td>2:1 mode</td>
<td>1990.05</td>
<td>12197.68</td>
<td>0.965</td>
</tr>
<tr>
<td>2:2 mode</td>
<td>2493.08</td>
<td>17685.18</td>
<td>0.947</td>
</tr>
<tr>
<td>1:3 mode</td>
<td>2788.09</td>
<td>10211.66</td>
<td>0.947</td>
</tr>
</tbody>
</table>

Table 4.7: Experiment results for the Si$_3$N$_4$ sample with 310 µm lateral dimensions.

The fundamental mode (shown in figure 4.19 (a)) which was found in the second Si$_3$N$_4$ thin film demonstrated a strange shape where much of the displacement appeared to be in one corner. The modes at 1:2 and 2:1 (shown in figure 4.19 (b) and (c)) do not give a pure mode splitting, but also exhibited a partial mixture in the splitting. In addition, the 2D-scan images for higher order modes were unclear.
Figure 4.19: The 2D-scan images of resonant modes for the Si$_3$N$_4$ sample with 310 $\mu$m lateral dimensions.

Figure 4.20: The samples images taken by an optical microscope. (a) is the first thin film with 315 $\mu$m side length, (b) is the second thin film with 310 $\mu$m side length.
The sample images of these two thin films taken using an optical microscope are shown in figure 4.20. Sample 2 is clearly dirty, with a section of silver glue with a circular shape which is located at a corner near the frame boundary (figure 4.20 (b)). This unclean condition is very likely to affect the mechanical properties of this thin film, which would give a difference in the average residual stress found for these two samples and would affect the 2D-scan images, giving the results in figure 4.19.

Further Si$_3$N$_4$ thin films were made from the same wafer on a close location to that for these two Si$_3$N$_4$ samples. They were also characterised by the research group of AG Scheer at Konstanz University using an imaging white light interferometry with different light sources. The average residual stress for the Si$_3$N$_4$ thin film found using these measurement was 1.097 ± 0.011 GPa, which is in good agreement with the value found from the first clean sample (Data provied by Fan Yang in research group of AG Scheer at 2018).

4.6 Summary

Three different materials and two different structures of suspended thin films were studied in this chapter. The sample sizes and behaviour are summarised in table 4.8. All these thin films behaved as membranes rather than plates confirmed by the resonant frequencies and the residual stress values being close to the predicted frequency values using equation 2.40 for membranes.

<table>
<thead>
<tr>
<th>Material</th>
<th>Structure</th>
<th>Lateral ($\mu\text{m} \times \mu\text{m}$)</th>
<th>Thickness (nm)</th>
<th>Mode</th>
<th>Residual Stress (MPa)</th>
<th>Behaviour</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge</td>
<td>Single</td>
<td>860×865</td>
<td>700</td>
<td>Mixing</td>
<td>215±6</td>
<td>Membrane</td>
</tr>
<tr>
<td>Ge</td>
<td>Single</td>
<td>2850×2855</td>
<td>4000</td>
<td>Mixing</td>
<td>220±7</td>
<td>Membrane</td>
</tr>
<tr>
<td>SiC</td>
<td>Single</td>
<td>1654×1726</td>
<td>600</td>
<td>Splitting</td>
<td>285±8</td>
<td>Membrane</td>
</tr>
<tr>
<td>SiC</td>
<td>Single</td>
<td>1902×1923</td>
<td>600</td>
<td>Mixing</td>
<td>195±4</td>
<td>Membrane</td>
</tr>
<tr>
<td>SiN</td>
<td>Single</td>
<td>310×310</td>
<td>300</td>
<td>Mixing</td>
<td>953±12</td>
<td>Membrane</td>
</tr>
<tr>
<td>SiN</td>
<td>Single</td>
<td>315×315</td>
<td>300</td>
<td>Splitting</td>
<td>1166±15</td>
<td>Membrane</td>
</tr>
<tr>
<td>SiC</td>
<td>Multilayer</td>
<td>2267×2564</td>
<td>570</td>
<td>Splitting</td>
<td>72±7</td>
<td>Membrane</td>
</tr>
</tbody>
</table>

Table 4.8: Summary of thin film properties.

The summarised data proves that the mode splitting can be influenced by several different conditions. The first condition is the material property such as isotropy (i.e.: Ge) or anisotropy (i.e.: SiN). The second condition is the lateral dimensions of the thin film, especially for an isotropic material. The third condition depends on the quality of the thin film, for example if it is dirty or has macroscopic defects, as shown by the two SiN thin films.
- Multi-layer sample: can be approximated as a single layer to get average stress.

- 3C-SiC shows defects can affect overall stress by comparing results for the single layer and multi-layer samples.
Chapter 5

Non-linear Vibration of Thin Films

In chapter 4, thin films were oscillated under a small driving force with a sinusoidal oscillation, and operation was predominantly in the linear regime at higher pressure. However, the linear regime is harder to maintain when the air damping is removed and the sample thickness or lateral dimensions grow smaller. Thus, the non-linear effects become more expressive and must be understood.

The problem of non-linearity for a vibrational thin plate has been formulated by using a variational method and can be incorporated with the effects of geometric non-linearity or other non-linear effects. A literature review of work on the non-linear vibration of plates is given by Chia (1980, 1988) [128, 129] and Sathyamoorthy (1987) [130]. Chu and Herrmann (1956) [131] did a fundamental study of the analysis of large amplitude vibration of rectangular plates. Complete experimental results with different boundary conditions are given by Amabili (2004, 2006) [132, 133, 134]. However, there are not many explanations or calculation results for nano or micrometre thickness thin films which are vibrated in the non-linear regime and behave as membranes, rather than plates. In this chapter, the theoretical foundation of non-linear dynamics is reviewed briefly, followed by a focus on the experimental results which were observed on different suspended thin film samples. These were all framed on a silicon-base and made from a single material layer, with the thin film material being germanium (Ge), cubic silicon carbide (3C-SiC) or silicon nitride (Si$_3$N$_4$). A three layer material with layers of 3C-SiC, Si$_{1-y}$C$_y$ and Si$_{1-x}$B$_x$ was also studied. The sample sizes (lateral dimensions) varied from a few millimetres down to around 310 µm, with thicknesses from submicron down to around 100 nm. To measure such a range of sample sizes, two different interferometer laser probe spot
sizes were used for the investigation, with diameters of 30 µm and 200 µm, as with the linear resonance measurements.

From the experimental results, the phenomenon of internal resonance (section 5.2.2) has been observed and studied in some detail. The thin films were vibrated using a large oscillation force in a close to air-damping-free regime (i.e., in a microbar low pressure) as described in chapter 3. The non-linear dynamics were analysed further using a spatial modulation which is being developed by the research group of AG Scheer at Konstanz University [100].

The curvature of the frequency response close to resonance exhibited some similarities to the characteristic non-linear jump phenomenon from the backbone of Duffing-oscillation (section 5.4.1) [135], but overall a different behaviour was found. The observation results of the non-linear behaviour strongly rely on the investigation conditions such as the strength of excitation force, the measurement position, or the size ratio between laser spot and thin film area. All these details will be discussed in the following sections.

The thermal effects from the probe laser are not discussed in this chapter, but are explained in detail in chapter 6. However, for these measurements of the heating influence due to the probe laser was minimised. All measurement results presented in this chapter were obtained when the sample temperature was close to its thermal equilibrium state, and low laser power were used.

5.1 Non-linear Oscillators

An ideal physical system subjected to an undamped linear sinusoidal oscillation with low driving amplitude should exhibit a simple sinusoidal waveform with a constant peak amplitude, and a phase which is determined by the starting value. The period is a constant, independent of the starting conditions, the amplitude of the motion, and the time. An example is a simple hanging pendulum with free motion, in the small angle regime.

In the real world, the ideal linear oscillator models need to be reconsidered primarily due to two reasons: firstly, there is inevitably some damping action (non-zero damping), such as air resistance; secondly, all real system will have some degree of non-linearity (e.g., non-linear stiffness), which in itself modifies the behaviour in important ways, such as when the pendulum is undergoing arbitrarily large oscillations [136].

For the simple undamped pendulum with free motion, the restoring force to bring the weight to its equilibrium position is \( W \sin \theta \), where \( W \) is weight and \( \theta \) is the
angle between the pendulum and equilibrium position. For small angles, the approximation \( \sin \theta = \theta \) can be made to linearise the equation and describe the system as a linear oscillator. However, in the large-amplitude oscillation limit, the approximation can no longer be made. This gives the equation, using Newton’s law of motion,

\[
l \frac{d^2 \theta}{dt^2} = -W \sin \theta,
\]

where \( l \) is the length of the pendulum, as a non-linear differential equation to describe the motion.

The solution of the non-linear differential equation can be obtained, after some algebra, in terms of elliptic integrals [135, 136]. With a given starting condition and without damping, a steady waveform of constant peak amplitude and period can be achieved. However, the waveform motion obtained is not sinusoidal and can be decomposed using a Fourier transform into a fundamental frequency plus odd higher harmonics.

According to the description of harmonic distortion, a sinusoidal input force (single frequency) used to drive a simple oscillator into the non-linear regime will give an output response which contains a set of frequencies including the driving frequency and its harmonics. The harmonic series components are related to each other by integer ratios, defined as \( f_n = nf_{\text{drive}} \), where \( n = 1, 2, 3 \).

The above results use the assumption that the non-linearity in an oscillator will become much more obvious with increasing driving amplitude. The frequency of the oscillator vibrations might also be changed and related the amplitude. With these two properties, the non-linear system can produce a very curious shape for the oscillation resonance curve when the oscillator is driven by different amplitudes.

### 5.2 Non-linear Dynamics

It is important to understand the non-linear dynamic behaviour, in order to avoid it when it is unwanted, or to use it where it is needed, and to exploit the efficiency of the micro or nano scale devices. Knowledge of the non-linear vibrations bears rich potential to expand the dynamic performance and the application limits of cutting edge technology to a broad range of areas such as noise sensing [137, 138, 139], optical quantum metrology [140, 141], nano-electromechanical logic gates [142, 143] and parametric oscillators [144, 145].

The theoretical framework for non-linear behaviour has been studied by many scholars [132, 134, 146, 147, 148, 149, 150]. The source of the non-linearity in vibrating
objects can be due to the oscillation amplitude, as described above, and the material, inertial, geometric, and damping non-linear behaviours.

The material non-linearities arise from non-linear stress-strain relationships, generally exhibited as softening in a polymer thin film. The inertial non-linearities exist through a non-homogeneous concentration or distribution of masses in the system [82, 132, 151]. The geometric non-linearities result from non-linear strain-displacement relationships for large deformations and are usually of the hardening type. The non-linear damping is much more complicated, and it has been suggested that it is related with resonant modes. In addition, non-linearities can also occur with non-uniform boundary conditions [152, 153, 154, 155].

For a large amplitude vibration of a thin film with small damping, the non-linearity behaviour observed may mainly arise from the material non-linearity and geometric non-linearity, for perfect support boundary conditions. Large deflections lead the geometric non-linearity to cause a non-linear strain-displacement relationship, and hence non-linearity in the governing differential equation due to the coupling of in-plane and transverse displacement fields [156, 157, 158]. As the deflection of the thin film increases, the bending effect becomes more pronounced, particularly when the thin film is considered as a stretched membrane. The restoring force for this is mainly from the residual stress and neglects the elastic force from the bending for nanometer sized curving of the film [135, 159].

The thin film vibrated in the linear regime was discussed in chapters 2 and 4. A general approach to mechanical oscillation is to consider a system as a simple harmonic oscillation. However, as shown here, this description does not hold the complete truth whenever a large deflection amplitude is involved, because of the geometric non-linearities arising from non-linear strain-displacement relationships due to a large deformation. Modifications to the simple theory are considered below.

5.2.1 Duffing Oscillators

For discussing the non-linearities in a single-degree-of-freedom system due to the geometric effects and linear forces, the most well-known non-linearity model is the Duffing Oscillator, modelled as a spring pendulum. This is governed by equation 5.1

\[ \ddot{u} + \omega_0^2 u = \epsilon f(u, \dot{u}) + E, \quad (5.1) \]

where \( u \) is the displacement, \( \epsilon \) is a small parameter, \( f \) is a non-linear function of \( u \) and \( \dot{u} \) and \( E \) is the external excitation force [135].

Because the thin film system considered here is driven by a stationary excitation,
the system can be treated with cubic non-linearities and linear damping \[^{135}\]. The Duffing equation can then be rewritten as

\[
\ddot{u} + d\dot{u} + \omega_0^2 u + \beta u^3 = F_0 \cos(\Omega t),
\]

(5.2)

where \(d\) is a positive damping constant, the non-linear coefficient \(\beta\) can be either a positive (hardening) or a negative (softening) constant, \(F_0\) is the force, \(\Omega\) is driving frequency and \(\omega_0\) is the primary resonant frequency.

The approximate solution of the linear damping Duffing equation is \[^{135}\]

\[
\Omega = \frac{1}{2} \left[ (2\alpha - d^2) \pm \sqrt{(2\alpha - d^2)^2 + 4\left(\frac{F_0^2}{A^2} - \alpha^2\right)} \right], \quad \alpha = \omega_0^2 + \frac{3}{4} \beta A^2.
\]

(5.3)

The amplitude of the resonance can be plotted against the frequency ratio between the excitation frequency and fundamental resonance. In figure 5.1(a), five different curves are shown for the following five values of the non-linear coefficients; \(\beta = -2\) (red line), \(-1\) (green line), \(0\) (black line), \(1\) (blue line), and \(2\) (purple line). Fixed values of the damping coefficient and excitation force were used for this calculation, \(d = 1\) and \(F_0 = 40\).

Figure 5.1: Amplitude response curves. (a) A simulated Duffing amplitude response curve under different non-linearities; (b) experimental results from Antonio et al. \[^{160}\].

In the example shown in figure 5.1(a), with a positive non-linear coefficient, the natural frequency increases as the amplitude grows, and the sample stiffness becomes harder with more bending. For the opposite behaviour (negative non-linear coefficient), an increased amplitude would result in the natural frequency decreasing, shown by the resonant peak bending over to the lower frequencies as the sample...
The non-linear stiffness behaviour gives an important phenomenon that can be observed in experiments, from the bent curve of the amplitude response to the driving frequency and applied voltage. The amplitude response will show a jumping motion when the excitation frequency is swept upwards or downwards around the resonant frequency, as shown by the experimental results in figure 5.1(b), which measured a clamped-clamped string oscillator using lateral comb-drive electrodes for actuation and detection [160]. With reference to figure 5.1 for a positive non-linearity (hardening type), the steady amplitude of vibration increases as resonance is approached. A further increase in frequency will cause a sudden decrease in the amplitude of the oscillation response of the system once the peak is reached, as shown by the arrow in figure 5.1(a). For reducing frequency from high to low (downwards frequency sweeping), the maximum amplitude for the resonant response cannot be approached as it is when sweeping upwards in frequency, but the behaviour will still illustrate a jump from lower amplitude to a larger value at a particular frequency (shown as the arrow with dash line). In contrast, for a softening non-linear system, the jumping behaviour will be reversed, with the non-linear jump in amplitude greatest when frequency sweeping from high to low frequency.

Figure 5.2: Measured resonance curve from upwards frequency scan on a Si$_3$N$_4$ thin film undergoes a 15 mV driving voltage and fitted with a Duffing curve.

An experimental result of the amplitude response curve with upwards swept driving frequency with a 15 mV excitation voltage is shown in figure 5.2 as the black line with points, for a Si$_3$N$_4$ thin film with 310 $\mu$m lateral dimensions and 300 nm thickness (first Si$_3$N$_4$ thin film, Sample SiN_1), with the full experiment discussed in section 5.4.2. A Duffing fitting was applied to this experimental result, shown as a red line on the same figure. Most of the result points are fitted reasonably close to
the Duffing curve, and the behaviour exhibits the jumping phenomenon. However, an additional small dip feature is observed in the section of steady amplitude, which does not fit into the Duffing curve well.

5.2.2 Internal Resonance

Variations from the Duffing behaviour may be due to the effect of internal resonance [160, 161, 162]. A system with a single degree of freedom, such as a simple pendulum, has only a single natural frequency and a single resonance mode. However, a thin film can have multiple natural frequencies and multiple corresponding natural modes, as shown in chapter 4. Therefore, thin films are systems with multi-degrees-of-freedom. Because a non-linear oscillation can generate harmonics from a single input frequency, one can consider each of the harmonics as an individual resonance, interacting with the other resonant modes for the system. The different resonant modes might be strongly coupled together, thus the harmonics may result in an internal resonance due to the modes mixing. When internal resonance occurs in a vibration system, energy imparted initially to one of the modes involved in the internal resonance will be continuously exchanged among all the modes involved in that internal resonance [163]. If damping is present in the system, then the energy will be continuously reduced as it is being exchanged from a higher frequency mode back to a lower frequency mode. If an harmonic external excitation at the resonant frequency acts on a multi-degree-of-freedom system, there might exist other resonant combinations such as the observation in references [160, 162, 165].

A theory of the combination of resonances was made by Malkin (1956) and was proved by Yamomoto’s experiments (1957,1960) [135]. For modal coupling, one of the earliest experiments was done in 2003 by Baskaran et. al [164] who observed modal coupling in a torsional oscillator. In 2012, Antonio et al. first reported an internal resonance observed in a clamped beam resonator, as shown in figure 5.3 [160].

According to a recent publication, the interaction with an internal resonance can lead to a dip in the Duffing behaviour, as shown in figure 5.3 which was reported by Antonio at al. [160]. In part (a), an amplitude drop observed in the Duffing amplitude curve at figure 5.3. The higher order frequency resonance is three times the driving frequency, shown in part (b) in the output power spectrum. In 1951, the non-linear axisymmetric vibrations of circular membranes were analysed by Eringen using the initial membrane strain as a perturbation parameter [165, 166]. In the 1990s, several scholars studied and developed the theory for the non-linear dynamics, and proved most of these theories with experiments [135]. A circu-
lar membrane with vibration was determined by Yen and Lee (1975) using the Lindstedt-Poincare technique, and they analysed the effects of the longitudinal inertia. However, most of these classical theories were tested by using a large scale size object in the experiments, such as a circular plate. The object sizes usually had a micron or larger scale thickness, and this predominantly gives the behaviour of metal plates rather than membranes. Even so, some new models of non-linear dynamics with internal resonance have been established and developed by starting from the classical elasticity theories such as the Duffing equation, for a small scale system \[162\].

A general equation to describe non-linear vibration behaviour with coupling between modes due to the internal resonance for two coupled modes, can be described as follows \[162\]:

\[
\ddot{u}_1 + \left(\frac{\omega_1}{Q_1} + \Gamma_1(u_1, u_2)\right) \dot{u}_1 + \left(\omega_1^2 + \Lambda_1(u_1, u_2, \dot{u}_1, \ddot{u}_1, \dddot{u}_2)\right)u_1 + \gamma_{12} u_2 \\
= F_1(u_1, u_2, \dot{u}_1, \dot{u}_2, t) \tag{5.4}
\]

\[
\ddot{u}_2 + \left(\frac{\omega_2}{Q_2} + \Gamma_2(u_1, u_2)\right) \dot{u}_2 + \left(\omega_2^2 + \Lambda_2(u_1, u_2, \dot{u}_1, \ddot{u}_1, \dddot{u}_2)\right)u_2 + \gamma_{21} u_1 \\
= F_2(u_1, u_2, \dot{u}_1, \dot{u}_2, t)
\]

where \(u_i\) is the modal coordinate (e.g. \(u_1\) for fundamental, \(u_2\) for the next higher order, which are coupled); \(\omega_i\) and \(Q_i\) are linear resonant frequency and quality factor, describing the linear dynamic aspects of each mode; \(\Gamma_i\) and \(\Lambda_i\) are the terms manifesting non-linearity in the global dynamic response by modulating the modal damping, stiffness and inertia with respect to the dynamic motions of excited modes,
and $\gamma_{12}$ and $\gamma_{21}$ are the linear coupling coefficients. Note that the functions of $\Gamma_i$ and $\Lambda_i$ can induce non-linearity by the amplitude and velocity of both their own mode and another mode when coupled. The geometry, material and boundary conditions should determine the types of these functions and the coupling coefficients, which will alter the global non-linear dynamics of the resonator with non-linear intermodal interactions.

For the clamped thin film system with internal resonance effects, a spatial modulation model is introduced in [100]. This model is founded on the experimental observations of vibrations of thin films measured using white light interferometry, which measure the entire film at one time rather than giving a point-by-point measurement as used here. When the internal effect is present, the thin film would vibrate in distinct sections of the thin film, with different frequencies in each section. Each frequency in the distinct sections of vibration is related to the driving frequency by integer ratios, by $f_n = nf_{drive}$ ($n = 1, 2, 3,$).

The description from the general equation (5.4) with coupling motions, can suggest a link between the whole vibrated thin film and coupled oscillators with different amplitude components in each section. The fundamental mode shape of a square shaped thin film in the linear regime is illustrated as a cone shape with circular symmetry, as presented in chapters 2 and 4. To simplify the discussion of the spatial modulation with internal resonance, here the thin film system is analysed in two distinct sections which are disc-shaped; the centre circle has a radius $r$ from the centre and the second section is localised as a ring of radius between $r$ and $R$ from the centre, with the schematic diagram shown in figure [5.4]. The vibration frequency in the ring section is set to the same as the driving frequency, which should be close to the thin film natural frequency ($f_d \simeq f_{res}$), and the vibration frequency in the centre section where internal resonance occurs is allowed to be at an integer times the driving frequency ($f_{inter} = nf_d$).

The global profile of such a vibrational state with the spatial internal resonance can be described for a square thin film as

$$u(r, t) \simeq q_C \cos(2\pi f_{inter}t)u_C(r) + q_R \cos(2\pi f_d t)u_R(R - r) \quad (5.5)$$

where $u_C$ and $u_R$ are two arbitrary spatial profile functions having a maximum, and the two amplitudes of oscillation are $q_C$ and $q_R$, respectively [100]. $q_C$ and $q_R$ are orthogonal and couple with one another because the linear resonance modes have spatial overlap with the higher frequency internal resonance. The equa-
Figure 5.4: Schematic diagram of resonance mode shape for a thin film with a second order harmonic internal resonance.

The equation for the amplitudes $q_C$ and $q_R$ when $f_{\text{inter}} = 2f_d$ are

$$\ddot{q}_C = -4(2\pi f_R)^2 q_C - 2\Gamma_C \dot{q}_C - \frac{1}{2} \lambda q_R^2,$$  \hspace{1cm} (5.6)

$$\ddot{q}_R = -(2\pi f_R)^2 q_R - 2\Gamma_R \dot{q}_R - \alpha_R q_R^3 + F_d \cos(2\pi f_d t) - \lambda q_R q_C,$$ \hspace{1cm} (5.7)

where $\alpha_R$ is the strength of the Duffing non-linearity, $\Gamma_C$ and $\Gamma_R$ are the damping constants for the two sections and $\lambda$ is the interaction strength between the oscillations in the two sections.

In figure 5.5 this spatial modulation model is applied to fit the same experimental results as presented in figure 5.2. In this case, the trend of the small feature observed in the top of the maximum steady amplitude section around 1.2585 to 1.2586 MHz has been fitted by this model. Although this fit is not perfect, there are significant improvements over the fitting using the Duffing model directly.

Figure 5.5: The same experiment results shown in figure 5.2 but fitted with the spatial modulation model.
5.3 Non-classical Elasticity Theories

For all the above classical non-linear dynamic theories, the main restoring force in all systems is the elastic force from dynamic deformation such as bending, and the initial residual stress which is already built into the materials or structures is neglected. However, for nano or submicron scale thickness thin films, the restoring force and residual stress might be reversed, with the residual stress causing the main effect because the deflection is too small in the mechanical motions \[80\]. It is possible to neglect the elastic force from the structure bending when a system is vibrated in a linear regime with a membrane behaviour, as discussed in chapter 2.

The properties of materials, including electrical conductivity, thermal properties, mechanical properties, and other known physical and chemical properties can change when samples have nanoscale dimensions \[167\]. According to all these changes, a question should be raised as to whether the classical elasticity theories which are based on the observations of a macro scale system are still suitable for the nano or submicron scale systems, especially if the main restoring force has shifted to the residual stress.

In the early 1900s, a theory was proposed by the Cosserat brothers to predict the behaviour of nanomaterials, and formed the beginning of non-classical elasticity theories \[167, 168\]. Other non-classical theories for studying the behaviour of small-scale systems include coupled stress and consistent coupled stress theories \[169, 170\], surface stress theory \[171\], non-local theory \[172, 173, 174, 175, 176\], and strain gradient theory \[177, 178, 179, 180, 181, 182, 183\]. These theories all include constants which affect the mechanical dynamics of the small-scale systems. To develop the theory of internal resonances for small-scale systems with non-linear coupling, much work was done in the 2000s \[162\], while experimental realization and applications matured starting in the early 2010s. Westra et al. reported the intermodal coupling in a microresonator in 2010 \[184\], a circular membrane resonator with a quadratic coupling was considered and observed in references \[185, 186\], and more investigation of internal resonance is presented in references \[160, 187, 188, 189, 190, 191, 192, 193\].

From the experimental results and theoretical predictions, one typical feature shown by a system with an internal resonance is the amplitude drop and saturation in the amplitude frequency response curve (figure 5.3). The amplitude dip occurs when the internal resonance is switched on. To form the higher frequencies of the internal resonance, the mechanical energy in the linear eigenmodes would be reduced, thus the amplitude would drop to a lower branch of the resonant curve \[160\]. Another
feature is that the oscillation frequencies of the internal resonance are integer $n$ times the driving frequency, as before.

Keivan A. et al. (2018) systematically reviewed the area and concluded that the non-linear behaviours in micro and nano-mechanical resonators are due to the internal coupling of multiple modes \[162\]. Mohammad H. et al. (2018) reviewed how the non-classical elastic theories with a size dependence influence the analysis in small scale systems \[167\]. Even though a large number of theoretical studies on non-linear vibration with internal effects, using classical and non-classical theories, are available in the scientific literature, experimental results are still scarce.
5.4 Experiment Results Measuring Non-linearity in a Thin Film System

5.4.1 General Amplitude Frequency Response

According to the description from the Duffing theory, typical non-linear behaviour of the amplitude frequency response curve would show the curve becoming asymmetric when a system is driven strongly through the resonance. The resonant frequency would be shifted, either to higher frequency for positive, known as hardening, non-linearity, or to lower frequency for negative non-linearity as it becomes softer (figure 5.1). If the system is a simple resonator, harmonics would be observed with a corresponding reduction in amplitude for the resonant frequency.

![Amplitude curves from a frequency scan on a Ge thin film with 700 nm thickness and 955 µm lateral dimensions with a varying transducer excitation voltage from 100 mV to 600 mV.](image)

Figure 5.6: Amplitude curves from a frequency scan on a Ge thin film with 700 nm thickness and 955 µm lateral dimensions with a varying transducer excitation voltage from 100 mV to 600 mV.

A square shaped germanium thin film, with a thickness of 700 ± 12 nm and lateral dimensions of 955 ± 20 µm, was excited with the transducer drive voltage varied from 100 mV to 600 mV at a 1.4 × 10⁻³ mbar pressure. The interferometer laser probe size used was 200 µm in diameter, which is smaller than the film lateral dimensions and hence the film is measured over a small region rather than seeing the
full film motion. The frequency sweeping range was around the natural fundamental resonance (153.09 kHz) measured in the linear vibration regime of this thin film. Six vibration amplitude versus frequency curves, measured at the centre position of the thin film, for various driving voltages, are illustrated in figure 5.6. (a) and (b) show typical vibration in the linear regime, with a clear resonance peak. Another four of the amplitude frequency response curves show a modification to this performance as the driving voltage, and hence transducer displacement, is increased. This is unlike the Duffing frequency response, even though the thin film was driven into the non-linear vibrational regime when the excitation voltage was above 300 mV. The resonant frequency does not shift to a higher or a lower position, but instead a feature is observed with the amplitude dropping to a lower value when the driving frequency is close to the initial resonant frequency. Although the behaviour observed is not Duffing-like, this is because it is a local measurement rather than measuring the whole sample at once, which will be discussed in the next two sections.

Figure 5.7: Oscillation raw data at 153.09 kHz driving frequency on the Ge thin film with 700 nm thickness and 955 µm lateral dimensions.

A superharmonic phenomenon is observed in the raw vibration data, which suggests the oscillation is in the non-linear regime when transducer driving voltage is increased. Figures 5.7 (a) to (f) show the raw vibration data for this Ge thin film with the six different transducer driving voltages. At 100 mV voltage, the oscillation trace shows the same sinusoidal shape as the input signal from a function generator. As the voltage is increased, moving from 200 mV to 600 mV, the signals initially
increase in amplitude but start to show deviation from sinusoidal. Following this, shown in parts (c) to (f) of the figure, the amplitude drops as the vibration energy moves into the higher harmonic frequencies, and this effect becomes much clearer at higher voltages. This obeys the classical theory statement in section 5.1 as a large amplitude oscillation can create non-linear vibration with harmonic distortion.

The vibration displacement profile of the thin film depends on several factors, including the resonant mode shape. For the fundamental resonance mode in the linear regime, the maximum displacement position is at the centre of the thin film. The vibration strength of the transducer itself during operation depends approximately linearly on its driving voltage at each frequency, as shown in figure 5.8. The displacement of the thin film should follow this linear behaviour for low amplitude displacement (i.e. in the linear vibration regime), but with larger amplitude due to the resonant vibrations.

![Graph of Amplitude vs Excitation Voltage](image)

Figure 5.8: Strength of transducer amplitude response with a varying excitation voltage, measured at a position on the sample substrate.

The displacement at the thin film centre with increasing excitation voltage, driven at the fundamental resonant frequency, has been measured on two different thin film samples. The first sample is the same Ge thin film with 153.09 kHz fundamental resonant frequency which is presented earlier, with results shown in figure 5.6. The second sample is a 3C-SiC thin film that was grown to a thickness of 685 ± 15 nm, with 1470 ± 20 µm lateral dimensions, with the fundamental resonant frequency found at 177.19 kHz.
Figures 5.9 (a) (Ge thin film) and (c) (3C-SiC thin film) show the relationship between measured displacement and excitation voltage at a position near the thin film center. The displacement is approximately linear until a critical displacement is reached ($10.4 \pm 0.1 \text{ nm}$ in Ge and $10.8 \pm 0.1 \text{ nm}$ in 3C-SiC), after which the amplitude is reduced. Parts (b) and (d) show fast Fourier transforms (FFTs) of the raw data for these two samples respectively, plotted as a function of the excitation voltage. The colour scale represents the magnitude, with the scale varying from blue (low) to white (high). The odd harmonics appear preferentially over even harmonics, as expected from geometric considerations. Around the point where the displacement is most reduced (about $0.42 \text{ V}$ in figure 5.9 (a), $0.16 \text{ V}$ in (c)) the second harmonic also appears. The exact voltage at which these occur depend on the coupling between transducer and sample, while the critical displacement is expected to be material and dimensions dependent.

When the same measurement was repeated on a Si$_3$N$_4$ thin film sample (Sample SiN$_1$) which has 300 nm thickness and 310 $\mu$m lateral dimensions using the 200 $\mu$m spot size for the detector, the experimental results change and the curve trend is more similar to the Duffing non-linearity, shown in figure 5.10. The characteristic shift in resonant frequency, followed by a drop in amplitude, is clearly observed.
However, for this sample, the 200 µm laser spot size is comparable to the sample size and the measurement is of the majority of the thin film.

Figure 5.10: Resonance curve of the fundamental mode, measured on Sample SiN_1 with 200 µm laser spot size in diameter. (a) with 15 mV driving voltage; (b) with 25 mV driving voltage; (c) with 45 mV driving voltage.

Figure 5.11: Resonance curve of the fundamental mode, measured on Si₃N₄ thin film with 478 nm thickness and 314.5 × 393.5 µm lateral dimensions using imaging white light interferometer with 27 mV driving voltage. (a) response from whole thin film area; (b) selected centre area. [Data provided by Yang Fan, PhD in AG Scheer research group, Konstanz University].

A similar frequency sweeping measurement was done on a Si₃N₄ thin film with 478 nm thickness and 314.5 × 393.5 µm lateral dimensions by the AG Scheer research group at Konstanz University. The resonance curve was measured using an imaging white light interferometer, obtaining the amplitude response by integrating the deflection profile over a selected area on the thin film, as shown in figure 5.11. The whole measurement area (boxed in by the blue dashed line) of the thin film was selected for integrating to give the amplitude-frequency response curve shown in part (a). The measurement result has a strong similarity to the Duffing backbone curve.

In figure 5.11 (b), the integrating area was concentrated into a smaller area (boxed...
in by the black dashed line) at the centre of the sample. The amplitude response curve becomes different when compared with the result shown in part (a). There is a small valley which appears in the amplitude frequency response curve, which is closer to the results observed on the Ge and 3C-SiC thin films, where the probe is smaller than the sample dimensions. The measurement shown in figure 5.10 shows similarities to figure 5.11 (a) and (b), depending on the driving voltage used. The technique using the white light interferometer gives a measurement of the whole sample at the same time. However, the frequency sweep data obtained with the two wave mixer interferometer is produced over a smaller area, given by the laser spot size. The results from different area analysis of the measurement on the Si$_3$N$_4$ thin film raises the questions of why the different integrating area size can affect the features of the amplitude response curve when a clamped thin film system is vibrated in a non-linear regime, causing it to move away from the standard Duffing picture. The reason behind the change and how strong the area effect is must be understood to explain why the local measurement used here for Ge and 3C-SiC typically gives non-Duffing-like results.

5.4.2 Local and Global Measurements of Amplitude Frequency Response

For a linear resonance, most of the mode shapes are predictable (see chapter 2). The most used mode shape for studying a vibrational oscillator (system) is the fundamental resonance, because of the symmetry and the inherent simplicity of the mode pattern. For a non-linear vibration system at resonance, the mode shape can change to a more complicated pattern, which is hard to predict because the non-linearity (or shape) has an oscillation amplitude dependence. Also, an internal resonance could introduce a higher order resonance mode shape to an initially linear resonance mode, which can also make the shape more complicated. The amplitude frequency response curve reflects the average vibrational mode shape over the measurement area, and the vibration strength at that position. Assuming a more complicated mode shape occurs, such a measurement would have a more complex amplitude-frequency response curve, and one would expect it to be position dependent.

In the internal resonance model (sometimes called the spatial modulation model) for non-linear vibration with internal resonances, each of the sample areas undergoing harmonic oscillation is distinct as an individual area with a nodal line surrounding it, forming a new boundary. Each of the sections could be treated as an independent oscillator, having different frequencies and phases as shown in figure 5.12. Parts (a) to
(d) and (i), (j) show the vibrational mode shapes for the fundamental and 1:3 mode. Parts (e) to (h) are the frequency with the maximum magnitude obtained from the FFTs at each position of the mode shapes shown in parts (a) to (d). Parts (a), (e) and (c), (g) are the 1:1 mode and 1:3 mode respectively in the linear regime. The FFTs show that the maximum magnitude frequency is the same at all points on the membrane, within errors. Parts (b) and (d) are the 1:1 mode and 1:3 mode which have been excited in the non-linear regime, showing a reduced amplitude at the centre of each feature. The response resonant frequency shows a higher order harmonic in each of these areas inside the mode shape, as shown in (f) and (h). Parts (i) and (j) also show the 1:1 mode and 1:3 modes in the non-linear vibration regime on a different sample, which were measured by the AG Scheer research group using the imaging white light interferometer. Dark and gray colours show the distinguished sections, but the technique does not give the response resonant frequencies.

Figure 5.12: Resonant mode shapes of 1:1 mode and 1:3 modes in the linear and non-linear resonance regime, and the maximum response resonant frequency at each position. (a) to (d) are the mode shapes; (e) to (h) are the 2D images of maximum magnitude resonant frequency using FFTs of the raw data from each scanning position; all measured on a 3C-SiC thin film. (i) and (j) were imaged on a Si₃N₄ sample using the white light interferometer when 1:1 mode and 1:3 modes were excited in the non-linear vibration regime [data proved by AG Scheer research group].

The basic working principle for an interferometer to accurately measure a dynamic displacement on a vibrating object is that it evaluates the phase compensation.
When the laser probe is fully over a section with a single vibration frequency, the laser probe can assess the displacement well. However, when the laser probe is over a section which overlaps area with different frequencies and phases, this would give some uncertainty for the displacement measurement. Each of the response curves measured will depend on the ratio of probe area to sample and resonance area size, and can be considered as the local amplitude frequency response or global amplitude frequency response depending on the overlap with the different sections of the thin film, as the vibration becomes non-linear.

**Zero Dimensional Scans – Fixed Position**

Figure 5.13 shows frequency sweep amplitude responses in part (a) and the phase in part (b). This measurement was done at around the centre of the 300 nm thickness and 310 µm lateral dimensions Si₃N₄ thin film (sample SiN₁) with a laser probe size of 200 µm diameter. This spot size covered most of the thin film area, thus this measurement should be considered as close to a global amplitude frequency response, similar to the white light interferometry technique.

![Figure 5.13: Resonance curves and relative phase difference measured on Sample SiN₁ with a 200 µm diameter laser spot size.](image)

(a) Amplitude response with varied excitation voltage for the fundamental mode. The frequency scan was done in both sweeping directions; a label of LfHf means upward sweeps, and vice versa. (b) Phase difference between driving and response oscillation of each frequency scan.
Figure 5.13(a) shows the linear resonance amplitude response curve for the 1:1 mode with a 4 mV transducer driving voltage. The phase change is shown in part (b) confirming the peak is a resonance. In the same frequency range, two categories of frequency scan have been done: with upwards and downwards frequency sweeping, for excitation voltages from 40 mV to 110 mV. These are shown in parts (a,2) and (a,3), respectively. Each of the phase changes are plotted in the same section of the figure in part (b).

The amplitude frequency response curves for the upwards frequency sweeping (figure 5.13(a,2)) have a similar trend, showing the expected Duffing-like bending of the resonance curves for a hardening spring. However, there is an extra feature of a drop at the initial linear regime resonance frequency position. This feature suggests an energy transform from a lower order to a higher (2nd) order harmonic with an internal resonance switched on as discussed earlier. After the drop, the amplitude follows a pattern similar to the Duffing oscillator, with a downward jump that depends on driving voltage. Then, the amplitude decreases progressively as the excitation frequency moves away from the resonance.

When the frequency sweep instead goes from high to low frequency, the amplitude response curve shown in figure 5.13(a,3) does not do the reverse to the up-sweep, as expected from the Duffing theory. In addition, it has the same valley at the position of the initial linear resonance. The classical Duffing theory explains the irreversible nature of the amplitude frequency response curve in the non-linear vibration, but does not predict the valley shape. The amplitude drops in the upwards frequency scan have been observed previously, but the researchers did not measure this performance on downward frequency sweeps [160].

Figure 5.14 illustrates the same set of frequency scan measurements on the same Si3N4 (sample SiN,1) thin film sample but with a smaller spot size of the laser probe of 30 µm diameter, again measured at a position close to the thin film centre. Comparing with the same measurement done using the 200 µm spot size, these results show more equivalence on the up and downwards sweeps, as expected for a local rather than global response. The resonance frequency in the linear vibration shows a small shift when measured with the smaller size laser probe. One reason for the frequency shift can be due to the thermal impacts from the laser heating (explained in chapter 6). Another reason could be due to overlapping of the laser probe with the different internal mode sections when the laser spot size is large.

For the upwards frequency sweep, the amplitude frequency response curves (local response) shown in figure 5.14(a,2) have some similarities compared with the global response curves (figure 5.13(a,2)), including the jump at higher frequencies for large
excitation voltage. Both of the measurements have a drop at the position of the resonant frequency in the linear regime. However, there are some obvious differences in the behaviours shown in these two figures (figure 5.13 and figure 5.14), primarily in the amplitude values of the peak before and after the drop, and in the amplitude value at the valley point, as well as the overall shape. Before the drop, the peak strength of the amplitude response from the local measurement is stronger than the result obtained from the global measurement. After the drop, both of the response curves show an increasing amplitude with increasing driving frequency away from the initial resonant frequency, but the amplitude reached in the local measurement with the same transducer driving voltage as applied in the global measurement is now smaller.

The amplitude frequency response curves (local response) given by the downward frequency sweeps are shown in figure 5.14 (a_3). These curves exhibit a typical structure of a hardening Duffing oscillator for this frequency scan direction. There is a sudden upward jump at a certain position as the frequency is reduced, then the amplitude retraces the path as frequency progressively decreases. However, there is a feature which shows these reversing frequency response curves are not exactly the same as the Duffing curve; the Duffing reversing frequency response cannot return
back to the maximum amplitude, as these curves do.

Figure 5.15: 2D images of (a) amplitude frequency response curves and (b) its maximum magnitude resonant frequency at each point on the frequency scan with varied excitation voltage, on sample SiN,1 with 200 \( \mu \text{m} \) diameter laser spot size. Colour scale represents the amplitude in (a) and the main response frequency in (b).

To seek more details for comparing the differences between the observations from measurements of local and global amplitude frequency responses, and give comparison with the predictions of the Duffing model, the upwards frequency scan was done over a wider range of transducer driving voltages, from 2 mV to a few volts. This scanning region covers the linear and non-linear regimes. Figures 5.15 and 5.16 show the results of the frequency scans on two SiN samples at different voltages in part (a) with scanning frequency on the y-axis and excitation voltage on the x-axis. The measured amplitude is shown by the colour scale. This is a single pictorial representation of data similar to that shown in figure 5.13(a). Note that the x-scale is not linear, but is done in sections. The spreading of the resonance and drop at the linear resonance frequency are shown clearly as voltage is increased. However, the drop position shows some variation at higher voltages.

The 2D map of the amplitude frequency response (figure 5.15(a)) measured using the larger laser spot shows the drop section becoming larger and shifting to lower frequencies with increasing excitation voltage. The drop is not always at its initial resonant frequency found at the linear regime, and drops further in frequency as the voltage is increased and further coupling of modes occurs. Part (b) shows the
maximum magnitude frequency in the FFT done on the raw data at every excitation frequency and driving voltage, with this frequency shown by the colour scale. This generally matches the driving frequency, apart from at the drop position, where it is dominated by a higher order frequency. These figures show data measured with the probe laser spot sizes of 200 μm and 30 μm diameter, respectively, on samples SiN_1 and SiN_2, which have 300 nm thickness and 315 × 315 μm lateral dimensions (same Si_3N_4 samples as used in chapter 4).

For the local measurement of the amplitude frequency response 2D map (figure 5.16(a)), the drop does not show the shift in frequency shown in the results from the global observation, and remains at the initial resonant frequency found in the linear regime. The strongest amplitude always appears in the first peak before the drop and the bandwidth of this peak becomes larger as the driving voltage is increased. There is a second smaller drop found around a frequency of 1363.2 kHz. This second region shows higher frequencies and the presence of a 3rd harmonic, and is shown in the next section to depend on measurement position.
One Dimensional Scanning

The shape of the local amplitude frequency response curve may be related to the measurement position, as demonstrated by the complicated mode shapes measured in the non-linear regime. To verify this statement, a frequency scanning measurement was done around the fundamental resonant frequency range, at different positions along one of the centre-lines of a square shaped Si$_3$N$_4$ thin film (sample SiN$^2$), using the 30 $\mu$m diameter laser spot, as a one-dimensional line scan.

Four different positions from the 1D scan results were selected for full analysis. These positions are located at the thin film edge (position 2) at two positions moving towards the centre, and at the centre (position 17). Figure 5.17 shows the response amplitude curves in part (a), and the related maximum magnitude resonant frequency found from the FFTs at each of the positions shown in part (b).

Figure 5.17: Amplitude frequency response curves and maximum magnitude frequency measured on sample SiN$^2$ with a 1D scan. (a) Resonance curves measured at four locations from the thin film edge (position 2) to the centre (position 17), undergoing a strong excitation (voltage from 100 mV to 500 mV). (b) Maximum magnitude response resonant frequency at each of the driving frequencies and voltages.

These local amplitude frequency response curves show very different features when measured at different locations and voltages. When the detection point was placed close to the thin film edge (position 2) and with a strong excitation voltage, the displacement response to the excitation frequencies exhibits the same features as a Duffing response, as shown in figure 5.17(a_1), with no drop at the linear resonant
frequency. When the detection position is shifted to closer to the centre, the response curves are deformed away from the Duffing behaviour. Firstly, at position 7, the amplitude decreases progressively as the excitation frequency moves further away from the resonance, without the drop behaviour, as shown in figure 5.17(a_2). For closer to the centre (position 12), the peak feature with a drop begins to appear, but not as clearly as shown in figure 5.17(a_4) measured at the centre. When the detection position is located at the centre, the amplitude frequency response curve exhibits an oscillating structure with increasing frequency, as shown in figure 5.17(a_4) and in figure 5.18.

Meanwhile, the main resonant frequency measured from the maximum magnitude in the FFTs also display differences between the data taken for a measuring position at the centre or at the edge of the sample. Away from the centre, the measured frequency is the same as the driving frequency (figure 5.17(b_1)-(b_3)). However, when the measurement is done at the centre of the thin film, the higher order harmonics resonances are built up as the excitation frequency sweeps upwards, and the main response frequency shows the harmonics dominating (figure 5.17(b_4)).

Figure 5.18 shows the FFTs of the raw data at these four selected positions, with different excitation voltages, plotted as a function of the applied frequency during a sweep. The colour scale shows the magnitude, with the scale varying from blue (low) to white (high). The fuzzy section (green area) is caused by noise, as the vibration displacement is weak at positions close to the thin film edge. By comparing these figures one can notice that the higher order harmonics appear at the centre at lower excitation voltage, and appear closer to the edge at much higher voltage. Each of the harmonics has a different strength at a different position, even when the thin film is vibrated at the same frequency and the same excitation voltage. These figures strongly support the model of the clamped thin film vibrating in sections in the non-linear regime, and agree with the internal resonance model.

The previous measurements at the thin film centre for the Ge and 3C-SiC samples, shown in figure 5.9, demonstrate the relationship between amplitude, excitation voltage and the corresponding resonant frequencies. There is a critical displacement point at which the higher order harmonics appear. According to the 1D frequency scan measurement results on the SiN_2 sample, the amplitude response also shows a strong dependence on the detection location. This could be due to position dependent variations in bending of the film.

Figure 5.19 shows the amplitude behaviour for the Si_3N_4 thin film sample SiN_1 as a function of the excitation voltage, driving with the fundamental resonant frequency (at 1256.17 kHz), at thirteen different positions from the edge (position 1) to the
Figure 5.18: FFTs of the vibration data at different measuring positions with the driving frequency around the 1:1 mode resonance amplitude curves in Figure 5.17. Each of these plots are related to the resonance, for a set of excitation voltages on sample SiN2 in the nonlinear regime.
Figure 5.19: Amplitude response at different excitation voltages for the fundamental resonance of sample SiN$_1$ at 1256.17 kHz at different measuring positions, moving from the thin film edge along a centre-line. The scanning results are shown at thirteen measuring points, with position 1 at the edge, and the centre of at position 12.

Figure 5.20: Four selected amplitude responses at different excitation voltages from figure 5.19 [(a)-(d)], and each of the corresponding FFTs [(e)-(h)].
centre (position 12). Figure 5.20 shows four of the curves measured at different positions selected from figure 5.19 in more detail, and the FFTs of the vibration raw data. From these figures, the biggest critical amplitude of vibration is at the centre of the thin film, as expected from geometrical considerations. The turning point following reaching critical displacement is not always observed because it is also location dependent, although higher harmonics still appear at the thin film edges.

Analysis of modes shapes

![Figure 5.21](image)

Figure 5.21: 3C-SiC thin film with 240 nm thickness and 2055 × 2090 µm lateral dimensions, 1:1 mode at 131.65 kHz, with varied voltages measured using 30 µm diameter laser spot size. (a) 1D mode seen shape along the film centre-line. (b)-(j) raw vibration data along the scanning line, for different excitation voltages.

To minimise the effect of area averaging due to the laser spot being a significant proportion of the film area, and any effects due to having different frequency and phase across this area, one can decrease the ratio of the area between the laser spot size and sample area, defined as $\eta = \frac{A_{\text{laser}}}{A_{\text{sample}}}$. A 3C-SiC thin film sample, with 2055 × 2090 µm lateral dimensions and thickness of 240 nm, was measured using the laser probe with the spot size of 30 µm diameter, giving $\eta \approx 0.0165\%$, to investigate the smallest available value of $\eta$ in our sample set. Figure 5.21 (a) shows a 1D-scan across the mid-line of the fundamental mode for the 3C-SiC thin film with a varying transducer driving voltage from 50 mV to 9 V at the fundamental resonant frequency of 131.65 kHz. A simple Gaussian-like peak would be expected if the resonance was purely linear, and the extra details must come
from non-linear behaviour. Figures 5.21(b) to (j) show the vibration as a function of time, driven with the fundamental resonance, at each point on the line. These are plotted as an image, showing time versus scan position with the amplitude shown as a colour scale, for three cycles of the driving frequency at each measurement point. The corresponding resonance spectra (FFTs) are shown in figure 5.22. Increasing non-linearity is observed as voltage is increased.

The 1D mode shapes measured across the cross-section demonstrate changes in the bending of this thin film with different excitation voltages. Even with a 50 mV driving voltage, the amplitude around the centre of the film has already begun to become smaller with internal resonance-like features, and the appearance of a higher harmonic in the FFTs. As the excitation voltage is increased, the fundamental mode shape is deformed further from a smooth peak (in the linear regime) to a clear crater-shaped profile with structure in between the two edges of the mode. Each of the wrinkles forming this structure has an individual resonance behaviour, shown by figure 5.21(b)-(j), and the section of the thin film over which it is measured can be regarded as an independent section, as stated in the internal resonance model. Inside of the crater shape, the number of higher harmonics observed is gradually
reduced as the probe laser moves from the centre to the edge of the film, and each of the dips in the 1D scan is the boundary formed to isolate the harmonics. The height of the crater edges is reduced as the level of harmonic excitation increases. At the edges of the crater, the resonant frequency is equal to the driving frequency. Generally, the highest order harmonic is observed at the centre of the thin film, with some exceptions shown in figure 5.21 (j) for a 9 V excitation voltage. The area averaging effect also corresponds to the number of wavelengths per spot size, which depends on sample material. When the spot diameter is the same size (or integer number) of the wavelength of the resonant vibration, then the measured magnitude of vibrational displacement would show as zero by the light coherence. This is perhaps the reason that the central area displacement is zero at the 9 V excitation driving voltage shown in figure 5.22 (a) for the higher harmonics.

![Figure 5.23: 3C-SiC thin film with 240 nm thickness and 2055 × 2090 µm lateral dimensions, 1:2–2:1 mode at 241.58 kHz, with varied voltages measured using 30 µm laser spot size. (a) 1D mode scan shape along a diagonal-line. (b)-(j) raw vibration data along the scanning line for each excitation voltage.](image)

The same 1D scan measurement was repeated for the 1:2–2:1 resonance mode with a driving frequency of 241.58 kHz. The scanning line was changed to a film diagonal due to the mode shape, with two Gaussian-like peaks expected in the linear regime. Similar behaviour to the 1:1 mode was observed in this 1:2–2:1 mixed mode. Firstly, the scan shape was modified away from the linear expectation, with an amplitude decrease and dips appearing when higher order harmonics appear. With increasing excitation voltage, more and more features appear in the dip. For this 1:2–2:1 resonance mode, there are two peaks with a 180° phase difference. Thus, there are
two craters generated in the non-linear regime. The 1D mode shape, vibration as a function of time, and response resonance spectra are shown in figures 5.23 and 5.24, respectively.

Two Dimensional Scanning

The experimental results from the 1D-scans depict a simple suggestion for how the higher order harmonics disrupt the relative mode shapes, through studying the cross-sectional view. This section expands the scanning to show a 2D fundamental mode shape image measured on a Ge thin film of $2850 \times 2855 \mu m$ with a laser probe size of $200 \mu m$ diameter, undergoing a set of strong vibrations with excitation voltages from 20 mV to 480 mV, and driving at the fundamental frequency of 49.57 kHz. This should show the effect of the internal resonance and the split of the sample into different regions in more details (videos of mode vibrations are available in the digital copy).

The mode shape images were done by performing a 2D-scan and measuring the amplitude from the peak to peak displacement at each measurement point. The first row in figure 5.25 (a) to (e) shows the vibration mode shapes at the funda-
mental frequency with the varying excitation voltages without a bandwidth filter to remove the higher order resonances. The second row from (f) to (j) are the same mode response but after the high order harmonics are filtered out, to show only the behaviour at the fundamental frequency. After the higher order resonances are removed, the amplitude at the centre section becomes smaller (colour changed from green to blue). The bottom from (k) to (o) shows the maximum magnitude frequency from the FFTs of the raw data at each scanning position. These 2D-scan images in figure 5.25 also show that the higher order resonances are initiated and spread from the centre section. To justify that the results from this 2D-scan with a larger laser spot size are comparable with the results from a 1D-scan in section 5.4.2, a centre-line cross-section scan which shown the mode structure, response resonance spectrum and vibration as a function of time is given in figure 5.26. The top row of figure 5.26 ((a)-(e)) are the mode shape and shows similar drips appearing as transducer driving voltage is increased. The second row is the FFTs at each position in the scan, showing onset of higher harmonics which are concentrated in the centre. The bottom row shows the oscillation raw signal at each position. All these results offer similar conclusions to those which were attained from the 1D-scan on the 3C-SiC sample, even though this measurement used a larger laser spot. The spatial averaging effect is different for the different of laser spot sizes, presenting as an alteration to the resolution and accuracy of the drops in the mode shape and raw oscillation signal.

The drops in the vibration displacement are related to energy dissipation in the thin film. The law of energy conservation states that the total energy of an isolated system remains constant. Considering a steady state force vibration thin film system with a constant energy dissipation (through damping), the loss of energy to damping is balanced by the energy which is supplied by the excitation. When the input energy increases progressively as the excitation voltage rises, the potential energy which is stored in the thin film due to bending and stretching increases, and will reach a limit where the extra energy cannot give a higher amplitude vibration. This energy is moved into higher harmonics with higher kinetic energy, thus changing its dynamic behaviour.
Figure 5.25: 2D-scan images of Ge thin film with 4000 nm thickness and 2850 × 2855 µm lateral dimensions, with a driving frequency at the fundamental resonance in the linear regime of 49.75 kHz, and transducer driving voltage from 20 mV to 480 mV and a laser spot size of 200 µm. (a)-(e) are the resonance modes at a bandwidth filter to remove the higher harmonics from the raw vibration data. (f)-(j) are the resonance modes from the raw vibration data. (k)-(o) are the maximum magnitude frequency at each point from FFT of raw oscillation data. (a)-(e) are the resonance modes at a bandwidth filter to remove the higher harmonics from the raw vibration data. (f)-(j) are the resonance modes from the raw vibration data. (k)-(o) are the maximum magnitude frequency at each point from FFT of raw oscillation data.

(a) 480 mV
(b) 400 mV
(c) 300 mV
(d) 200 mV
(e) 20 mV
Figure 5.26: Sample and setup as in figure 5.25. (a)-(e) are the 1D-cross-section resonance mode shapes on a centre-line from the 2D mode images shown in figure 5.25. (f)-(j) are the related FFTs of raw data from each scanning point on the 1D-cross-section resonance mode shapes. (k)-(o) are the vibration raw data at each scanning position; the colour scale presents the vibration strength and phase.
Figure 5.27: Total amplitude of resonances of Ge thin film with 4000 nm thickness and $2850 \times 2855$ µm lateral dimensions, by summing the bending displacement at each position on a selected area in the 2D-scan mode shapes. There are three different curves calculated from three different areas; the first curve summed all the points on the 2D scan mode map, second curve summed over a smaller section which removed the substrate area (whole thin film area), the last curve demonstrates the results calculated from the centre section which only contains the higher harmonics when excitation voltage is 480 mV.

Figure 5.27 shows a different way of visualising the vibration. A total amplitude response for the thin film is calculated by summing all the amplitudes of the fundamental mode shape taken at each measurement point in three selected area: the first area is the whole scanning area including the substrate section; secondly, the whole thin film area was summed without the section of substrate; the final area is a small section on the centre of the thin film which would contain most of the higher harmonics. This total amplitude of the deflection profile is partially related to the potential energy of bending. On slowly increasing the excitation voltage from 20 mV to 480 mV, the total amplitude grows for the results when summing over all scanning areas (first selected area) or the area of the whole thin film (second selected area). However, when the summing area is reduced to the small section which at is the centre of the thin film, inside of the crater in the mode shape, the total amplitude reaches a steady value and shows a slight reduction as voltage is
further increased. This phenomenon agrees with the premise that there is a limitation for the potential energy which is stored in the thin film during bending. Also, when using the total amplitude calculated from all scanning areas to reduce the value calculated from just the thin film area, the remaining value shows a linear relationship with increasing voltage. This linear behaviour shows that the transducer is operating in the linear regime, as discussed in section 5.4.1.

Figure 5.28: Analysed amplitude relation with the excitation voltage on sample 3C-SiC with 240 nm thickness and 2055 × 2090 µm, based on the results of 1D-scan as shown in figures 5.21 (a) and 5.23 (a).

Figure 5.28 shows the same amplitude analysis done on the 1:1 and 1:2 – 2:1 modes from the 1D cross-section scan results on the 3C-SiC sample. Figures (a) and (d) are the total amplitude obtained by summing over all scan positions and without considering the phase difference effect for 1:2 – 2:1 mode. (b) and (e) are the maximum amplitude found from the mode shape. (c) and (f) show the relationship between the excitation voltage and the position on the film which the maximum amplitude is measured.

Comparing figures 5.27 and 5.28 (a) and (d), they show as amplitude that initially increases with voltage before reaching approximately a steady value. However, with a further increased driving voltage, the total amplitude and the maximum vibration amplitude reduce until they reach to a lower steady level. This feature is clearly shown in figure 5.28 for a widely varying excitation voltage from 2 mV to 9.6 V, and parts (c) and (f) show that the position on the film at which the critical displacement
is reached moves to the thin film edge as the voltage increases. All these experimental results from the 1D and 2D scans on different thin film samples and different resonance modes show an agreement for the premise that energy transfer between potential and kinetic occurs because of the internal resonance which is created by a non-linear vibration. When the vibration displacement is excited to its limit (critical displacement), there is some internal conversion of potential energy between the thin film bending and stretching.

5.4.3 Multilayer Thin Film

Figure 5.29: Amplitude response curves measured on the multilayer sample with both upward and downward frequency scan, undergoing a wide range of excitation voltage from 2 mV to 600 mV. (a) and (d) show the frequency scans at two excitation voltages. (b) and (e) are the 2D frequency scan for a set of excitation voltages, the colour scale represents the amplitude. (c) and (f) are the related FFTs of raw data from each point of the frequency scan.

The multilayer structure with layers of 3C-SiC, Si$_{1-y}$C$_y$ and Si$_{1-x}$B$_x$ was also studied using upward and downward frequency sweeping. The transducer excitation voltage was changed from 2 mV to 600 mV. The resonance amplitude curves for different driving frequencies and excitation voltages, and the maximum magnitude frequency in the FFTs from the raw data at each position, are plotted and shown in figure 5.29. The lateral dimensions and thickness of this square shaped sample are 2650 µm and 500 nm, respectively. The probe spot used was 200 µm in diameter to measure the amplitude frequency response. For this measurement, $\eta$ is about
0.45%.

The resonance amplitude curves achieved from the upward and downward frequency scans are the same when the vibration is in the linear regime (figure 5.29(a)) as expected. These are also very similar and well aligned when the vibration is in the non-linear regime (figure 5.29(d)). This result does not match the predictions of the classical theories, but shows the same behaviour observed in the 3C-SiC thin film when it was driven using an excitation voltage bigger than 2 V (figure 5.30(a)).

Figure 5.30: Resonance curves, relative phase difference and maximum magnitude frequency of FFTs at each scan point, measured on single layer 3C-SiC thin film with 240 nm thickness and 2055 × 2090 µm dimensions.

However, in figures 5.29(c) and (f), which show sweeps from low to high and high to low frequencies respectively, the main resonant frequency in the upward frequency sweeping increases with frequency rising until the amplitude jumps up through an energy transfer. For the downward frequency sweeping, only the highest order of response resonance has a shift.

Overall, the multilayer structure thin film has similar non-linear behaviour to the large lateral dimension single material layer thin films studied.

### 5.5 Summary

Table 5.1 gives sample information and size ratios with the two probing laser spot sizes.

- Non-linear behaviour with higher order harmonics are found in all samples.
Table 5.1: Ratio of the area between probe and samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Size (µm)</th>
<th>$A_{\text{sample}}$ (µm$^2$)</th>
<th>Probe (µm)</th>
<th>$A_{\text{probe}}$ (µm$^2$)</th>
<th>$\eta$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge</td>
<td>955×955</td>
<td>912025</td>
<td>200</td>
<td>31415.93</td>
<td>3.444634</td>
</tr>
<tr>
<td>Ge</td>
<td>2850×2855</td>
<td>8136750</td>
<td>200</td>
<td>31415.93</td>
<td>0.386099</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>1470×1470</td>
<td>2160900</td>
<td>200</td>
<td>31415.93</td>
<td>1.453835</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>2055×2090</td>
<td>4294950</td>
<td>30</td>
<td>706.8583</td>
<td>0.016458</td>
</tr>
<tr>
<td>Si$_3$N$_4$ (SiN$_1$)</td>
<td>310×310</td>
<td>96100</td>
<td>30</td>
<td>706.8583</td>
<td>0.735545</td>
</tr>
<tr>
<td>Si$_3$N$_4$ (SiN$_1$)</td>
<td>310×310</td>
<td>96100</td>
<td>200</td>
<td>31415.93</td>
<td>32.69087</td>
</tr>
<tr>
<td>Si$_3$N$_4$ (SiN$_2$)</td>
<td>315×315</td>
<td>99225</td>
<td>30</td>
<td>706.8583</td>
<td>0.712379</td>
</tr>
<tr>
<td>Si$_{1-x}$B$<em>x$-Si$</em>{1-y}$C$_y$-SiC</td>
<td>2650×2650</td>
<td>7022500</td>
<td>200</td>
<td>31415.93</td>
<td>0.447361</td>
</tr>
</tbody>
</table>

- This does not agree with the predictions from classical theories, e.g. Duffing.
- Measuring over the whole sample area shows a more Duffing-like behaviour.
- There are extra features observed which are related to internal resonance.
- Comparison of global and local measurement shows the effect of this internal resonance and confirms that the films vibrate with different frequencies in different regions. Using a smaller probe size can see more detail of the resonant structures that appear on the film.
- Most people only look at the global picture when studying these materials, but the local picture shows a resonance that has some internal structure, confirming the local resonance idea.
- This might open a new area for applications such as extending the emission wavelength of the laser source [194], enhancing sensitivity measurements [138, 141, 144], and possible use as bit storage or logic gates for nanomechanical computers [142, 195].
Chapter 6

Thermal Influence of the Laser Probe

The previous two chapters presented how the laser interferometer can be used to analyse mechanical properties and study oscillations of suspended thin films in the linear and non-linear regimes. However, there are some side effects to the measurements that should be considered carefully and also controlled when a measurement of the thin films is done with a laser. One of the most well known functions of a laser is as a good optical heat source. The laser heating effect on the thin film measurements must be accounted for. The temperature rise for an absorbing medium in air or vacuum has been discussed by M. Lax in 1977 [196]. Thus, this chapter is orientated as supplementary information for the experiments described in the previous two chapters, to discuss how the detector laser influences the experimental results.

In the first section of this chapter, a brief introduction to the heat transfer and the temperature rise induced by a continuous wave (CW) laser beam is given. Then, the following sections discuss the experimental observations, including the temperature distribution on suspended thin films measured using a thermal imaging camera, and the variations of mechanical properties measured as the local temperature changes. This chapter primarily considers the laser heating impact on characterisation of suspended thin film properties for the localized heating effect which is produced by the incident laser detection spot, but the general heating effect shown as a temperature rise over the whole sample is also discussed for a multi-layer structure (different materials for the thin film and the substrate).

The investigation was applied to two main suspended thin film materials on a silicon substrate: one is germanium, the other is cubic silicon carbide. The samples have different lateral dimensions and thicknesses. The materials have different optical
properties at the near infra-red laser wavelength of 1550 nm (absorption, transmission and reflection), and different thermal properties compared with each other and the silicon substrate. Further more, the thermal effect due to the optical and thermal properties of the thin films are not only dependent on the materials, but also rely on some further conditions such as thin film thickness, strain, and temperature. To consider fully all these parameters would require an extremely large number of different measurements, which is beyond the scope of this study. However, this chapter targets a general idea of how to control and to minimize the heating effect from the detector laser on the results obtained, rather than discussing these properties in detail, in order to give weight to the reliability of the measurements performed.

6.1 Laser Impacts

Heat can be transferred by three basic mechanisms: conduction, convection, and radiation, and is usually transferred by a combination of the three. Conduction is the transmission of heat through gases, liquids, and solids. Convection requires a movement of fluid. Radiation heat transfer does not require a material medium through which energy travels, but occurs due to the surface material properties including reflectivity, absorption, and transmission. Laser heating is a typical radiation heat transfer [197]. Laser technology has made a large contribution to scientific investigations and optical measurements, and is implemented in many areas of research and applications. Lasers used for ultrasound detection are characterised by a number of key optical properties, most of which play an important role in the interaction with ultrasonic dynamic vibrations. The four major laser optical properties are monochromaticity, coherence, directionality and high intensity, which are inter-related [198]. Coherence is an important property for an optical system for measuring ultrasonic waves because it describes the correlation of waves between one point and another in space or time. Intensity is another important property for laser reception of ultrasound, and is related to the sensitivity of a single-mode laser interferometer system through the signal to noise ratio for a fixed bandwidth [198] [119].

When a laser beam is incident on a sample surface, in general, some of the optical energy is absorbed and gives various effects such as strain changes, depending upon the nature of the sample and the frequency of the radiation, while the remainder is reflected or scattered from the surface or transmitted through the sample. The
incident laser beam energy $I_0$ can be described as

$$I_0 = I_r + I_a + I_t,$$

(6.1)

where $I_r$, $I_a$ and $I_t$ are the reflected intensity, the absorbed intensity and the transmitted intensity, respectively.

The absorbed energy mostly takes the form of heat, so that the laser irradiation induces a temperature rise at the surface over the beam spot, and the heat eventually becomes distributed throughout the sample because of the thermal conductivity. This is shown schematically in figure 6.1 (figure is not to scale). The laser beam used in most experiments was 200 $\mu$m in radius and the power from the probe laser is varied up to 2 W. Different laser powers are used for different thin film samples because there is a minimum requirement for the reflection intensity, to ensure a good signal to noise ratio.

Figure 6.1: Laser heating schematic diagram (not to scale).

Figure 6.2: Thermal images of sample heated and damaged by laser heating at different powers with a spot size of 200 $\mu$m diameter. (a) Suspended Ge thin film (in red box) with 1 $\mu$m thickness before applying a incident laser. (b) Thin film sample heated by 50 mW laser power. (c) Thin film sample destroyed by 350 mW laser power.
A number of different physical processes may take place when a sample surface is illuminated by a laser, such as thermal, mechanical, electrical, and metallurgical changes [199]. In this chapter, the discussion is restricted to the thermal impact. For different incident laser powers, the ultrasound detection laser can generate different thermal impacts. The laser induces temperature gradients across the sample which set up stress and strain fields via thermal expansion. These temperature changes will influence the measured residual stress, through extra thermoelastic stresses and strains, plastic deformation, a formation of cracks or even melting of the solid and damage to samples. Three thermal images taken before and after applying a laser are shown in figure [6.2] which displays an example of sample damage for a suspended thin film of Ge with 1 µm thickness by laser heating with a spot size of 200 µm in diameter. The position of the Ge thin film sample is indicated by the red box in figure [6.2]. In part (a), the grey colour in the red box confirms the thin film existed with a maximum temperature on the sample of 23.39 °C before applying an incident detector laser. The maximum temperature increased to 24.69 °C on the sample with a 50 mW incident laser shown in part (b), while in part (c) the whole grey region cannot be observed as the thin film has been destroyed by the laser when a power of 350 mW was used, and only a few bits of residual debris remain at the bottom left corner. Here, the substrate temperature (with laser heating) was raised to 25.96 °C with the 350 mW laser power. In order to avoid the occurrence of such events in measurements, all the detecting laser power levels were restricted to the non-destructive regime in the full measurements presented in chapters 4 and 5. However, the heating effect must still be considered to ensure the stress measured is a good approximation to the real value.

6.1.1 Heating by continuous wave (CW) Laser

A normally incident (along the $z$-axis) laser beam profile generally has a Gaussian intensity distribution described by $I_0 e^{(-r^2/w^2)}$, where $w$ is the beam radius, $r = \sqrt{x^2 + y^2}$ is the position relative to the beam centre, and $z = 0$ is at the sample surface. For a continuous wave laser, the heating effect near the sample surface is due to the surface absorption. The steady-state temperature rise, ignoring the heat dissipation, is given by $\Delta T = T - T_R$, where $T_R$ denotes the room temperature, and can be calculated by considering the incident laser power $P$ [196, 200]:

$$\Delta T(R, Z, W) = \frac{P(1 - R_c - R_l)}{2\sqrt{\pi}Kw} N(R, Z, W), \quad (6.2)$$
\[ N(R, Z, W) = \frac{W}{\sqrt{\pi}} \int_0^\infty J_0(\lambda R)e^{-\frac{1}{4}\lambda^2} \frac{We^{\lambda Z} - \lambda e^{WZ}}{W^2 - \lambda^2} d\lambda, \quad (6.3) \]

where \( R_c \) is the reflection coefficient, \( R_t \) is the transmission coefficient, and \( K \) is the thermal conductivity. The \( N(R, Z, W) \) is a numerical factor depending on these dimensionless variables \( R = r/w, Z = z/w, \) and \( W = \alpha w, \) where \( \alpha \) is the attenuation constant of the laser beam in the solid. \( J_0(x) \) is the zeroth order Bessel function, and \( \lambda \) is the wavelength.

Figure 6.3: Thermographic images of a suspended 3C-SiC sample (in the white colour box) with 210 nm thickness and 1541 \( \mu \)m side lengths. (a) Sample without incident laser, suspended thin film section in the dark blue frame in the image centre. (b) Sample heated by a 400 mW power. (c) Incident laser power increased to 1400 mW.

Figure 6.3 illustrates three thermographic images which have been taken for a 3C-SiC sample with 210 nm thickness and 1541 \( \mu \)m lateral dimensions. The whole sample, including the substrate, is framed by the dashed white line box and the suspended thin film is at the centre. Figure 6.3 (a) shows the sample without any incident laser. The blue frame on the sample surface is the boundary between the suspended section and the solid region. The darker blue colour means it has a lower temperature, which could be caused by the substrate fully contacting the heat sink formed by the equipment. Parts (b) and (c) show the temperature conditions when the sample is undergoing the CW laser heating with powers of 400 mW and 1400 mW, respectively. The Gaussian distribution of the temperature gradients on the sample at the detection region is much clearer at high laser power, where a larger temperature difference is generated between the lower spot position and the rest of the film.

Figure 6.4 shows three sets of analysed data measured on two thin film samples of suspended Ge and 3C-SiC using a thermal imaging camera. Two of the images obtained using this camera are shown in figures 6.2 and 6.3. The first set of data in figure 6.4 (a) and (b) shows the relationship between the increased temperature \( (\Delta T) \) and position cross the horizontal centre line of the sample level, showing
Figure 6.4: Temperature distribution with different laser powers at different locations on (a) 3C-SiC and (b) Ge. The value of FWHM of each distribution with different laser power is in (c) for 3C-SiC and (d) for Ge. The difference of temperature between laser spot centre and the boundary of the suspended region is in (e) for 3C-SiC and (f) for Ge. Data is taken from the therographic images shown in figure 6.2 (Ge) and figure 6.3 (3C-SiC).
the laser spot. A distribution of temperature across the sample is shown, with a maximum temperature observed at the centre of the laser spot. The thin film shows a Gaussian distribution for the temperature gradients. As expected, the strained Ge material thin film can absorb more power from the 1550 nm wavelength laser than a strained 3C-SiC thin film. The Ge sample was destroyed for a laser power above 300 mW, which gave a maximum temperature of 56.8 °C at the laser spot centre. In contrast, the 3C-SiC thin film survived with a 2 W incident laser power with a maximum temperature of 59.6 °C and achieved the temperature of 56.8 °C at the spot centre when the power was 1.6 W. A smaller temperature rise on the 3C-SiC with a higher laser power may be caused by the higher transmission for this sample with the 1550 nm wavelength laser [201, 202, 203].

The second set of data in figure 6.4(c) and (d) is the value of the full width half maximum (FWHM) of the temperature profile of the thin film for different incident laser powers, found by fitting each of the distributions of temperature in parts (a) and (b) to a Gaussian function. Both of the FWHM of the temperature distributions do not simply follow a linear growth with increasing laser power. In addition, the numerical values of the FWHM for these two materials is not as expected 3C-SiC has a higher thermal conductivity (3-4 W cm$^{-1}$ K$^{-1}$) than Ge (0.6 W cm$^{-1}$ K$^{-1}$), found at a temperature of 300 K. This means, theoretically, that the value of the FWHM obtained from the temperature distribution of 3C-SiC should be bigger than the value found from the Ge sample, but the measurement results exhibit a reversed relationship. This unexpected result could be caused by the thickness difference between these two samples, which influences their optical properties; the 3C-SiC (210 nm thickness) thin film is about five times thinner than the Ge (1 µm) sample. For a similar thickness thin film to the Ge, the 3C-SiC needed a higher laser power to achieve the same reflection intensity as a Ge thin film as the 3C-SiC thin film has a smaller absorption and higher transmission than Ge for the near-infrared laser used.

The final group of data in figure 6.4(e) and (f) is the temperature difference between the maximum temperature on the suspended region (laser spot centre) and the temperature on a boundary of the suspended thin film and substrate region ($\delta T$). The relationship between the temperature difference ($\delta T$) and incident laser power shows an exponential growth with increasing laser power for both materials, which shows a consistency of thermal impact from laser heating for suspended thin film structures with the theoretical predictions.

The thermographic images and measured temperature data were obtained at room pressure with a room temperature of about 23 °C controlled using air conditioning.
and samples without any cover. Thus, the heat can easily be dissipated into the air. However, most of the measurements done in the previous chapters were in a vacuum chamber in a low-pressure environment. Thus, a higher temperature can be achieved on the sample as one heat dissipation mechanism is removed. However, thermal imaging is only possible outside of the vacuum chamber.

6.1.2 Thermal Stress: Local and Overall

The rise in temperature of the surface region of the sample at the incident laser position shown in figures 6.4 (a) and (b) is accompanied by a thermal expansion, which in turn generates stress and strain within the sample as it thermoelectrically deforms. As a result of the temperature change $\Delta T$, there is a strain given by $
abla T = \alpha \Delta T$, where $\alpha$ is the coefficient of thermal expansion of the thin film. Assuming that the sample material is isotropic, the expansion can be considered as equivalent to the insertion of a small extra volume of material at the heating position of volume $\delta V$. This produces the strains ($\varepsilon$) locally:

$$
\varepsilon_{xx} = \varepsilon_{yy} = \varepsilon_{zz} = \frac{1}{3} \frac{\delta V}{V} = \alpha \Delta T.
$$

(6.4)

It also generates the stress ($\sigma$) field locally as a hydrostatic pressure, which can be written as

$$
\sigma_{xx} = \sigma_{yy} = \sigma_{zz} = B \frac{\delta V}{V},
$$

(6.5)

where $B$ is the bulk modulus of elasticity.

A compression stress is generated by the localised irradiation on the thin film and along an outward direction from the centre of the laser spot, because the temperature gradient is negative from the centre to the edge. Thus, for a membrane under tension the measured tensile residual stress would be smaller than its initial value when the thin film is without a incident laser. An obvious feature for this thermal effect was demonstrated as a shift to the measured resonant frequencies in the previous measurements. Experimental results will be shown in the next sections.

Thermal stresses in elastic multilayer systems should be considered in this study, because the samples are bilayer systems formed of substrate (Si) and the layer of suspended material. A general equation was found by Hsueh and Evans to describe the strain distribution in a multilayer system [204, 205]. The changes in thermal stress with temperature change $\Delta T$ can most simply be described by

$$
\Delta \sigma_{th} = \frac{E}{1 - \nu} \frac{\alpha_f(T) - \alpha_s(T)}{\alpha_s(T)} \Delta T,
$$

(6.6)
where \( E \) is Young’s modulus, \( \nu \) is Poisson’s ratio and the subscripts \( f \) and \( s \) denote the thin film layer and the substrate, respectively.

According to the basic mechanisms of heat transfer, the heat can be transferred within the sample primarily by conduction. So that, when the thin film temperature is raised by absorbing radiation, the heat will also be conducted to the substrate during the measurement process.

The total thermal effect caused by the laser heating is

\[
\sigma_{total,thermal} = \sigma_{local} + \sigma_{multilayer}.
\] (6.7)

In a real measurement, it is not easy to distinguish the impact of the two components of the thermal stress separately, especially when these two components have an opposite effect on the sample. However, by careful consideration of experimental conditions, for example applying the laser probe for a short or a long time, some separation can be made. The following sections describe such measurements.

### 6.2 Applying Different Laser Powers

Figure 6.5 illustrates the thermal effect generated by the localised incident laser (local heating). The measured resonant frequency with different incident laser powers for a 3C-SiC (Sample 1) thin film with 500 nm thickness and \( 1455 \times 1565 \mu m \) lateral dimensions is shown in figures 6.5(a) and (b) for resonant modes 1:1 and 1:3, respectively. The incident laser power was varied from 20 mW to 350 mW and the measurements were done in a vacuum chamber for a microbar pressure environment. According to equations 2.40 and 6.6, it is expected that one will obtain higher resonant frequencies under higher residual stress. The higher thin film temperature should create higher thermal stress in the 3C-SiC thin film which was deposited on a Si substrate. However, the resonant frequencies of both modes are shifted downwards, in disagreement with as the theoretical expectation, as power increased. Figure 6.5 (c) shows the relations between the percentage changes in resonant frequency and incident laser powers. There is the same gradient for both modes, showing that this is due to a change in the stress in the thin film, which is the same for both modes. The Q-factor was also measured for each mode as shown in figure 6.5 (d). The Q-factor value has a strong oscillation before the laser power reaches 160-200 mW, but, overall the changing trend is that the Q-factor becomes smaller as the laser heating is increased.

The same measurement was done on another 3C-SiC thin film sample, which had a larger thickness of 600 nm with \( 1654 \times 1726 \mu m \) lateral dimensions, but over a
smaller laser power range from 30 mW to 100 mW. The measurement result is shown in figure 6.6. In part (a), it shows the relationship between the percentage change of the resonant frequency and incident laser power. The three resonant modes, 1:1, 1:3 and 3:1, all show the same gradient for the resonant frequency shift, as with the previous sample. The related Q-factor values as shown in part (b), with no clear change observed, as with the previous sample at lower laser powers.

The gradients of the resonant frequency shifts obtained from the measurements on these two 3C-SiC samples were $-0.00164$ for the sample with 500 nm thickness, and $-0.00924$ for the sample with 600 nm thickness. The sample area of the thicker thin film is more than 1.25 times larger than the thinner thin film. The thicker sample showed a stronger thermal effect than the thinner sample, even though its larger area can potentially dissipate more thermal energy. This means that the thicker thin film absorbed more energy from the 1550 nm wavelength infrared laser than the thinner one. The absorption coefficient for the 3C-SiC with 1550 nm wavelength is not currently recorded in the literature, as most research results are

![Figure 6.5: Measurement results affected by localised heating for a 3C-SiC thin film with 500 nm thickness and 1455 \times 1565 \mu m lateral dimensions at microbar pressure. (a) and (b) The resonant frequency shifts for the 1:1 mode and 1:3 modes. (c) The percentage change in the resonant frequencies for 1:1 mode and 1:3 mode. (d) Q-factors for these two resonant modes.](image)
Figure 6.6: Thermal influence measured on a sample of 3C-SiC with 600 nm thickness and 1654 × 1726 µm lateral dimensions at microbar pressure. (a) Percentage change in resonant frequencies for 1:1, 1:3 and 3:1 modes. (b) Q-factor obtained from each measurement.

for wavelengths under 1000 nm. However, the different thickness of the thin films has varying percentage of light transmission as shown in references [201, 203]. There is not enough data to fully give an accurate picture for an exact prediction of stress release, but it shows that the thermal effect has some size dependence, related to the optical properties, thin film sizes and strain. The shift gradient is related with the material mechanical properties from Young’s modulus and Poisson’s ratio as shown in equation [6.6], but absorption within the material plays an essential part.

### 6.3 Thermal Effect of Heating Time

The sample temperature not only rises locally due to the localised incident laser but would also over time be raised for the whole sample (including substrate) because of the thermal conductivity through the sample. The sample also loses some heat energy at the same time as it is heated because of radiation into the vacuum chamber and conduction through the sample holder. Over time, the temperature will be built up until it reaches a thermal equilibrium state.

The measurement results in this section are divided into two subsections, looking at two different materials to investigate the effect of material properties such as the coefficient of thermal expansion (CTE), considered in equation [6.6]. One type of sample is a Ge suspended thin film with a Si substrate, which will generate compressive stress on the thin film when cooling from the growth temperature to room temperature due to the thermal expansion because Ge has a larger CTE than Si. The other type of sample is a 3C-SiC suspended thin film with a Si substrate, which has the opposite thermal effect to Ge as the sample is cooled from the growth
temperature because 3C-SiC has a smaller CTE than Si, with a turning point in measurements of the CTE at a temperature of around 220 °C. Values of thermal expansion coefficient of thin film and substrate material are given in table 6.1 [44].

<table>
<thead>
<tr>
<th>Material</th>
<th>CTE (10^{-6} , ^\circ\text{C}^{-1})</th>
<th>Temp. range (^\circ\text{C})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>(3.084 + 0.00196 , T)</td>
<td>20 to 700</td>
</tr>
<tr>
<td>Ge</td>
<td>(6.05 + 0.0036 , T - 3.5 \times 10^{-7} , T^2)</td>
<td>20 to 810</td>
</tr>
<tr>
<td>3C-SiC</td>
<td>(1.6 + 0.0042 , (T+273) - 5.9 \times 10^{-7} , (T+273)^2)</td>
<td>20 to 900</td>
</tr>
</tbody>
</table>

Table 6.1: Representative values of thermal expansion coefficient of materials used as substrate and thin film (the temperature unit is \(^\circ\text{C}\)) [44].

3C-SiC

Figure 6.7 (a) shows the temperature growth as time passed on a square shaped 3C-SiC (Sample 2) thin film with 500 nm thickness and 1600 \(\mu\text{m}\) side length, at microbar pressure. Five different laser powers with a 10 mW difference, from 50 mW to 90 mW, were applied during this measurement. The temperature was measured by a platinum thermocouple at the edge of the boundary between suspended section and substrate. The experimental set up was shown in chapter 3. The temperature can be increased far higher than the sample in an open space at room pressure (figure 6.4), with the edge temperature reaching about 31 \(^\circ\text{C}\) with a 50 mW laser power after thermal equilibrium is reached. Thus, assuming the centre position of the detecting laser would have a higher temperature than the position at a boundary edge on the substrate side, a higher temperature is reached at the centre. The temperature grows rapidly as the laser is turned on, with a reduction in the growth speed at later times, until the temperature achieves equilibrium. A higher laser power has a higher equilibrium temperature and larger temperature growth gradient, and also needs a longer time to reach the equilibrium state.

The relationship between temperature and fundamental resonant frequency for five different laser powers is shown in figure 6.7 (b). With the varied incident laser power and heating time, the temperature changes over time would involve two thermal processes. The temperature gradient between the centre of the laser spot and the boundary will become larger with a higher power incident laser. Thus, a stronger local heating effect can be generated for higher powers leading to a larger resonant frequency shift downwards, due to the tensile stress in the thin film prior to heating, and local relaxation around the heating spot. However, in the meantime, the heat is also diffused out to the whole sample by conduction, then the higher sample temperature creates more tensile stress for the 3C-SiC thin film on the Si substrate.
Figure 6.7: The temperature and the related fundamental resonant frequency results obtained from a square shaped 3C-SiC thin film with 500 nm thickness and 1600 µm side length at microbar pressure. (a) The temperature rise over time for different fixed laser powers. (b) The fundamental resonant frequency as a function of temperature. (c) The percentage change of the fundamental resonant frequency at equilibrium compared to the value measured with 50 mW.
(at T < 220 °C), and the resonant frequency will shift upwards.

Figure 6.8: Square shaped 3C-SiC thin film with 500 nm thickness and 1600 µm side length at microbar pressure. (a) 2D image of the amplitude-frequency response curves measured using a 50 mW laser power around its fundamental frequency range. Colour scale represents the amplitude. (b) Frequency shift over time until an equilibrium state is reached. (c) Q-factor reducing as time passed until an equilibrium state is reached.

The percentage change in the fundamental resonant frequency versus the incident laser power is shown in figure 6.7 (c) with a gradient of $-0.00555$ (3C-SiC sample 2). Compared to the gradient found from the 3C-SiC sample (Sample 1) in section 6.2 (gradient is $-0.00164$), both thin films have the same thickness, but the gradient of frequency shift is about 3.38 times different between samples ($\frac{\text{gradient}_{\text{sample 2}}}{\text{gradient}_{\text{sample 1}}}$). The dimensional size difference between these two samples is about 1.12 times ($\frac{A_{\text{sample 2}}}{A_{\text{sample 1}}}$).

From the resonant frequencies, the residual stress is about 0.2947 GPa for sample 1 and 0.0932 GPa for sample 2, thus the ratio of stresses is about 0.316 ($\frac{\sigma_{\text{sample 2}}}{\sigma_{\text{sample 1}}}$). Based on the three values, there is coincidently a relationship between them as

$$\frac{\sigma_{\text{sample 2}}}{\sigma_{\text{sample 1}}} \sim \frac{A_{\text{sample 2}}/A_{\text{sample 1}}}{\text{gradient}_{\text{sample 2}}/\text{gradient}_{\text{sample 1}}}.$$  \hspace{1cm} (6.8)

According to equation 6.6, the gradient of stress shift (expressed as a frequency shift)
not only depends on the different thermal expansion coefficient between substrate and thin film, but is also related with the mechanical properties of the thin film. Converting equation 6.9 and expressing as

\[
\frac{\sigma_{\text{sample 1}} \times \text{gradient}_{\text{sample 1}}}{A_{\text{sample 1}}} \simeq \frac{\sigma_{\text{sample 2}} \times \text{gradient}_{\text{sample 2}}}{A_{\text{sample 2}}},
\]

allows easier observing of the relationship between these two samples. As both samples are made from the same wafer with the same thickness, it could be assumed that they have the same mechanical properties. The relationship between the stress and the gradient support this suggestion, but further work is required to confirm it.

More measurement results are shown in figure 6.8(a). A 2D image of the amplitude frequency response curves can be observed for real-time monitoring during the measurement, as shown in figure 6.8(a) with a 50 mW laser power. The colour represents the amplitude, and it clearly shows the resonant frequency shift to a higher value. The resonant frequency and related Q-factor can be obtained by fitting the amplitude curves with a Lorentzian or a Gaussian function as shown in figure 6.8(b) and (c). The Q-factor reduced to a lower value because of increased sample temperature. The higher temperature might cause a softening boundary which intensified energy dissipation due to the thermoelastic effect.

Ge

The same measurement was repeated on a square shaped Ge thin film sample, which had a 700 nm thickness and 955 \(\mu\)m side length. The sample temperature was increased to an equilibrium state after a certain laser heating time with 50 mW laser power, as shown in figure 6.9(a). The temperature reached a steady state in a very short period compared with the heat up time used for the 3C-SiC sample shown in figure 6.7(a). However, the sample temperature was only raised by about 0.63 \(\degree\)C at the edge of the Ge sample, which is far smaller than the temperature change on the 3C-SiC sample with the 50 mW incident laser. In addition, the fundamental resonant frequency shifted downwards to a lower frequency for the Ge sample, which is opposite to the results observed on the 3C-SiC samples, as shown in figure 6.9(b). The downwards frequency shift agreed well with the theoretical predictions. The Q-factor has the same variation trend with a lower Q-factor value at higher temperatures as shown in figure 6.9(c). More details of the frequency shift and Q-factor changes with time passing are shown in figure 6.10.
Figure 6.9: Laser thermal effect on square shaped Ge thin film with 700 nm thickness and 955 \( \mu \)m side length, undergoing a 50 mW incident detecting laser at microbar pressure. (a) Temperature variation with time. (b) Fundamental resonant frequency obtained at a different temperature which related to the heating time. (c) Related resonant Q-factors versus the sample temperature.
Figure 6.10: The relationship between the fundamental resonant frequency and related Q-factors and laser heating time for Ge sample (same sample as figure 6.9).
(a) 2D image of the amplitude frequency response curves measured using a 50 mW laser power around its fundamental frequency range. Colour scale represents the amplitude. (b) Frequency shift against time. (c) Q-factor versus the laser heating time.
6.4 Effect of Laser Position: Sample Scans

Static thermal effects can be induced when the laser is placed at a fixed position, as observed in the previous sections from the frequency scans. The thermal effects included two impact factors, localized and uniform heating, and the gradient of frequency change against laser power is sample and size dependent. Thus, changing the laser spot location on the thin film might also vary the resonant frequencies as a different thermal field is generated in the system. For earlier measurements, accurate analysis needs identification of the resonant mode, which requires a full 2D scan to obtain the resonant mode image, therefore, the laser will be moved across all the areas on the suspended thin film. A dynamic thermal field would be generated during the 2D scan procedure. Most of the earlier experiments used a low laser power but the effect must still be understood.

To examine the assumption described above, a 1D frequency scan was done along the mid-line of the thin film to obtain the resonant frequencies. This scan did a frequency scan at every point along the line and found the resonant frequency by where the amplitude was largest. Because the thin film shape has axial symmetry, the results from the 1D scan can be used as a reference to predict the full thermal influence in 2D. The dimensional frequency scan was again applied on two materials.

3C-SiC

Figure 6.11 shows the dynamic thermal influence during the 1D scanning measurements. The scanning investigation was done twice to prove the consistency of measurement results. The sample was a square shaped 3C-SiC thin film with a 500 nm thickness and 1610 µm side length at microbar pressure, using a 50 mW power of detecting laser. In part (a), the thin film displacement profile is shown along a mid-line which crossed the whole suspended area. The displacement profile was obtained by selecting the maximum amplitude from the frequency scan measurement at each scanning position, with the driving frequency range around its fundamental resonance. The displacement profile found from this measurement has the same shape as the cross-section of the fundamental resonant mode.

The resonant frequency at each scanning position along the mid-line 1D scan can be found by fitting the amplitude curves from the frequency scan measurement with a Lorentz function and choosing the peak, with results shown in figure 6.11(b). The resonant frequencies formed a near-symmetric valley shape along the mid-line across the suspended thin film. In parts (c) and (d) the related Q-factors and stresses are shown, calculated from the resonant frequencies by using equations 2.40 and 2.47.
Figure 6.11: Results measured from a square shaped 3C-SiC thin film with 500 nm thickness and 1610 µm side length, at microbar pressure. (a) 1D fundamental resonant mode shape along with a mid-line cross the suspended thin film from the substrate boundary. (b) Exact resonant frequency measured at each scanning position. (c) Q-factor found at the resonant frequencies. (d) Analysed stress from each resonant frequency.
Most of the values of the Q-factors are maintained in a region between 1100 and 1400, and there are no clear shifts with the frequency shift. However, the calculated average stresses are directly related with the resonant frequency, thus they have the same shift trend as the shifted frequencies. The fully mapped fundamental resonant frequency shift for a 2D scan is shown in figure 6.12. The shift profile has a concave cone shape.

Figure 6.12: 2D scan image of fundamental resonant frequency, measured on the 3C-SiC thin film with 500 nm thickness and 1610 µm side length using a detecting laser with 50 mW power at microbar pressure. Colour scale represents the resonant frequency.

According to the relationship between temperature gradient and strength of frequency shifts found in section 6.2, a higher temperature gradient can generate a larger frequency shift. Thus, the strongest temperature gradient was created when the detecting laser spot was placed at the centre of the suspended thin film, and the effect gradually became weaker when the spot was closer to the boundary. Because the size of the substrate is larger than the thin film, it acts like a heat sink, absorbing an amount of thermal energy until it has the same temperature as the thin film.

The resonant mode shapes from the 2D scan measurement can be affected by the choice of frequency to use, for example applying the different resonant frequencies found at different positions on the thin film will give a different shape. An example is shown in figure 6.13 with four resonant frequencies identified in the 2D scan measurement at different positions. The 2D scan measurement used four selected excitation frequencies, measured from the thin film centre to the edge with
Figure 6.13: The resonant modes from 2D scan measurement for square shaped 3C-SiC thin film with 500 nm thickness and 1610 μm side length at microbar pressure with a 50 mW detecting laser power. (a) Driving frequency is 67.41 kHz, found from the thin film center. (b), (c) and (d) were excited with frequencies of 67.49 kHz, 67.57 kHz and 67.65 kHz, which were found between thin film centre and the edge, respectively.
Figure 6.14: 1D scanning measurement repeated on the 3C-SiC thin film with 500 nm thickness and 1610 µm side length at room pressure. (a) 1D mode shape. (b) Resonant frequencies found from each scanning position. (c) Related Q-factor with resonant frequencies at each scanning position.
frequency values of 67.41 kHz (at the centre), 67.49 kHz, 67.57 kHz and 67.65 kHz (near the edge), respectively. Figure 6.13 (a) shows the 2D scan measurement using the frequency measured at the thin film center, thus, the mode shape is the same as the fundamental resonant mode with a large amplitude at the centre. When the vibration driving frequency was shifted to the values found at other positions, the mode shape deformed to a doughnut for the fundamental mode shape. The maximum amplitude position was shifted to the position where the resonant frequency used was measured. The reason for the mode shape deformation is due to the detecting laser moving, with the dynamic thermal field changing the material strain and affecting the wave propagation. The thermal effect exhibited a stronger impact in a vacuum system than in an open environment at atmospheric pressure because of the smaller heat dissipation in the vacuum. A comparison measurement was done at atmospheric pressure on the 3C-SiC thin film, with the investigation results are shown in figure 6.14 with the same laser conditions. Part (a) shows the measured 1D fundamental mode profile, which appears smoother than the mode shape shown in figure 6.11 (a). The measured resonant frequencies along the scanning line do not show a clear shift, and remain in a small region of width ±0.05 kHz (<< the value of FWHM obtained from the f-scan) as shown in figure 6.14 (b). As expected, the Q-factor obtained at atmospheric pressure is about 70 times smaller than the values achieved from the measurements at microbar pressure. Thus, the 2D mode images are less affected at a higher pressure as there is a weaker dynamic thermal effect.

Ge

The same dimensional frequency scan under the same experimental conditions was repeated on a square shaped Ge thin film (700 nm thickness, 955 µm side length) around its fundamental resonance (see figure 6.15) and over a frequency range including 1:3 and 3:1 resonant modes (see figure 6.16). The results on the Ge thin film around the fundamental resonance show the same frequency shift behaviour as observed on the 3C-SiC thin film. An unexpected feature was detected when the scanning frequency range was around the 1:3 and 3:1 modes as shown in figure 6.16. There is a splitting and joining of the 1:3 and 3:1 modes occurring at the peak frequency of resonance from the frequency scan. When the scanning point was placed at the centre position of the thin film, the amplitude response curve only shows a single peak. But, when the scanning position moved away from the centre, the single peak begins to split into two. This phenomenon might be caused by anisotropy in the material, and the magnitude of the properties
Figure 6.15: 1D frequency scan measured at microbar pressure with a 50 mW detecting laser power on the square shape Ge thin film with 700 nm thickness and 955 µm side length. Scanned frequency range is around its fundamental resonance. (a) 2D image of amplitude frequency response curves along the horizontal mid-line (x-axis). (b) Displacement profile along the scanning line. (c) and (d) are the related resonant frequencies and Q-factors, which are obtained by analysis of the amplitude curves in (a).
along the different orientations could be changed by the dynamic thermal field. Thus, two different sets of measurements have been designed to investigate the assumption which included varying the laser powers at the microbar pressure (see figure 6.17) and increasing the pressure to at 160 millibar with two different laser powers (see figure 6.18).

Figure 6.16: Ge sample measured at microbar pressure with 50 mW laser power in the scanning frequency range at 1:3 and 3:1 modes. (a) 2D image of amplitude frequency response curves. (b) and (c) are the amplitude frequency response curves measured at the thin film center and edge, respectively.

Figure 6.17 shows the scanning results under microbar pressure and repeating the 1D frequency scan around the 1:3 and 3:1 resonant frequency range using different laser powers at the same position. Firstly, the resonant frequency has been shifted more by a higher laser power as predicted by the theory. The difference of resonant frequencies between the split peaks has a larger value for a higher laser power, as shown in figure 6.17 (c). These results suggest that a higher thermal gradient can induce and amplify the magnitude of anisotropy properties inside of a material.

Figure 6.18 shows the resonant frequencies for 1:3 and 3:1 modes from the 1D frequency scan with 50 mW and 70 mW laser powers, under two different pressures.
Figure 6.17: Ge sample measured at microbar pressure with varied laser power from 30 mW to 100 mW in the scanning frequency range for 1:3 and 3:1 modes. (a) and (b) are the first and second peak values obtained from the frequency scan measurement along a mid-line across the thin film. (c) The frequency difference between the two peaks in (a) and (b).
of microbar and 160 millibar, respectively. These results agree that, using the same heating source in a lower pressure would induce stronger thermal effects due to a smaller thermal loss, as a larger frequency shift occurred with position. Meanwhile, there is a subtracting effect for a higher laser power under a higher pressure, as shown in figure 6.18 (c) by a frequency difference between the two peaks and the split of the 1:3 and 3:1 modes, for two measurements done with a 50 mW laser power at microbar pressure, or with a 70 mW laser power at 160 millibar. Thus, the same changing gradient possibly suggests that there is the same thermal influence induced under the two different conditions.

![Graphs of resonant frequency vs. scanning position for different laser powers and pressures.](image)

Figure 6.18: Ge sample measured at two different pressures with two different laser powers. (a) and (b) are the split peak values found in the resonant modes of 1:3 and 3:1. (c) is the percentage change in the 1st peak resonant frequencies.

However, the measurement results of the frequency difference between two peaks shown in figure 6.19 disagrees with this suggestion because of the features obtained from different pressures. The unexpected features were observed at different pressures, which suggested the anisotropy properties might have some relation with the pressure. Therefore, the thermal influence should be carefully considered in material characterisation, especially when the measurement involves a pressure variation.
6.5 Summary

This chapter focuses on studying the thermal impact from the incident probe laser. Two materials (Ge and 3C-SiC) as thin films on a Si substrate were studied to explain the change in the vibration resonance frequency and the deformation of the 2D mode image which was observed and presented in chapter 4.

- The work shows that the thermal influence introduced by the probe laser can impact measurements, shifting the frequency and altering the 2D mode image.
- The thermal influence is dependent on the input laser, laser heating time, and system vacuum level.
- The thermal influence is dependent on the input laser, laser heating time, and system vacuum level.

Figure 6.19: The frequency difference between two peaks shown in figure 6.18.
Chapter 7

A Non-Destructive Method for Measuring Sub-micron Scale Cracks

As the previous chapter discussed, solid thin films have been used in a wide variety of engineering systems, and are commonly used in development of micro-electro-mechanical systems (MEMS) and nano-electro-mechanical systems (NEMS) [25, 206, 207]. The suspended square thin film is a simple MEMS structure and can be used in pressure sensors, or as a platform for integrating devices for applications such as near-infrared photo-detectors, flow sensors, photonic modulators, and lasers [26, 80, 208, 209, 210]. The use of tensile strained membranes, such as Ge on Si, can change the electronic or optoelectronic properties by reducing the energy band gap [24, 211]. However, the residual stress in thin films, especially within a multilayer structure, is effectively temperature dependent. The residual stress in thin films can induce mechanical deformation, which can subsequently lead to damage or failure, influencing the properties of functional devices formed using thin films. Therefore, knowledge of the mechanical properties and stability of the thin film structures at their working temperatures are important. Key threats to the stability of the structures are crack initiation and growth, and delamination between layers, which influence the mechanical reliability. These failure or delamination processes are related to variations in the mechanical interactions between the substrate and thin film layers. In turn, these variations are linked to the residual stress in the thin film, through processes such as thermal relaxation, in-plane stretching or contraction during operation in an environment subject to vibrational noise, and substrate curvature or plastic yielding of the thin films [44]. Hence a measurement
of the residual stress can be used to determine the risks for cracking and delamination [212]. Chapter 6 discussed and focused on heating induced by the probe laser. In this chapter, the thermal stress of thin films was varied and controlled using a self-built thermal cycling system which controlled the whole thin film temperature. The experimental details of thermal cycling were described in chapter 3. One 3C-SiC and two Ge suspended thin films on the silicon substrate frame were investigated by measuring the resonant frequencies and mode patterns in the frequency range between 50 kHz to 500 kHz, with thermal cycling in the temperature range from 23 to 105 °C. The temperature dependence of mechanical properties, including residual stress and Q-factor, were studied at microbar pressure and at atmospheric pressure. The experimental results of thermal stress percentage changes with changing temperature are obtained from Ge thin film samples, measured at low pressure, and are compared with finite element modelling (FEM) results. The temperature dependence of the stress allows identification of the thermal contribution to the stress by comparison with the model of the thermal expansion of the thin film and substrate for this geometry. The change in biaxial stress and Q-factor with temperature variation agrees well with the FEM for a perfect thin film, as predicted. However, in this study, cracks were generated on two samples (one Ge, one 3C-SiC), through large amplitude vibration and thermal cycling. The 3C-SiC thin film was destroyed during the investigation. These cracks give an imperfect thin film. Crack initiation and growth are key issues when it comes to the mechanical reliability of microelectronic devices and MEMS, especially when a system is fabricated using a suspended thin film as a platform. A scanning electron microscope (SEM) measurements with temperature variation were applied to analyse the effects of sub-micrometer or nanometer cracks on these samples, after an unexpected behaviour was observed in the cracked Ge thin film, which showed the Q-factor increasing with temperature rises at low pressure, but showed the opposite behaviour at room pressure. This phenomenon was caused by crack healing with thermal expansion when temperature was increased at low pressure, but the same expansion size requires higher input temperature at room pressure because of the thermal losses. This behaviour is discussed in this chapter.

The total residual stress $\sigma_T$ at an arbitrary temperature is governed by two parts: an intrinsic component $\sigma_i$ which is generated by the lattice mismatch between membrane and substrate during growth [44, 212], and a temperature dependent component $\sigma_{th}$ which is determined by the coefficients of thermal expansion (CTE) of the
membrane and substrate. The total residual stress is given by

\[ \sigma_T = \sigma_{th} + \sigma_i, \]  

(7.1)

and the thermal component can be approximated as

\[ \sigma_{th} = \frac{E}{1 - \nu} (\alpha_f(T) - \alpha_s(T))(T_0 - T), \]  

(7.2)

where \( E \) is the Young’s modulus and \( \nu \) is the Poisson’s ratio of the thin film material, and \( \alpha_f \) and \( \alpha_s \) are the CTE of thin film and substrate [213]. \( T_0 \) is the growth temperature of the thin film. The thermal component is taken to be zero during growth, then changes due to contraction or expansion of the thin film and substrate on heating or cooling. A positive value of \( \sigma \) represents tensile stress, while negative is compressive stress [213]. This was used to model the expansion behaviour for each sample, using CTEs for each material taken from the literature.

7.1 3C-SiC Thin Film with Cracks

According to equation 7.2 and the values of CTEs in table 6.1, the thermal stress on the 3C-SiC thin film with a Si substrate has a turning point, as the tensile stress becomes compressive stress, at a temperature of around 212 °C. This phenomenon of stress direction overturn is caused by the CTE temperature dependent behaviour, as the CTE of 3C-SiC becomes larger than that of Si after the turning temperature. However, the reversal trend of thermal stress was found in the experiments presented here when the 3C-SiC thin film sample was heated past 70 °C (see figure 7.1(a) and (b)).

Figure 7.1 (a) shows the frequency scans with increasing temperature, plotted as a 2D map with a colour scale which represents the magnitude of displacement response. Parts (b) to (d) are the analysed data from part (a), which include the fundamental resonant frequency, average residual stress, Q-factor, and maximum amplitude of the vibrations, respectively. From the temperature frequency scan map and stress change trend (in figure 7.1 (a) and (b)), the thermal stress turn at around 70 °C can be observed clearly, but it is not at the expected temperature. Also, the relationship between Q-factor and temperature was not as predicted (in figure 7.1 (c)). In previous studies, the Q-factor should become larger as the resonant frequency increases (or the film has a larger tensile stress), although this effect should be small for the frequency changes shown here. Additionally, at increased temperature the Q-factor in these experiments should become smaller because of
Figure 7.1: Results of thermal cycle frequency scan around the fundamental resonance of 3C-SiC sample with 500 nm thickness and 1600 µm lateral dimensions, at $10^{-3}$ mbar. (a) 2D colour map of frequency amplitude response, the colour represents the displacement. (b) fundamental resonant frequency and average residual stress with temperature variation. (c) and (d) show the Q-factor and the vibration amplitude at resonance with different temperature.

...the coupling between sample and transducer softening at temperatures greater than 40 °C. The Q-factor in these experiments was raised sharply in the range of temperature from 36 °C to 45 °C. This anomaly behaviour was also reflected in the amplitude response (in figure 7.1(d)). The maximum vibration amplitude showed a few big jumps when the sample was heated from 75 to 90 °C. After this temperature the thin film was destroyed.

The behaviour of the stress and Q-factor was indicative of changes to the sample during the measurements as the temperature was swept. The turn in residual stress could have indicated the onset of defects releasing stress, which led to breakage. Q-factor changes compared to predictions were also seen in a Ge sample which did not break but exhibited damage, and are discussed in the next section.
7.2 Ge Thin Films

7.2.1 Ge Thin Film with Cracks

Measurements were repeated for a Ge thin film sample (Ge\_Sample\_1) with 2 \( \mu \text{m} \) thickness and 1370 \( \mu \text{m} \) lateral dimensions, which had undergone high amplitude oscillation and thermal cycling to simulate extended use.

Figure 7.2: First heating cycle for Ge\_Sample\_1. (a) shows the percentage change in stress, (b) shows the Q-factor with changing temperature, (c) shows the vibration amplitude at resonance.

A simulation of the temperature-dependent stress and experimental results from the first heating cycle are shown in figure 7.2. The simulation of thermal stress behaviour for a square thin film was set up using COMSOL, considering the geometry of the thin film and substrate and their changing dimensions as a function of temperature using the CTEs for the constituent materials. Young’s modulus \( (E) \) was set at 103 GPa and Poisson’s ratio as \( \nu = 0.26 \) for the Ge membrane. Equations for the CTE of Si and Ge were taken from references [214] and [215] which give a
temperature dependent polynomial for each CTE in this range: Si has

\[
\alpha_s = -3.0451 \times 10^{-6} + (3.5705 \times 10^{-8})T \\
- (7.981 \times 10^{-11})T^2 + (9.5783 \times 10^{-14})T^3 \\
- (5.8919 \times 10^{-17})T^4 + (1.4614 \times 10^{-20})T^5 \text{ K}^{-1};
\]

Ge has

\[
\alpha_f = (5.6569 \times 10^{-6}) + (34.22 \times 10^{-12})(T - 273) \\
+ (10.17 \times 10^{-15})(T - 273)^2 \\
- (0.66 \times 10^{-18})(T - 273)^3 \text{ K}^{-1}.
\]

The relative changes in dimension of the thin film and substrate, which will not expand equally, govern the change in stress, and hence frequency, as the temperature is changed. The process is simplified by assuming a single growth temperature, \(T_0\), which is used as the free parameter to fit the experimental data. A value of 450 °C was used for the fit in figure 7.2(a). This is a realistic fit parameter compared to the two growth temperatures; the first, low temperature layer grown at 450 °C will contain most of the defects due to lattice mismatch and dominate the intrinsic stress, while the higher temperature growth layer will show reduced stress. Careful consideration of the growth temperatures and full simulation of the behaviour would allow identification of the intrinsic stress for each thin film. However, this would require knowledge of the thermal behaviour of all of the material properties over an extended range.

At the start of the temperature sweep, as temperature rises, the stress and Q-factor follow the trends shown in the simulation. However, as temperature increases some variations away from this behaviour are observed. The stress, in figure 7.2(a), shows variation away from the simulated values above \(\Delta T = 10 \, ^\circ\text{C}\), with a different gradient to the predictions of the simulation. Similarly, the Q-factor and amplitude (in figures 7.2(b) and (c)) shows similar behaviour to the 3C-SiC sample but at a higher temperature with \(\Delta T = 40 \, ^\circ\text{C}\); beyond that point, the Q-factor enters a period of growth and then further reduction, indicating a significant change to the thin film (\(\Delta T\) and percentage change in stress are set to zero at 23 °C, the temperature of the room).

Sample Ge_Sample_1 was then subjected to further thermal cycling at low pressure, with results shown in figure 7.3. Part (a) shows the percentage change in stress. Repeatability of the measurements following the changes observed in figure 7.2 is now found, with the stress changing by 0.28% per °C, which is more than predicted in the simple model over the temperature range observed. The Q-factor shows a cy-
Figure 7.3: (a) Percentage change in stress found by fundamental resonance and (b) Q-factor behaviour for sample Ge_Sample_1 at 10^{-3} mbar as temperature is changed, for later heating cycles. (c) shows the measurement of the frequency response for the fundamental resonant as a function of temperature. (d) Percentage change in stress calculated using higher resonant modes. ∆T = 0 is set at room temperature 23 °C.

cle and temperature dependent behaviour (see figure 7.3 (b)). There is some small variation in the starting values of the Q-factor at ∆T = 0, but overall the same trend with temperature is observed in each sweep, with the Q-factor increasing with increasing temperature. The behaviour stabilises following the second heating cycle, indicating a stabilisation to the further changes to the damage to the thin film. For a perfect membrane the Q-factor is seen to reduce as temperature is increased, primarily due to softening of the coupling between transducer and sample; this increase in Q-factor shows an improved resonator as temperature increased. The amplitude response (figure 7.3 (c)) also displays the opposite behaviour to expectation before a temperature of 73.1 °C; a reduction in amplitude was expected as temperature is increased due to the softening of the coupling, but the rise in amplitude again sug-
gests an improved resonator. The calculated stress change obtained from different resonant modes has the same behaviour as found from the fundamental resonance (figure 7.3(d)).

Figure 7.4: (a) Percentage change in stress and (b) Q-factor behaviour for sample Ge_Sample_1 at atmospheric pressure undergoing thermal cycles.

Figure 7.4 shows the results of the thermal cycling measurement which was repeated at atmospheric pressure on the sample Ge_Sample_1. The stress reduced following the increase in temperature, as expected from the simple model. However, this trend stopped at ∆T = 25 °C. Meanwhile, the Q-factor remained the same below ∆T = 17 °C to 23 °C, which is the temperature of coupling softening (at 40 °C). Because of the coupling loss, the Q-factor then dropped rapidly, but the drop ended at ∆T = 32 °C, then began to reverse the variation, going from reducing to increasing. The stress change also displayed an unpredicted behaviour after that temperature position. All these unexpected phenomena will be explained in section 7.3.2 (SEM with temperature control).

7.2.2 Perfect Ge Thin Film

Figure 7.5 shows the measurement results obtained from a perfect Ge sample with 700 nm thickness and 955 µm lateral dimensions (Ge_Sample_2). Part (a) shows analysis of the frequency shift for sample Ge_Sample_2, using equation 2.40 to calculate the percentage change in the stress from the shift in resonance frequency as the sample is heated above room temperature. Data is shown for heating and cooling twice from room temperature to the maximum temperature, to show reproducibility, with the reference value (zero percentage change) set at room temperature for each temperature cycle. The stress shows a change of approximately 0.2% per °C.
temperature change.

Figure 7.5(b) shows the variation in Q-factor as temperature is increased. Some variation is observed closer to room temperature, primarily due to variations in the coupling between transducer and sample over thermal cycling. Any changes in residual stress, boundary conditions and coupling will affect the measurement. However, a consistent trend of decreasing with a raising temperature is observed for all cycles which is different to the behaviour of sample \(Ge_{.Sample.2}\).

Comparing the frequency scans for the fundamental resonance of both Ge thin films at several different temperatures, as shown in part (c) in figures 7.3 and figure 7.5, both exhibit a change in Q-factor and resonance amplitude, as well as a shift towards lower resonant frequencies, as the temperature is increased. Some change in amplitude is expected due to the coupling loss due to the slight softening of the glue holding the sample onto the transducer following the increased temperature.

Figure 7.5: (a) Percentage change in stress and (b) Q-factor behaviour for sample \(Ge_{.Sample.2}\) at \(10^{-3}\) mbar as temperature is changed. \(\Delta T = 0\) is set at room temperature. (c) Frequency amplitude response with a varied temperature.
Figure 7.5 (c) shows this behaviour.

Both samples, \texttt{Ge\_Sample\_1} and \texttt{Ge\_Sample\_2}, exhibit the expected resonant vibrations for the fundamental resonant mode measured both at room pressure and without atmospheric damping. This has a resonant frequency of 103.32 kHz for sample \texttt{Ge\_Sample\_1}, and 155.33 kHz for sample \texttt{Ge\_Sample\_2} at microbar pressure. A value of $\Gamma$ of 0.40 was found for both samples before sample \texttt{Ge\_Sample\_1} underwent heating and cooling. Following this, the resonant frequency of sample \texttt{Ge\_Sample\_1} changed to 104.69 kHz, and $\Gamma$ changed to 0.78. The increased NAVMI factor shows changing of the boundary conditions as cracks form around the edge of the thin film, with the presence of cracks confirmed by microscopy (details of the calculation and parameters were described in detail in chapter 2).

### 7.3 Microscopic Testing

#### 7.3.1 Optical Microscope

Figure 7.6: Microscopy image of \texttt{Ge\_Sample\_1} (a) before and (b) after the first thermal cycle. (c) shows a zoom-in.

Figure 7.6 shows images of sample \texttt{Ge\_Sample\_1} taken using an optical microscope. Figure 7.6 (a) shows the thin film condition before the testing, with no cracks visible. (b) shows the sample after extended vibration followed by the first heating cycle, and small cracks can be seen at the corner regions. (c) shows more detail of these cracks. The diversion from the predicted stress and the change in Q-factor was attributed to this initiation of cracks. However, the exact size of the cracks are too small to measure using the optical microscope.
7.3.2 SEM with Temperature Control

Following this measurement the condition of the two Ge thin films were measured using scanning electron microscopy (SEM, Zeiss SUPRA 55-VP) with preliminary results shown in figure 7.7. This measured a corner on each of the two thin films, with the image taken from the substrate and interface side. Figure 7.7(b) shows sample Ge_Sample_2 to be a good quality thin film. However, sample Ge_Sample_1 shows a series of macroscopic crack-like defects clustered around the membrane edges (figure 7.7(a)). These are likely due to the previous high amplitude vibrations and thermal cycling, and may have initiated during the measurement shown in figure 7.2, explaining the change in behaviour of Q-factor, stress and vibration amplitude.

A further SEM measurement utilising temperature control, for a temperature range of 32 to 95 °C, was taken on sample Ge_Sample_1 to understand the temperature-dependent behaviour of the stress and Q-factor. The sample was placed on a copper ring, then onto a manually controlled heating stage which replaced the standard sample holder inside the SEM chamber. The resolution of the temperature control stage was 0.1 °C. The SEM chamber pressure was reduced to 10^{-6} mbar. Temperature was directly measured by a sensor which was fixed inside the copper stage under the sample. Before each measurement the system was left for 30 minutes to reach thermal equilibrium. The measured SEM pictures under different temperatures are shown in figure 7.8. Part (a) was measured at a temperature of 35.4 °C, part (b) is at the same position as (a) but for a temperature of 94.6 °C, with the image zoomed.
in to show more detail. Parts (c) and (d) show cracking at another position on the membrane measured at 81.6 °C.

Figure 7.8: SEM images of sample Ge_Sample_1 taken at 10^{-6} mbar. (a) and (b) show the same region of the membrane at 35.4 °C and 94.6 °C respectively. (c) and (d) show another region at 81.6 °C.

The SEM images under temperature control in figure 7.8 shows interesting thermal behaviour. Parts (a) and (b) show a non-uniform growth and bulging of the Ge, leading to the formation of apparent joins between the two sides of a crack. Similarly, (c) and (d) show a partial bridging of the cracking which appears as the temperature is raised. This is due to thermal expansion of the Ge into the cracked region. The depth of the cracks are measured to be around 200-400 nm, i.e. less than the thickness of the thin film, hence the non-destructive nature of the cracking and the fact the thin film was not destroyed. The openings of the cracks range from tens of nm to around 1 µm, with most of the bridges formed when the width is smaller than 500 nm. The cracks at corner positions did not shown any healing as the temperature was increased because the size of these was around 2 to 3 µm. This self-healing phenomenon as the temperature is raised can explain the resonant
behaviour and the longevity of the Ge materials during operation. As the temperature is raised and bridges are formed the thin film resonance behaviour will improve, leading to an increase in Q-factor (as shown in figure 7.3(b)), counteracting the reduction in Q due to frequency shifts and temperature dependences in the experimental set-up. This also explains the amplitude increase shown in figure 7.3(c) for sample Ge_Sample_1 as the temperature is increased. The connections improve the propagation of the wave on the thin film, reducing energy dissipation.

7.4 Summary

- Individual cycle testing of large amplitude oscillation and thermal cycles on suspended thin film structure would not easily generate cracks with a macroscopic scale. However, prolonged vibrations can lead to damage.

- Cracks can be initiated more quickly when combining the two measurements of large amplitude oscillation and thermal cycles together, which may occur in an industrial environment.

- Cracks can be healed using thermal expansion when their size is smaller than 500 nm.

- Measurement of the resonant behaviour over a small range of temperature variation provided a potential technique for a quick, non-destructive testing method for very small cracks on these samples.
Chapter 8

Conclusions and Future Work

8.1 General Conclusions

The main objective of this work was to develop and enhance the capability of a non-destructive testing method for characterising the mechanical properties of materials in the form of suspended structures. The development and application of non-contact ultrasonic testing techniques based on an optical interferometer as a probe have been discussed. This testing method opens the applicability of ultrasonic measurements to a wider range of materials and structures than have previously been studied, including studying the linear and non-linear dynamic behaviours of small size suspended structures for MEMS or NEMS.

A laser scanning system was developed by combining a two-wave mixing interferometer, a self-made X-Y-stage and a vacuum chamber. The setup allowed the time-resolved transverse displacement of suspended structures to be measured. By using an external actuator with a broadband excitation, individual resonant frequencies and modes could be excited and separated out in post-processing. The results showed a good agreement with the theoretical predictions and enabled testing of some new theoretical models for nonlinear oscillations.

In chapter 4, the mechanical properties of suspended thin films made from different materials (Ge, 3C-SiC, SiN) on a Si substrate, with different sizes, were investigated through linear resonance testing with small amplitude vibrations. These measurements used the resonant frequencies and related mechanical modes to analyse the residual stress by inputting these parameters into a theoretical equation. The simplest method is to treat the thin film as an ideal membrane. This is not the most accurate model for thin films in the real world because the thin film is best considered as a thin plate under tension, however, the approximation to an ideal membrane
works well for these materials due to their geometry. For large amplitude oscillation with a strong excitation voltage for the piezoelectric transducer used to generate the oscillations, the suspended thin films exhibited non-linearities in the vibration with a super-harmonic or internal resonance observed. The experimental results from suspended thin films with fully clamped boundaries in chapter 5 displayed a relationship between the starting point of nonlinearity, and a critical amplitude of displacement as a turning point for exciting the vibration into non-linear oscillation. At the same time, a location or integrating area dependent performance of the amplitude response curve was discovered. The non-linear behaviours do not match those described by the Duffing equation for nonlinear resonances because the Duffing equation was developed from plate bending rather than a plate under tension. These measurements provide further experimental results to support the development of new theories of non-classical non-linear vibrations; there are not many experimental results reported in the literature.

The thermal influence induced by the probe laser was investigated to analyse how the residual stress was affected by thermal expansion differences for the suspended thin film and the substrate. A dynamic thermal field created by the probe laser might be able to be used as a new tool to investigate the material properties such as isotropy (or anisotropy) by observing a resonant frequency splitting which varies with temperature and laser probe position on the sample. This splitting was discussed in chapter 6, section 6.4, where the modes 1:3 and 3:1 on a Ge thin film split depending on laser probe position on the membrane.

A thermal cycling test was conducted on several samples. The residual stress change was analysed for both experimental and theoretical investigations and combined with monitoring of variations in the Q-factor. This suggested a new way to inspect the fatigue of MEMS or NEMS devices with submicron or nanometer size cracks on suspended structures.

8.2 Suggested Future Work

This work has developed a new technique for materials analysis and a potential new non-destructive testing technique for small-scale samples through analysing the changes in vibration behaviour. There are a large number of potential avenues for further investigation.

Preliminary measurements were also made of the effect of thin film thickness on the results, covering measurements of the residual stress, thermal influence of the probe laser, and membrane dynamics, but are not reported here. Part of the work
of estimating the thickness and defect density effects was started and preliminary results are shown in figure 8.1. Two methods were used to investigate the thickness effect. The first used a set of membranes grown with different thicknesses initially. However, as shown in earlier chapters, there is some variation in residual stress between samples. Further measurements used etching to reduce the thin film thickness. Etching was done from either the top of the film (just reducing the thickness) or from the interface with the substrate (also removing the layer containing more defects). Preliminary results suggest that the defect removal has an effect on the measured stress, but that the thickness dominates the measurement. Further investigation is required.

Figure 8.1: (a) Stress change after 3C-SiC thin film etched to different thickness on either side (towards from top to interface and opposite direction). (b) Cross section TEM image of a 3C-SiC thin film. TEM picture taken by G. Colston [21].

The thermal effects investigated in chapter 6 suggested some sample geometry effects on the thermal influence of the probe laser. The thickness measurements could be continued to investigate fully these effects.

There is the potential for a non-destructive testing technique for these small-size samples which could be applied on-line during production of suspended structures for MEMS devices, or applied for testing of devices following operation in vibration environments to look for damage. To change this to a fully non-contact inspection would require investigation of alternative methods of generating the vibrations, such as using air-coupled transducers. The air-coupled transducer can help to remove the coupling impact which comes from physical contacting with the piezoelectric transducer. However, this contactless benefit only can be applied in an air environment.
which can strongly reduce the thin film vibration strength and give a large damping effect. The frequency and bandwidth of such transducers would need to be developed and potentially improved. There is the potential that 2D scans would not be required; with full knowledge of the expected behaviour with and without damage, measurements could be done at a reduced number and potentially just one measurement location to improve analysis time.

Many different geometries of suspended structures are being produced, such as the beams and spiderwebs shown in figure 3.5 from the experimental methods chapter 3 and in reference [21]. There is a theoretical basis for linear vibrations of beams, which could be applied to measurements of such samples, and extended to analyse linear and non-linear vibrations. The spiderweb structure is very complicated for vibration analysis, but these structures are prone to damage and the vibration behaviour could simply be used as a pass/fail test, simplifying the measurement and removing the need for complex microscopy investigations to test for damage.
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