Atomic force microscopy using a novel on-chip interferometric readout

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Abstract

Currently, the most common readout approaches for atomic force microscopy (AFM) systems are based on the optical beam deflection (OBD) technique. OBD relies on monitoring the deflection of the sensing cantilever probe by measuring the position of the laser beam reflected from the free end of the cantilever using two or more detectors. Alternative AFM readouts consist of electrical readouts, including piezoelectric solutions and tuning fork probes. Although the OBD readout approach achieves subnanometer resolution and video rate imaging speeds, it is hindered in terms of size of the reflected laser beam and the noise originating from mechanical vibration of the free-space optics. A limitation of the OBD readout is its size, which is difficult to minimise as the extent of leveraging is directly associated with the distance travelled by the reflected light beam. System miniaturisation can be accommodated by adopting electrical readout solutions, often at the expense of measurement sensitivity. Electrical readouts can be integrated on-chip and provide scalability for ultrafast imaging using multiprobe arrays. However, imaging in liquids might be challenging, requiring additional electrical isolation. To date, no cost-effective AFM readout solution exists without sacrificing either the measurement sensitivity, system miniaturisation, or multiprobe array scalability. This gap is addressed in this thesis.

We present the realisation of an AFM probe with integrated on-chip optical interferometric readout based on silicon photonics. Our AFM probe merges the advantages of subnanometer resolution of optical readouts with miniaturisation previously available only via electrical readout solutions. Our readout does not require any alignment and can operate in air and liquid. The adopted approach determines the deflection of the cantilever in contact with the imaged surface using an integrated on-chip photonics waveguide monitoring the distance to the sensing cantilever via an interrogating Bragg diffraction grating. The implemented methodology provides ultimate interferometric resolution, on-chip miniaturisation, and scalability, to ultrafast multiprobe-array imaging.

The AFM probe response was characterised by electrostatic actuation, achieving the expected interferometric response and measurement resolution. We present AFM imaging of reference samples using the developed probe, demonstrating a significant reduction of the measured noise level compared to a modern commercial AFM tool (Bruker Dimension ICON). We achieve an AFM static image root-mean-square (RMS) noise floor of 19 pm surpassing the performance of OBD (25 pm) and piezoresistive readouts (32 pm). The noise spectrum measurements of our probe indicate that our readout is shot noise limited, achieving a deflection noise density (DND) of 36 fm/√Hz.

In this thesis, we present the design, fabrication and characterisation of a micromachined integrated on-chip AFM probe operating in contact mode, with the prospect of tapping mode operation. We analyse light coupling to the silicon photonics chip viable for our application, and demonstrate device fabrication as well as probe assembly. In the mechanical
design of the sensing cantilevers, we adopt a double anchor geometry to eliminate spring effects of the surface micromachined anchors. To achieve a useful mechanical excursion and to minimise the effects of squeezed film damping, thick sacrificial layers around 8 µm are required, which are not commonly used for microelectromechanical systems (MEMS) fabrication. Accordingly, effort is devoted to the development of a flexible device fabrication process utilising material of either inorganic (amorphous silicon) or organic (polyimide) alternatives for the 8 µm thick sacrificial layers. In addition, the attachment of a sharp standard AFM tip using a focused ion beam (FIB) is detailed.
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Authorship declaration: co-authored publications

This thesis contains work that has been [published and/or prepared for publication].

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Location in thesis:
Chapter 4

Student contribution to work:
Carried out all processing, in situ optimisation of ICPRIE etching processes, and characterisation of the results. Primary contributor to manuscript preparation.

Co-author signatures and dates:

Details of the work:

Location in thesis:
Chapter 4

Student contribution to work:
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Co-author signatures and dates:

Details of the work:

Location in thesis:
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Student contribution to work:
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Co-author signatures and dates:

Student signature: [Redacted]
Date: 21/12/17

I, Lorenzo Faraone certify that the student statements regarding their contribution to each of the works listed above are correct.

Coordinating supervisor signature: [Redacted]
Date: 21/12/17
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<tr>
<td>ADC</td>
<td>analog-to-digital converter</td>
</tr>
<tr>
<td>AFM</td>
<td>atomic force microscopy</td>
</tr>
<tr>
<td>AlGaAs</td>
<td>aluminum gallium arsenide</td>
</tr>
<tr>
<td>AlN</td>
<td>aluminium nitride</td>
</tr>
<tr>
<td>AOM</td>
<td>acousto-optic modulator</td>
</tr>
<tr>
<td>CCPRIE</td>
<td>capacitively-coupled plasma reactive-ion etching</td>
</tr>
<tr>
<td>CMOS</td>
<td>complementary metal-oxide-semiconductor</td>
</tr>
<tr>
<td>CMP</td>
<td>chemo-mechanical polishing</td>
</tr>
<tr>
<td>CVD</td>
<td>chemical-vapour deposition</td>
</tr>
<tr>
<td>DBG</td>
<td>distributed Bragg grating</td>
</tr>
<tr>
<td>DBR</td>
<td>distributed Bragg reflector</td>
</tr>
<tr>
<td>DC</td>
<td>direct current</td>
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<tr>
<td>DND</td>
<td>deflection noise density</td>
</tr>
<tr>
<td>DUV</td>
<td>deep-UV</td>
</tr>
<tr>
<td>FDTD</td>
<td>finite-difference time-domain</td>
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<tr>
<td>FEM</td>
<td>finite element method</td>
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<tr>
<td>FIB</td>
<td>focused ion beam</td>
</tr>
<tr>
<td>FPI</td>
<td>Fabry-Pérot interferometer</td>
</tr>
<tr>
<td>FWHM</td>
<td>full width at half maximum</td>
</tr>
<tr>
<td>GaAs</td>
<td>gallium arsenide</td>
</tr>
<tr>
<td>GRIN</td>
<td>graded index</td>
</tr>
<tr>
<td>HF</td>
<td>hydrofluoric acid</td>
</tr>
<tr>
<td>HWCVD</td>
<td>hot wire chemical-vapour deposition</td>
</tr>
<tr>
<td>ICP</td>
<td>inductively-coupled plasma</td>
</tr>
<tr>
<td>ICPCVD</td>
<td>inductively-coupled plasma chemical-vapour deposition</td>
</tr>
<tr>
<td>ICPRIE</td>
<td>inductively-coupled plasma reactive-ion etching</td>
</tr>
<tr>
<td>InGaAs</td>
<td>indium gallium arsenide</td>
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<tr>
<td>JFET</td>
<td>junction gate field-effect transistor</td>
</tr>
<tr>
<td>KOH</td>
<td>potassium hydroxide</td>
</tr>
<tr>
<td>LETI</td>
<td>Laboratoire délectronique des technologies de l’information</td>
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<tr>
<td>Abbreviation</td>
<td>Definition</td>
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<td>--------------</td>
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<tr>
<td>LPCVD</td>
<td>low pressure chemical-vapour deposition</td>
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<tr>
<td>MDD</td>
<td>minimum detectable deflection</td>
</tr>
<tr>
<td>MEMS</td>
<td>microelectromechanical systems</td>
</tr>
<tr>
<td>mSPM</td>
<td>metrological scanning probe microscopy</td>
</tr>
<tr>
<td>MWCVD</td>
<td>microwave chemical-vapour deposition processes</td>
</tr>
<tr>
<td>OBD</td>
<td>optical beam deflection</td>
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<tr>
<td>OES</td>
<td>optical emission spectroscopy</td>
</tr>
<tr>
<td>OSNR</td>
<td>optical signal-to-noise ratio</td>
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<tr>
<td>PDF</td>
<td>probability density function</td>
</tr>
<tr>
<td>PDMS</td>
<td>polydimethylsiloxane</td>
</tr>
<tr>
<td>PECVD</td>
<td>plasma enhanced chemical-vapour deposition</td>
</tr>
<tr>
<td>RF</td>
<td>radio frequency</td>
</tr>
<tr>
<td>RIE</td>
<td>reactive-ion etching</td>
</tr>
<tr>
<td>RMS</td>
<td>root-mean-square</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscope</td>
</tr>
<tr>
<td>SiC</td>
<td>silicon carbide</td>
</tr>
<tr>
<td>SNAM</td>
<td>scanning near-field acoustic microscopy</td>
</tr>
<tr>
<td>SNR</td>
<td>signal-to-noise ratio</td>
</tr>
<tr>
<td>SOI</td>
<td>silicon-on-insulator</td>
</tr>
<tr>
<td>STM</td>
<td>scanning tunnelling microscope</td>
</tr>
<tr>
<td>TFT</td>
<td>thin film transistors</td>
</tr>
<tr>
<td>TIR</td>
<td>total internal reflection</td>
</tr>
<tr>
<td>TMAH</td>
<td>tetramethyl ammonium hydroxide</td>
</tr>
<tr>
<td>VCSEL</td>
<td>vertical-cavity surface-emitting laser</td>
</tr>
<tr>
<td>VHF</td>
<td>very high frequency</td>
</tr>
<tr>
<td>VSA</td>
<td>vector signal analyser</td>
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<tr>
<td>ZnO</td>
<td>zinc oxide</td>
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Chapter 1

Introduction

Atomic force microscopy (AFM) is an important imaging technique that allows surface feature resolution on an atomic scale, not only for hard surfaces, but also soft biological samples in liquid environments. AFM is the imaging of surfaces using a physical probe, generally a cantilever with a sharp tip that scans the specimen. An image of the surface is obtained by recording, as a function of sample X-Y-position, either the probe-surface interaction via cantilever deflection, or by recording changes in the sample or probe Z-position under a condition of a constant probe-surface interaction, for example, constant cantilever-to-sample force [1].

The optical beam deflection (OBD) readout is the most common technique used for monitoring the deflection of the sensing cantilever in current commercial AFM systems. As depicted in Figure 1.1, the OBD systems determine the cantilever deflection by measuring the position of the laser beam reflected from the cantilever probe on a relatively distant position-sensitive detector [2, 3]. This external optical path is bulky, requires stable free-space optics, needs careful alignment and is particularly sensitive to vibration. The extent of optical leveraging is directly related to the distance travelled by the reflected laser beam, however, the sensitivity to vibration is also exacerbated by a high degree of optical leveraging. Nevertheless, the OBD method has been adopted since it is sufficiently sensitive for the most demanding applications. The sensitivity of the OBD method can be surpassed by optical interferometric methods, and several interferometric cantilever deflection readouts have been presented in the literature [4, 5]. However, none of them were implemented in commercial imaging systems as the presented interferometric readouts require precise and intricate alignments. Most AFM systems with OBD use standardised cantilever probes that are interchangeable between various manufacturers. The AFM probes are classified as consumables in the measurement systems, as the lifetime is strictly limited due to the fragile structure of the probe as well as the wear of the even more delicate sharp tip.

An alternative to optical beam cantilever monitoring is electrical readout. There are several successful commercial electrical readout implementations and some examples can achieve accuracies approaching that of OBD readout. The piezoresistive electrical readouts use buried piezoresistors integrated within the AFM cantilever probe to determine probe deflection [6–8]. This readout methodology can be characterised by high deflection...
measurement accuracy with concurrent high imaging speed. However, in comparison to standard AFM OBD readout probes, a higher cost is associated as they require ion implantation to form piezoresistors. Since the AFM probes are consumable items, they should be as cheap as possible. Furthermore, tuning fork piezoelectric based AFM probe readout is currently achieving the ultimate sensitivity, however, at the expense of further probe complexity and with limited imaging speed [9–11]. An emerging salient feature of next generation AFM systems appears to be large-area ultrafast imaging solutions facilitated by the adoption of sensing using multiprobe arrays [12–14]. Given the limitations associated with the available readout techniques, a technology gap exists for a cost-effective AFM readout solution without sacrificing either the measurement sensitivity, system miniaturisation, or multiprobe array scalability.

The goal of this thesis is to evaluate silicon photonics-based integrated on-chip interferometric readout for use as a novel AFM probe [15–17]. We aim to develop a sensing probe technology, preferably compatible with current AFM systems, requiring minimum changes to the existing tools to achieve significant improvement of imaging performance by using existing X-Y-Z scanners, as well as allowing future fast and multiprobe imaging with new systems. We adopt an AFM surface micromachining approach for AFM probe fabrication, leading to the fabrication of an AFM probe integrated with a silicon photonics readout. The performance of the developed prototype is characterised and compared to a modern commercial AFM tool, and we demonstrate that the imaging performance of our integrated readout exceeds that of the commercial OBD readout.

This thesis is divided into seven chapters. The following chapter discusses in-detail the current state of the AFM field, presenting the AFM readout approaches available in
the open literature. Chapter 3 details the operation principles of our readout approach providing the device design and a simple optical ray-path analytical solution describing the device operation, as well as simulation results of the optical resonant cavity. We discuss several requirements that needed to be addressed before probe fabrication. Chapter 4 presents details of the materials and methods used in the adopted microelectromechanical systems (MEMS) fabrication process. The discussion includes the analysis of the structural layer material and of two distinct materials that can be used as the sacrificial layer. Chapter 5 presents the device fabrication process based on the outcomes of Chapter 4, along with the device assembly and the measurements set-up. Chapter 6 presents the characterisation of the realised AFM probe, including the cantilever probe interferometric response to electrostatic actuation, which provides the cantilever deflection to optical response transfer function. Subsequently, the AFM imaging capability is demonstrated along with an investigation of signal noise sources and levels for the presented AFM probe. Chapter 7 presents the thesis summary, conclusions and the proposed future work for further development of the integrated AFM probe interferometric readout technology.
Chapter 2

Literature review

This chapter starts with a short introduction to AFM operation principles and a description of contact and non-contact measurements, followed by a section outlining how the performance of AFMs are characterised. Subsequently, the main part of the chapter will introduce different methods to measure cantilever tip displacement ranging from simple optical beam steering and optical interferometric approaches, through self-sensing techniques, which include capacitive, piezoresistive and piezoelectric readouts, to various integrated on-chip optical readouts. The chapter concludes with a summary comparing the different approaches to highlight the benefits of our method presented in this thesis.

2.1 Introduction to atomic force microscopy

Atomic force microscopy (AFM) is based on the interaction forces between a sharp tip and a sample when in close proximity (mainly attractive van der Waals forces and repulsive Coulomb forces). During a typical AFM measurement, schematically depicted in Figure 2.1(a), a sharp tip attached to a long cantilever probe is scanned above the sample surface and, due to the interaction between tip and surface, the cantilever deflects, which when sensed, allows the generation of a topological image of the surface. Under ideal conditions, the probe has a sharp tip such that a single atom at the apex is interacting with a surface. Minimising the tip area corresponds to maximising the spatial resolution of the measurement as shown in Figure 2.1(b).

In 1982 Binnig et al. [19] presented a scanning tunnelling microscope (STM), which later evolved into the first AFM [1]. An STM uses the quantum tunneling effect and by measurement of the tunneling current between the conductive sample surface and a sharp tip the distance between them can be estimated. During the imaging process, the current between the STM probe and the surface is held constant by adjusting the distance between them with a Z-direction mechanical movement of the sample or cantilever. The varying displacement in the z-direction is recorded as a surface topology as the sample is scanned in the X-Y plane.

In 1986, Binnig et al. received the Nobel Prize for the invention of the STM, which was followed by invention of the AFM. The first AFM used an aluminium cantilever with
an attached diamond tip. The AFM cantilever was attached to a piezoelectrical actuator used to vibrate the cantilever near its resonant frequency. The deflection of the cantilever was monitored using an STM, which was measuring the distance between the STM probe and the back-side of the metal AFM cantilever. The sample was attached to a 3-axis piezoelectric scanner that was used for sample imaging (X-Y-direction movement) and to maintain a constant interaction force between the sample and the cantilever (Z-direction movement). During that experiment, Binnig et al. [1] achieved a lateral resolution of 3 nm and vertical resolution below 0.1 nm in air.

2.2 AFM operation modes

AFMs can operate in several modes, which can be distinguished in terms of: (1) the force of interaction; (2) the way we control that interaction; and (3) the type of interaction between the tip and a surface.

The interaction force between the probe tip and sample surface is a crucial parameter as it is used to "feel" the sample’s surface and create 3D images. The interaction force, as shown in Figure 2.2, can be divided into two main regions: (1) contact region where we can observe a repulsive force between tip and sample; and (2) non-contact region, where we can observe an attractive force between tip and sample. In the contact region, the repulsive force is due to the AFM tip being in physical contact with the sample. During operation in the non-contact region, the tip is in close proximity with the sample surface, and the attractive van der Waals force can be observed between the AFM probe tip and a sample surface.
Figure 2.2: Interaction forces between the AFM tip and a surface as a function of the distance between the sample and AFM tip after Bruker Corporation [20]. Three zones can be distinguished: (1) The contact zone, where we observe only repulsive force; (2) non-contact zone, where the tip is not in physical contact with the sample and we can observe attractive force; and (3) intermittent contact region, which include both contact and non-contact regions.

The interaction between AFM tip and the surface can be controlled in two ways, which are described as open and closed feedback loop operation. In the open feedback loop operation, the deflection of the probe is fixed during the approach. The cantilever tip is set in contact with some initial deflection. The varying deflection of the cantilever probe as it traverses the surface is directly translated into a surface topographical image. Open feedback loop operation is sensitive to misalignment between the sample surface level and the scanning plane. It is limited to relatively flat samples and comes with a significant risk of damaging the tip or the sample surface during scanning due to excessive interaction forces between tip and sample surface. The main advantage of open loop operation is the possibility of high-speed imaging since the scanning rate is not limited by the feedback loop response time.

Nevertheless, most commercial AFMs use closed feedback loop operation mode, which is also often referred to as constant force mode. The interaction force between a sharp tip and a sample surface is kept at a constant level using a feedback loop controlling the tip to surface separation. The Z-stage movement during the scanning process is recorded as a topological image of the sample surface. Constant force mode guarantees the same force of interaction between the AFM tip and the sample surface during the imaging process, but the scanning operation is limited by the speed of the feedback loop response. The improvements towards fast scanning AFMs often involve new approaches to operating the feedback loop at higher operating bandwidth.

The type of interaction between the sharp tip and sample surface can be divided into two operation modes: contact mode and tapping mode. In contact mode, the tool operates in the contact interaction force region. The AFM tip is dragged along the sample surface,
and the deflection of the cantilever is translated either on feedback loop response or directly used to generate an image. Contact mode is the most basic AFM operation mode allowing for surface profile measurements with atomic resolution [21, 22]. However, the forces between tip and sample can quickly destroy the surface of some sensitive samples [23]. Furthermore, due to the physical contact, the AFM tip will wear quickly and needs to be replaced more often.

An alternate to contact mode of operation is tapping mode, which can be conducted entirely in the non-contact region or extended into intermittent contact. In tapping mode, an AFM probe vibrates at a frequency close to its resonance and the change in the interaction between probe and surface (as the probe taps the surface) can be observed as a change of vibration amplitude or frequency of the cantilever. In comparison to contact mode, tapping mode allows characterisation of more sensitive samples since the interaction forces between the AFM tip and a sample surface are limited [23].

During an AFM measurement, in any operation mode, we need to determine the deflection of the cantilever precisely. The readout method needs to be stable over time and, if possible, insensitive to external vibrations. There are several methods for measurement of cantilever deflection, including the most frequently used OBD, interferometric readout, as well as integrated electrical and optical readout. These readout methods are described in the Section 2.4.

2.3 AFM performance characterisation

The performance of AFM systems is generally characterised using three different factors: as a static AFM noise image; minimum detectable deflection (MDD); and deflection noise density (DND).

Static AFM noise images characterise noise of the whole imaging system, which includes readout noise and other potential noise sources, originating from mechanical construction of the tool and its vibration isolation, as well as from AFM electronics, not directly related to cantilever deflection readout.

The noise images are acquired in contact between the AFM tip and sample while the AFM tip remains stationary in one position on the sample and the piezo-scanner controlling Z-axis movement is disabled or set to very slow response to prevent the cantilever from drifting off the surface. The calculated root-mean-square (RMS) surface roughness ($R_{RMS}$) is given by the standard deviation of the recorded height values:

$$R_{RMS} = \sqrt{\frac{\sum_{n=1}^{N} (Z_n - \overline{Z})^2}{N}}$$  \hspace{1cm} (2.1)

where $Z_n$ is the height of point $n$ and $N$ is number of points on the sample. The static AFM noise images are measured for a particular measurement bandwidth used in the imaging system. This makes it difficult to compare values recorded for two systems if they use significantly different measurement bandwidth.
The minimum detectable deflection (MDD) is the minimum change of the cantilever deflection which can be detected by measuring the change in the output signal produced by the readout, which means that MDD defines the cantilever deflection which causes the output signal change to be equal to the root-mean-square (RMS) noise value. It is also known as the noise floor of the measurement system. The MDD can be defined as a function of signal-to-noise ratio (SNR) [24]:

\[ MDD = \frac{1}{\sqrt{SNR}} \quad [m] \] (2.2)

MDD, similar to static AFM noise images, depends on measurement bandwidth, while the deflection noise density (DND) noise power per unit bandwidth, is defined as [25]:

\[ DND = \frac{MDD}{\sqrt{\Delta f}} \quad \left[ \frac{m}{\sqrt{Hz}} \right] \] (2.3)

where \( \Delta f \) is the measurement bandwidth, and hence DND is independent of bandwidth.

2.4 AFM probe displacement measurement techniques

An essential part of an AFM system is the measurement of the cantilever probe displacement, and one of the most critical inventions in that field is the OBD readout method. It was demonstrated first in 1988 by Meyer and Amer [2], where they used a laser beam directed to a mirror in rigid contact with the cantilever as shown in Figure 2.3. Light is reflected from the mirror towards a position sensitive detector. Small deflections of the cantilever were translated to large movements of the light spot on the detector (optical beam leveraging). Shortly after that publication, Alexander et al. [3] presented results of an optimised experimental set-up where they achieved atomic resolution imaging of the surface of

![Figure 2.3: Experimental set-up of the optical beam deflection (OBD) readout method presented by Meyer and Amer [2]. In this readout method laser light, directed towards the cantilever is reflected from the top side of the cantilever to the position sensitive detector. Small changes in the cantilever position (mirror) are translated into large changes of the beam position on the detector.](image-url)
native oxide formed on a silicon sample. To date, the OBD method remains the most popular readout method in commercially available AFMs. However, the most significant disadvantage of OBD method is its susceptibility to vibration. Systems use bulk optics attached to substantial mechanical supports which can vibrate during measurements introducing additional noise in the measurements. Binnig et al. [1] in the first AFM set-up attached all components of their system to a block of aluminium to minimise the vibrational impact on system performance.

In parallel with the OBD method, an interferometric readout was also developed. In 1987, Martin et al. [26–28] presented an AFM with the interferometric readout shown schematically in Figure 2.4. Their system used a laser heterodyne interferometer to measure the amplitude of the probe vibration, and the measured changes were used to generate a topographic image of the sample. The presented interferometer used an acousto-optic modulator (AOM) to modulate laser light that is split subsequently into two optical paths. The first part is reflected directly towards the detector, whereas the second part is directed towards the cantilever, reflected back to the photodetector and mixed with the first part of the signal. The amplitudes of both signals combine interferometrically and create a response signal representing the vibration of the cantilever. In their work, Martin et al. [26–28] achieved a height resolution down to 5 nm in topographical imaging.

![Figure 2.4: Adapted schematic of experimental set-up of the AFM using a laser heterodyne interferometer readout method presented by Martin et al. [26–28]. Laser light is modulated inside the laser probe using a Bragg cell and then part of the light is reflected directly towards the photodetector, whereas the remaining light is transmitted towards the vibrating cantilever and reflected back towards the photodetector. The interferometric effect between the two beams provides information about cantilever motion.](image-url)
Schönenberger and Alvarado [4] improved the sensitivity and DC stability of the interferometric displacement sensing approach. The main difference to prior reported work was the use of a beam polarisation splitter to produce a reference optical beam. The interferometer measures the motion of the cantilever and at the same time the motion of its anchor point using an additional beam. The differential measurement allows common mode noise to be minimised, which is caused by mechanical vibrations between the cantilever and the optics.

Rugar et al. [5] presented an AFM with a fibre-optic interferometer designed for measurement of cantilever displacement. As depicted in Figure 2.5, the interferometric effect occurs between the light reflected back from the glass-air interface at the end of the optical fibre and the light reflected from the cantilever. The difference between the two light optical paths leads to an optical interferometric effect in the optical fibre. The fibre-optic interferometer used a directional coupler, a device which allows separation of the optical signals which travel in opposite directions inside an optical fibre, and then used a photodetector to measure the deflection [29]. Use of fibre optics simplified the device construction, thereby eliminating the need for bulk free-space optics and making the interferometer less susceptible to external vibrations.

![Figure 2.5: Interferometric effect in fibre-optic interferometer. Part of the light is reflected back from the glass-air interface at the end of the fibre, as well as from the cantilever. The difference in the distance between the two optical paths of light causes the optical interference effect.](image)

Figure 2.6 schematically shows the set-up presented by Rugar et al. [5] that is compact and robust due to an all-fibre construction and the use of a semiconductor laser source. The adopted design allows the achievement of low noise levels, with peak-to-peak noise less than 10 pm in a 1 kHz bandwidth. Their demonstration was conducted on thin film samples where magnetic-domains were mapped by sensing magnetic forces between the tip and the sample in non-contact mode.

Kong et al. [30] presented surface-micromachined AFM probes with an integrated electrostatic actuation circuit and an interferometric position readout based on a fibre-optic interferometer. Their design used a poly-silicon cantilever as depicted in Figure 2.7 with an attached sharp tip, and the optical fibre rigidly connected to the chip. The interferometric cavity is created between the face of the sensing optical fibre attached to the silicon substrate and the back-side of the cantilever. The advantage over the method used by
Rugar et al. [5] was the reduction of common mode noise since all optical readout elements were in rigid contact with each other and the carrier wafer that was used as a substrate during fabrication of the silicon cantilever. Additionally, the demonstrated cantilever sensor had integrated on-chip electrostatic actuation, which could be used during dynamic measurements in tapping mode.

Putman et al. [31] presented a theoretical comparison between the interferometric and optical beam deflection techniques. They compared a Michelson interferometer and two implementations of OBD readout in order to determine which system can achieve better sensitivity. In summary, their final results show that both approaches are equal under optimal conditions.

Figure 2.7: Cross-section of the surface micromachined AFM probe with a poly-silicon cantilever equipped with electrostatic actuation and external fibre optic interferometer for cantilever position readout presented by Kong et al. [30].
2.5 Self-sensing displacement

Measurement techniques where the cantilever displacement sensing method is integrated with the cantilever may be termed as "self-sensing". What is meant here is that the cantilever probe does not require reference to any external mechanical datum such as an external position readout. These techniques allow for miniaturisation of the readout system, minimisation of common mode noise, and often significantly reduces the cost of the whole measurement system.

2.5.1 Piezoresistive self-sensing AFM cantilevers

MEMS based piezoresistive self-sensing AFM cantilevers are the most popular alternative to optical readouts. Tortonese et al. [6] presented the first AFM probe with piezoresistive position readout. This method uses a piezoresistive layer formed by ion implantation in the top side of the moving cantilever as shown in Figure 2.8. When the cantilever deflects, and the piezoresistive layer changes its geometrical dimensions, the effective resistance of the layer changes. Tortonese et al. [6] present cantilevers fabricated from silicon-on-insulator (SOI), with the top silicon etched to a thickness of 2 μm. The piezoresistive layer was implanted on the top side of the thin silicon. Subsequently, cantilevers were patterned, with a thin oxide grown to passivate the top of the cantilever, and piezoresistors were connected with the metal electrodes. Then, the top side with the cantilevers was protected with polyimide while the thick silicon substrate was removed under the cantilevers. Finally, devices were released by removing the buried oxide layer under the cantilevers.

Subsequent work conducted by Linnemann et al. [7] and Gotszalk et al. [8] showed that by adopting the piezoresistive approach, it is possible to achieve atomic resolution, with the method being limited by Johnson noise of the resistors. Modern AFM probes

![Figure 2.8](image.png)

**Figure 2.8:** Top view schematic of the self-sensing piezoresistive AFM probe proposed by Tortonese et al. [6]. Piezoresistor was fabricated using ion implantation in top surface on the whole area of the cantilever and connected to the measurement circuit using two metal electrodes.
with piezoresistive deflection readout, presented by Gotszalk et al. [8] were based on a Wheatstone bridge approach and designed using finite element method (FEM) to optimise the sensing performance (reduced noise and maximised sensitivity). Instead of a single long piezoresistor spanning the full cantilever length as proposed previously by Tortoñese et al. [6] and Gotszalk et al. [8] used four piezoresistors located at the point of highest mechanical strain of the cantilever connected as shown in Figure 2.9(a). Subsequently, in the centre of the bridge, a hole was etched to concentrate mechanical strain in the piezoresistor area, as shown in Figure 2.9(b), in order to further increase the sensitivity.

In 2002, Brook et al. [32] presented an AFM probe with a Hall sensor for magnetic imaging based on an AlGaAs/GaAs cantilever with a piezoresistive deflection sensor as shown in Figure 2.10. The fabricated sensor allows for simultaneous topographical and magnetic imaging. During topographical imaging with the AFM cantilever tip, the Hall probe was located in close proximity to the sample surface allowing the collection of the magnetic image.

Based on an approach by Johansson et al. [33, 34], where they formed a piezoresistor using a gold thin film buried between two layers of SU-8, Schneider et al. [13] proposed a multi-cantilever AFM probe. In their work, they fabricated SU-8 cantilevers with integrated tips with a radius of 300 nm to 475 nm in size, which can be considered as sharp for organic tips, but they have a large radius compared to commonly used silicon tips which have about 10 nm tip radius. An array of parallel cantilevers allowed a speed-up of the imaging process by the simultaneous acquisition of six parallel images with a constant offset.

In 2006, Arlett et al. [35] presented microcantilever sensors with piezoresistive deflection readout for force detection. They demonstrated displacement sensitivity of 0.1 pm/√Hz in vacuum at room temperature and a sensitivity of 7.3 fm/√Hz at 10 K. Li et al. [36] presented a NEMS-based cantilever sensor with piezoresistive deflection readout. They presented SiC micro- and nanocantilevers and demonstrated cantilever sensors operating

![Figure 2.9](image.png)

**Figure 2.9:** (a, b) AFM cantilevers with piezoresistive deflection readout presented by Gotszalk et al. [8]. Four piezoresistors are located at the point of highest strain of the cantilever. (b) To concentrate mechanical strain in the piezoresistors, a hole was etched between them as seen in the image.
at 127 MHz frequency (VHF) that were limited by Brownian motion with a displacement sensitivity of 39 fm/√Hz.

Dukić et al. [37] presented a comparison between the piezoresistive readout approach and the OBD method. Dukić et al. [37] showed that an OBD method requires wider cantilevers than piezoresistive readout as they are limited by the laser spot size of the focused laser beam which averages about 20 μm in diameter [38]. Although the latest systems are able to use laser spot sizes as small as 3 μm [39], it is a feature only available in the most expensive, state-of-art equipment. For shorter cantilevers, the sensitivity of the OBD method slightly decreases whereas improvement of sensitivity for piezoresistive readouts is several times larger. In their study, Dukić et al. [37] used two cantilevers with dimensions of 300 μm × 100 μm and 70 μm × 30 μm. For the smaller investigated cantilevers, they achieved better noise performance using piezoresistive readout than an OBD readout. However, the fabrication of the necessary piezoresistors is challenging and costly [40, 41].

2.5.2 Piezoelectric self-sensing AFM cantilevers

MEMS-based piezoelectric self-sensing AFM cantilevers directly compete with the more commonly used piezoresistive technologies. Piezoelectric sensing is based on materials such as ZnO or AlN, which under mechanical strain generate an electric charge. The reverse piezoelectric effect also takes place, which means that thin films can generate mechanical strain as a result of an applied electrical field. The ability to self-actuate is the most significant advantage of the piezoelectric technology over the piezoresistive approach, where such a reversal effect does not occur, and in the piezoelectric approach, the same material can be used for both actuation and sensing. In 1982, a ZnO piezoelectric transducer was initially presented by Chen et al. [42] as part of a silicon micro-beam accelerometer. The accelerometer used a ZnO piezoelectric sensing layer deposited on top of a silicon beam as presented in Figure 2.11. The ZnO piezoelectric layer is encapsulated within a SiOx thin film and capacitively coupled using two electrodes, one created within the silicon beam and the second deposited on top of the structure. The deflection of the cantilever was measured as the change of electric charge within that capacitor.

In 1993, Itoh and Suga [43] presented the first ZnO piezoelectric AFM sensor designed for operation in non-contact mode. New work on piezoelectric AFM sensors was presented
by Ruppert et al. [44–46] and Moore et al. [47] using AlN piezoelectric thin film deposited on top of silicon cantilevers for actuation and sensing. The proposed design of the active layers allows the use of different cantilever vibrational mode shapes during AFM measurement as shown in Figure 2.12. Electrodes with opposite polarity were used to activate selected vibration modes. Optimisation of piezoelectric electrode shapes using numerical simulations allowed for maximizing both deflection of the cantilever as well as the readout sensitivity. The proposed design approach for the piezoelectric structures has the potential to be used in multi-frequency AFM sensors.

Doll and Pruitt [40] presented a comparison between piezoresistive and piezoelectric deflection readouts. The performance was compared in terms of measurement noise

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<th>Mode 1</th>
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<td>Experimental measured mode shapes</td>
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Figure 2.12: AFM cantilever design with AlN piezoelectric thin film coatings presented by Moore et al. [47]. The demonstrated different electrode shapes are used to actuate different resonance modes. Grey and black areas marked on top images represents electrodes with opposite polarity. Achieved mode shapes are presented for each electrode design.
and sensitivity for both readout methods. This work found that piezoresistive sensors have better performance for sub-micron thick cantilevers while piezoelectric sensors are better for thicker cantilevers. Additionally, piezoelectric sensors have significantly higher variation in resolution caused by fabrication and operating conditions (up to 30%). Despite these constraints, both piezoresistive and piezoelectric readout systems allow sensing with pico-newton/sub-nanometre accuracy.

2.5.3 Capacitive AFM probe displacement readout

Capacitive readout of displacement can be realised in two ways. One approach is to use a simple parallel plate capacitor created between the moving cantilever and an external co-planar electrode located near the cantilever. Alternatively, one can use fringing capacitance, which relates to the capacitance between two electrodes located within the same planar surface that will change if any object is located in proximity to that surface.

In 1992, Brugger et al. [48] presented an AFM cantilever probe with capacitive readout. The presented system used a static silicon electrode located parallel to the cantilever. The cantilever itself acted as the second, movable electrode as shown in Figure 2.13. The electrodes create a parallel plate capacitor where the capacitance varies with changes in the gap between the electrodes. The capacitance between two electrodes was measured to determine the deflection of the cantilever. The initial separation between electrodes was selected as 1.5 μm. This value for the electrode separation was a compromise between sensitivity and reliability. A smaller gap between electrodes would result in increased sensitivity, but at the same time, it would increase the risk of snapping-down the cantilever to the static electrode. Brugger et al. [48] demonstrated dynamic measurements with cantilever deflections on the order of a few hundred nanometres. The capacitive readout, due to difficulties in fabrication and with no significant improvements in sensitivity above other electrical readout methods, is not popular in AFM systems. However, capacitive sensing is commonly used as part of the internal feedback loop for piezoelectric AFM stages, where the capacitance measurement is used to increase the accuracy of 3-axis piezoelectric stage movement allowing significant improvement over previously used operation without an internal feedback loop.

![Figure 2.13: SEM image of the microfabricated AFM capacitive sensor presented by Brugger et al. [48]. The sensor uses parallel plate capacitance developed between the moving cantilever and the static silicon arm.](image-url)
Another capacitive approach, presented in 1998 by Chen and Luo [49] used fringing capacitance-based sensing for proximity sensors. Their paper presents a comparison between standard parallel plate capacitive sensing, as used by Brugger et al. [48], and sensing based on fringing capacitance as shown in Figure 2.14. The fringing capacitance uses two in-plane electrodes with an isolator between them. The moving cantilever disturbs the electric field between two electrodes decreasing the capacitance as it moves closer to them. When the gap between electrodes and cantilever increases, the capacitance between electrodes also increases. The significant advantage of fringing capacitance sensors above the standard parallel plate approach is the possibility to perform distance measurement between the static electrode and an AFM cantilever, which can be either conductive or non-conductive. The sensors demonstrated by Chen and Luo [49] had a linear response over several micrometers of cantilever motion. This characteristic can be useful in AFM sensors where the larger gap between the cantilever and the static capacitive electrodes allows minimising the risk of snapping-down the cantilever; however, one must be prepared to sacrifice sensitivity.

2.5.4 Quartz tuning fork (QTF)

Devices based on quartz tuning forks need to be classified as a separate group of probes for AFM, although they use the piezoelectric effect. The tuning fork is a U-shaped mechanical structure which resonates at a specific and constant frequency. Tuning fork oscillators

![Figure 2.14: An adapted graph shows a comparison between the operation of parallel plate and fringing capacitance proximity sensors presented by Chen and Luo [49]. The parallel plate capacitive sensors use two electrodes, one is static located behind the cantilever, and the other movable is a part of the cantilever. The fringing capacitance proximity sensors use two in-plane electrodes, separated by dielectric. The parallel plate sensors provide higher sensitivity for short distances between electrodes. The fringing capacitance proximity sensors provide linear response for a significantly higher dynamic range.](image)
are typically made from quartz, which is a piezoelectric material, and have two electrodes which can be used to both actuate and readout movement of the device. Using a reverse piezoelectric effect, a tuning fork can be set into vibration at its resonant frequency, which can subsequently be monitored piezoelectrically. The resonant frequency of the tuning fork depends on the length of the arms. Figure 2.15 shows a quartz tuning fork design, that was historically first developed to be used as stable oscillators for electronic circuits with the resonant frequency of 32.768 kHz and high quality factors, reaching values of 10,000 in air [50, 51]. For AFM measurements, the sharp tip is attached to one of the arms. The mass of the tip is low in comparison to the mass of a tuning fork arm, so it causes only a small change in the resonant frequency and Q-factor. Surface topology can be measured based on the change in the resonant frequency of the tuning fork during tapping-mode scanning along the sample surface due to AFM tip-sample interaction forces. The most significant limitation of tuning fork AFM probes is the limited scanning speed, with Edwards et al. [9] demonstrating operation at 1 Hz per line, which is similar to standard AFM systems, but it is significantly lower than high-speed or video rate AFM operation, which allows reduced scanning time.

![Diagram of quartz tuning fork with attached AFM tip](image)

**Figure 2.15:** Adapted schematic of quartz tuning fork with attached AFM tip to one of its arms for atomic force microscopy presented by Edwards et al. [9]. The interaction force between tip and sample surface modulates the oscillation frequency of the tuning fork.

Günther et al. [52] used a high-Q quartz resonator (quartz tuning fork) with a sharp tip interacting with sample’s surface for surface mapping using scanning near-field acoustic microscopy (SNAM). When the tip is in proximity to the surface, the resonant frequency and amplitude of the vibration change due to hydrodynamic forces in the gas between the tip and the sample. Günther et al. [52] generated surface topology maps achieving a vertical resolution of 5 nm in their first experiments.

Edwards et al. [9] presented the use of a quartz tuning fork with an attached AFM tip in an AFM operating in standard AFM tapping mode. This study achieved noise levels, resolution, and scan rates comparable to other AFM devices. Rensen and Hulst [10] used a quartz tuning fork with an attached silicon cantilever with a sharp tip for non-contact atomic force microscopy. They presented measurement results resolving 0.33 nm high atomic steps on a Si(111) sample with a noise level of 0.09 nm, showing that in terms of sensitivity the tuning fork approach can compete with optical position readout systems. In 2012, Babic et al. [11] presented a metrological scanning probe microscopy (mSPM) where they used a quartz tuning fork with the attached tip as a surface imaging sensor. The mSPM operates in tapping mode within the non-contact interaction region. The AFM tip
oscillates with an amplitude of 89 nm at a frequency of 32.565 kHz. The mSPM provided position measurement uncertainty below 1 nm due to the use of several interferometers, and use of advanced mechanical construction (metrological frame), enabling minimisation of the measurement uncertainty, as shown in Figure 2.16.

2.5.5 Integrated optical readouts

Integrated optical readout techniques are a subset of self-sensing displacement technologies that do not require any external bulk optics, and where the optical readout is an integral part of the sensing probe. Therefore, they do not require any alignment before use in comparison to standard optical readouts which include optical beam deflection (OBD) and interferometric readout. The fibre top cantilever deflection optical readout was demonstrated by Iannuzzi et al. [53–55] followed by Li et al. [56, 57], where they used a cantilever carved across the face of an optical fibre as shown in Figure 2.17. The interferometric cavity between the cantilever and the fibre face was exploited to measure the deflection of the cantilever. The difference between the solution of Iannuzzi et al. [53–55] followed by Li et al. [56, 57] and standard fibre-optics interferometric readouts is the integration of the probe with the optical fibre, which allows the reduction of common mode noise, thereby increasing the reliability and allowing use in extreme environmental conditions. Probe integration with an optical fibre eliminates all difficulties of interconnects between light source, probe and receiver, which are present in other integrated optical readouts. However, high volume fabrication of the AFM probes after Li et al. [56, 57] may be impractical, as they require fabrication using a focused ion beam (FIB).
Figure 2.17: SEM images of the fabrication process for a cantilever on the face of an optical fibre presented by Iannuzzi et al. [53–55]. The fabrication process starts from (a) removing material on the sides of the cantilever, and then (b,c) under and from the top of beam, creating a double supported structure (d). In the next step the AFM tip is created (e) and finally the device is released by removing material under the free-end of the cantilever. The fibre-optics interferometer is used to measure deflection of the cantilever during measurements.

In 2006 Onaran et al. [58] presented an AFM sensor with an integrated diffraction based interferometer. The AFM tip was located on top of a micromachined membrane as shown in Figure 2.18(a). The sensor uses an aluminium membrane suspended above a diffraction grating, which is also the second electrode for aluminium membrane actuation. Figure 2.18(b) presents the principle of operation of that device schematically. Laser light illuminates the membrane through the substrate, where the light is reflected back from the membrane towards the resonant cavity created between the substrate and the above suspended membrane. The signal amplitude of +1st order diffracted light is recorded as the sensor response. The authors demonstrated the capability of fast scanning and achieved a significant increase in image quality over standard AFM commercial cantilevers with OBD readout. This approach can provide an excellent alternative to OBD readout, since it is capable of eliminating unwanted light reflections from the sample and with the potential for high-speed imaging. The authors presented results of imaging with a scan rate up to 60 Hz and claim that significantly higher imaging speed is possible after upgrading other elements of the imaging system. This method was demonstrated using a standard external AFM light source and detector. However, it should be possible to fully integrate this optical readout approach on a chip. Using a membrane allows many flexural modes, which may add useful information if they can be discriminated using multiple detectors.

Liu et al. [59] and An et al. [60] presented an alternative approach to optical readout, which is suitable for fast scanning AFM. The adopted approach is presented in Figure 2.19. The optomechanical transducer uses a microdisk resonator to sense cantilever movement. The light is coupled in and coupled out from microdisk optical resonator using a waveguide
suspended near the disc. The AFM probe is located on the other side of the microdisk.
The AFM probe is attached to two anchors located far from the microdisk to minimise
their influence on the measurements. When the microcantilever moves, it influences the
optical evanescent field between the microdisk resonator and the optical fibre, which then
modulates the amplitude of the output optical signal. Using the proposed structure, An
et al. [60] presented results where a readout noise of 7 fm/√Hz was achieved with operation
of the AFM in contact mode, demonstrating an AFM topography image with an average
noise level of 20 pm RMS.

Figure 2.19: Optomechanical transducer-based nanoscale cantilever
presented by Liu et al. [59] and An et al. [60]. (a) The presented device
uses a suspended waveguide to couple light to a microdisk resonator
using the evanescent field. The AFM probe is located on the other side of
the of the microdisk. The AFM probe is attached to two anchors located
far from the microdisk to minimise their influence on the measurements.
When the microcantilever moves, it influences the optical field around
the microdisk resonator and then modulates the amplitude of the output
optical signal. Image (b) shows the SEM image of a fabricated microdisk
resonator and the AFM probe suspended near its surface.
2.5.6 Integrated on-chip optical readout – LumiMEMS™ (this work)

This work uses a silicon photonics waveguide with read-out Bragg gratings integrated on-chip with the AFM probe [17]. The presented technique is based on an optical resonant cavity interferometric measurement of cantilever deflection. The proposed concept for optical readout is schematically shown in Figure 2.20. The optical readout is built using a silicon photonics waveguide fabricated using a silicon-on-insulator substrate via standard silicon photonics processing [61]. A distributed Bragg grating (DBG) is etched into the waveguide. The suspended micro-cantilever fabricated above the DBG has a metal reflective surface facing the DBG, and the gap between the DBG and metal surface forms an optically resonant cavity. Part of the incident light propagating in the waveguide is coupled out by the DBG towards the cantilever. Light reflected by the cantilever metal surface returns to the DBG and is re-coupled into the waveguide. Interference between (a) light traversing the gap, and (b) light remaining in the waveguide, modulates the optical power in the waveguide. The interaction between the scanned sample and the AFM tip modifies the gap, allowing the surface topography to be imaged by monitoring the optical signal transmitted through the waveguide. In this work we will demonstrate that this technique has the potential to achieve noise levels an order of magnitude lower than state-of-the-art self-sensing electrical AFM probes [15, 37] and allows for AFMs to be fabricated using industry standard surface micromachining techniques.

![Figure 2.20: Isometric view of integrated on-chip cantilever deflection readout with the interrogating waveguide structure. Topography of a surface scanned by the tip modulates the amplitude of the waveguided optical signal via changes in the separation between the grating and the cantilever.](image-url)
The integrated on-chip optical readout being adopted in this work is referred throughout the thesis as LumiMEMS™ [62] as termed by industry partner, Panorama Synergy Ltd\(^1\). The LumiMEMS™ readout was invented in 2012 at the University of Western Australia and patented [63, 64] as a platform for cantilever sensors. The development of this technology is carried out in cooperation with Panorama Synergy Ltd, which provided support for development and commercialisation of the integrated on-chip sensors.

### 2.6 Summary

This chapter begins with an introduction to AFM and then follows with a historical account of the different methods used to measure cantilever displacement, with special emphasis on self-sensing techniques. The position of the sensing cantilever for AFM applications can be monitored via several different methods, and most of them can be integrated with the sensing probe (self-sensing). The most popular in commercial AFM systems is the optical beam deflection (OBD) readout, where the probe can readily be replaced and tailored to suit the application. The OBD readout can achieve low noise and high sensitivity, but it is difficult to use with very small cantilevers, which are required as we move towards large arrays of sensors. The alternative optical readout to OBD uses interferometry, which can achieve the same sensitivity as OBD readouts, without requiring free space optics used in OBD readouts. However, the interferometric approach is not popular in commercial tools as it can be difficult to align, and it does not provide any performance improvement above an optimally implemented OBD readout.

Table 2.1 presents a comparison between the several different self-sensing readout approaches investigated in this chapter, and lists the available parameters of several devices utilising that readout, thereby allowing a direct comparison of the available readout methods used in AFM systems. The self-sensing readouts do not require external elements, like free space optics in OBD or optical fibres in the interferometric readout. The electrical self-sensing displacement methods range widely and include piezoresistive and piezoelectric techniques. Both can be easily integrated with currently available AFM systems without significant modifications of the tools. In particular, piezoresistive readouts are currently accessible in commercial systems where highest accuracy can be sacrificed for the added advantages of self-sensing cantilevers and with the possibility to scan with multiple parallel probes at the same time. The capacitive readout presented in the literature was not further developed for AFM sensing as it does not show any advantages above other electrical readout methods currently available. Electrical self-sensing technologies using a quartz tuning fork AFM probe have been shown to provide ultimate high sensitivity due to its high spring constant (>1000 N/m) and high quality factor [65]. Quartz tuning fork sensors are commonly used in AFM systems as they have very high readout precision. However, their operating speed is limited, so they are not suited to new, fast scanning AFMs for imaging applications. Quartz tuning fork AFM probes can be found in metrological scanning probe

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\(^1\)See: [http://www.panoramasynergy.com/](http://www.panoramasynergy.com/)
microscopy (mSPM) [11] and all other AFM systems which require ultimate sensitivity; however, the trade off is a sacrifice in scanning speed.

The integrated optical interferometric readout presented by Iannuzzi et al. [53–55] and Li et al. [56, 57] has an extremely simple construction that can provide reliability in extreme conditions, e.g. temperature sensors operating from 20 °C to 500 °C. However, the fabrication process might be challenging to implement in high volume production. The work presented by Onaran et al. [58] can form the basis for reliable integration of AFM probes capable of fast scanning. The optical readout presented by Liu et al. [59] and An et al. [60] is competitive with our approach in noise performance as we will demonstrate in Chapter 6. The primary disadvantage of Liu and An’s technology may be limited robustness of the sensor, since it appears that the AFM tip can be easily damaged due to excessive shear forces. Furthermore, the presented fabrication process is complicated and requires multiple non-traditional fabrication steps, which may be difficult or impossible to perform in a commercial MEMS fabrication facility. Our integrated on-chip interferometric readout provides an alternative approach to AFM fabrication, where the whole process can be undertaken using standard silicon photonics and MEMS surface micromachining processes, which provides compatibility with currently used fabrication techniques. As we will show, the predicted performance is better than most state-of-art self-sensing probes.
### Table 2.1: Comparison of several selected integrated AFM sensor readout methods

<table>
<thead>
<tr>
<th>Buining et al. [1]</th>
<th>Martin et al. [26]</th>
<th>Edwards et al. [9]</th>
<th>Ouasan et al. [58]</th>
<th>Liu et al. [59], An et al. [60]</th>
<th>Moore et al. [47]</th>
<th>Dukic et al. [37]</th>
<th>LumMEMS™ (this work)</th>
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<tbody>
<tr>
<td>Readout method</td>
<td>Optical beam</td>
<td>External optical</td>
<td>Piezoelectric</td>
<td>Optical (interferometric)</td>
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<td>10</td>
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<td>7</td>
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<td>Low</td>
<td>Medium</td>
<td>Medium</td>
<td>Low</td>
<td>Medium</td>
</tr>
</tbody>
</table>

²reported as possible value
³estimated DND value from provided graph for C4 M4 voltage driven arrangement
⁴estimated DND value from reported RMS noise of 0.3 Å for 20 kHz bandwidth; limited by measurement system
⁵theoretical noise limit reported by Putrino et al. [13]
⁶post-fabrication tip attachment required
⁷low cost: bulk micromanufacturing, surface micromachining; high cost: focussed ion beam processing. The cost is very sensitive to production volume. If the volume is high, the costs can be low, depending also on yield per wafer.
⁸low cost: electronics only, medium cost: fibre optics, high cost: free-space optics
Chapter 3

Design of AFM readout module

This chapter begins with the performance analysis of a proposed AFM probe deflection read-out using a MEMS micromachined cantilever integrated with interferometric readout using a silicon photonics chip. Coupling light into and out of the photonics chip has a potential impact on the suitability of this approach to a low-cost AFM and, accordingly, the second part of this chapter investigates three different light coupling methods. The last part of this chapter presents the mechanical design of the AFM cantilever used in this thesis followed by the theoretical noise analysis of the interferometric readout.

As outlined in Chapter 2, optical read-outs and in particular OBD are commonly used in commercial AFM systems. They simultaneously provide ultimate sensitivity with high imaging speeds and the use of low costs cantilever probes. The optical read-outs of cantilever deflection can be divided into two main techniques: OBD and interferometric. Putman et al. [66] have shown that both techniques can theoretically achieve the same noise level for conditions ideal to each approach. The interferometric read-out has two requirements to achieve a sensitivity comparable to OBD readouts: (i) the reference and measurement light need to overlap completely to create the ideal interferometric effect, (ii) the phase change between the reference and measurement light paths due to the cantilever displacement must be maximal to achieve optimum sensitivity. Fulfilling these requirements for the interferometric read-out has hitherto posed to be technically difficult, and consequently OBD read-out is the primary approach in commercial systems.

Cantilever deflection read-out presented in this work uses a silicon photonics chip with a buried single mode waveguide and interrogating Bragg grating (see Figure 2.20). Interferometry within the waveguide is used to measure the deflection of the cantilever via a change in the optical path length between light remaining in the waveguide and the light coupled out of the grating, which is reflected by a metal mirror deposited on the underside of the cantilever, and then coupled back into the waveguide by the same grating. For this work, we used an existing silicon photonics chip designed by Putrino [67]. Each waveguide contains three gratings: input, interrogating and output. The gratings are connected in series using a single-mode waveguide and adiabatic tapers [68] as shown in Figure 3.1. The adiabatic tapers allow connecting narrow waveguides and wide gratings with low optical loss. The large area of the gratings allows for greater alignment tolerances between
Figure 3.1: Top view schematic of the silicon photonics waveguide with gratings and the cantilever suspended above the interrogating grating. The output optical signal is modulated by the cantilever deflection. Image adapted from Putrino [67].

single mode optical fibres and the photonics chip for input and output light coupling. The maximised area of the interrogating grating allows high efficiency of the interferometric measurement of the cantilever deflection.

Our integrated on-chip optical interferometric read-out fulfils all of Putman’s [66] requirements for the interferometric read-out: (i) the optical beams perfectly overlap within the single mode waveguide; (ii) the change between the two beams is guaranteed to be maximal as the readout beam reflected from the cantilever is only a function of the cantilever to the grating gap.

The sensitivity of our device depends on several factors such as grating and reflector design, laser input power and light coupling efficiency. However, there are two additional aspects associated with the mechanical design of the cantilever: (i) the distance between the interrogating grating and reflective backside of the cantilever; and (ii) the parallelism between the reflective mirror and the cantilever. Additionally, the cantilever reflective area needs to cover the grating area to avoid additional light losses.

3.1 Analytical model of readout operation

An analytical model of an AFM interferometric readout based on fibre optics was presented by Rasool et al. [69]. The optical fibre interferometer as depicted in Figure 3.2(a) uses the interferometric effect between the portion of light reflected from the fibre end-face (glass-air interface) and the portion of light reflected from the cantilever. The interference effect can be described using the equation for an ideal Fabry-Pérot interferometer (FPI) [70]:

$$\frac{P_r}{P_i} = \frac{F \sin^2 \frac{\delta}{2}}{1 + F \sin^2 \frac{\delta}{2}}$$  \hspace{1cm} (3.1)

where the parameters $F$ and $\delta$ are defined as:

$$F = \frac{4R}{(1 - R)^2}$$ \hspace{1cm} (3.2)
Figure 3.2: The comparison between (a) fibre optics interferometric readout after Rasool et al. [69] and (b) our integrated on-chip interferometric readout of cantilever deflection after Putrino [67]. Our approach has a more complex optical path to determine cantilever deflection.

\[ \delta = \frac{4 \pi n_{\text{gap}} z}{\lambda} \]  

(3.3)

where \( P_i \) is incident power, \( P_r \) is reflected power, \( R \) is reflectivity, \( n_{\text{gap}} \) is refraction index of the medium between the surfaces, \( \lambda \) is wavelength of the light source in vacuum, and \( z \) is the distance between the surfaces. Conceptually, our interferometric readout can also be simplified to the FPI as presented in Figure 3.2(b). Our interrogating grating is symmetrical, which means that only some light is coupled out towards the cantilever, while the other is coupled out towards the substrate, where some can be reflected back towards the surface by the interface between the waveguide and the bottom oxide layer. The portion of light coupled towards the substrate can be almost completely reflected upwards by use of a distributed Bragg reflector (DBR) [71] or metal mirror [72–74] on the underside of the bottom oxide layer. The thickness of the bottom oxide layer can also be optimised to maximise usage of this light. We do not include these grating optical losses in the theoretical analysis, since the grating waveguide interaction makes the analysis less amenable to a closed-form solution and, furthermore, these losses are more readily treated in a finite element analysis (see below).

Equation 3.1 is valid for a classical fibre optics interferometer where the light is reflected. In our readout we monitor transmitted signal modulated by the interferometric effect. That signal can be described with following equation [70]:

\[ \frac{P_r}{P_i} = \frac{1}{1 + F \sin^2 \frac{\delta}{2}} \]  

(3.4)

where \( P_t \) is transmitted power. For an ideal case the reflected and transmitted light ratio described by Equations 3.1 and 3.4 are complementary [70], such that:

\[ \frac{P_r}{P_i} + \frac{P_t}{P_i} = 1 \]  

(3.5)
However, in our case we need to include additional losses ($\alpha$) in the transmission equation, not considered in the above analytical solution, which will allow matching the calculated values with simulation results by an associated shift on the vertical axis. Consequently, Equation 3.5 becomes:

$$\frac{P_r}{P_i} + \frac{P_t}{P_i} = 1 - \alpha$$

(3.6)

The losses of the interrogating grating, $\alpha$, are defined in a range of 0 to 1 where 0 means no additional losses are introduced by the interrogating grating and 1 means that all available light is lost. The finesse of the interferometric cavity, $F$, can be estimated using the following equation [70]:

$$F = \frac{\pi \sqrt{F}}{2}$$

(3.7)

The optical path length of light in our readout previously described by Putrino [67], consists of two components: part of the light remaining in the optical waveguide $L_r$ and part of light coupled-out from the grating and reflected back from the cantilever $L_d$, which can be described using the following equations:

$$L_r = n_{eff} \Delta x = 2n_{eff} G \tan \theta$$

(3.8)

$$L_d = n_{gap} D = 2n_{gap} G \cos \theta$$

(3.9)

where $\theta \approx 10^\circ$ is the coupling angle of our grating, $G$ is separation between the interrogating grating and the cantilever, and $n_{eff} = 2.83$ is the effective refractive index of the waveguide TE mode [75], $\Delta x$ is the length of the path of the light remaining in the waveguide, $n_{gap}$ is the refractive index of the medium in the cavity between the grating and the cantilever (equal 1 for air), and $D$ is the length of the light path within the cavity (see Figure 3.2).

The TM mode is not guided in our silicon photonics chip and is not considered in this work [67]. The length of the light path of interest in our readout is equal to the difference between the length of optical paths of the part of light coupled-out from the grating and reflected back from the cantilever $L_d$ and the part of the light remaining in the optical fibre $L_r$:

$$z = L_d - L_r = \frac{2n_{gap} G}{\cos \theta} - 2n_{eff} G \tan \theta$$

(3.10)

The calculated transfer function for the gap up to 9 µm is presented in Figure 3.3. For this calculation we used $R = 0.48$, $\theta = 10.6^\circ$ and $\alpha = 0.12$, in which case the finesse of the cavity is approximately $F = 4$. Effectively, the interferometric effect modulates the light power transmission through the waveguide as a function of the separation between the cantilever and grating, with the periodic peaks indicating constructive interference and the nulls indicating destructive interference. The periodicity is about $\lambda / (2 \cos \theta)$, as expected from Equation 3.10.
3.2 Sensitivity of interrogating grating for cantilever displacement

Our silicon photonics chip utilises gratings with a period of 630 nm and a fill factor of 0.5, which means that each etched trench is 315 nm wide. The silicon photonics chip operation was previously simulated for devices with the distance between the interrogating grating and cantilever surface up to 7 μm [17]. When the gap is small, squeezed film damping reduces the cantilever Q-factor [76] and increases the risk for cantilever snap-down when used as an AFM. Gaps of 8 μm allow sufficient working distance for a practical AFM cantilever and significantly improve the Q-factor [76]. To verify that this gap does not compromise the performance, we performed a finite-difference time-domain (FDTD) simulation of the light propagation for the different separation values around 8 μm between the interrogating grating and the cantilever, using MEEP software [77]. Figure 3.4 shows the optical field distributions for propagated 1550 nm light for the cantilever-waveguide separations required to create constructive (see Figure 3.4(a)) and destructive interference (see Figure 3.4(b)). During the constructive interference case, most of the light is guided, and during the destructive interference case, the light couples into substrate modes and is no longer guided.

Figure 3.5 shows the simulated signal level as a function of the gap between the interrogating grating and the cantilever. The interferometric effect can be observed as the periodic response repeating approximately every λ/(2 cos θ) where λ = 1550 nm, θ = 10.6°, and taking into account the refractive index of the different light paths. The sensitivity of the read-out is given by the slope of the linear region of the response. The extent
Figure 3.4: Finite-difference time-domain (FDTD) simulations of the optical field distributions for propagated 1550 nm light inside silicon photonics chip for the cantilever-waveguide separations required to create constructive (a) and destructive (b) interference. During the constructive interference case, the light is guided through the output waveguide towards the detector, while during the destructive interference case, the light couples into substrate modes and is dissipated.

of the linear region of the slope defines the dynamic range. The dynamic range can be extended by the use of two (or more) laser sources with slightly different wavelengths [17]. Figure 3.5 shows the fitted theoretical solution (Equation 3.4) to the simulated graph for the highest and lowest peaks, which allowed us to estimate the finesse of the cavity for our devices, including losses introduced by larger gaps between the interrogating grating and the cantilever. We find that the finesse for the lowest gap was $\mathcal{F} = 4.2$ while for the highest investigated gaps (above 7 $\mu$m) the finesse drops to $\mathcal{F} = 1.5$. The achieved results show that there is good agreement between the simple theoretical model and the MEEP simulations in the shape and periodicity, particularly for smaller gaps.
3.3 Light coupling to silicon photonics chip

The passive silicon photonics chip requires light coupling from a laser source to the chip and from the chip to a detector, both using the existing grating couplers fabricated on the photonics chip. Although current technology allows integrating an on-chip light source and detector [78], this was beyond the scope of this work. In this project, we used optical fibres to couple laser light to the photonics chip and from the chip to a low dark current photodetector. There are several approaches that can be used for light coupling to and from the silicon photonics chip.

3.3.1 Optical fibre from the top

Light coupling using optical fibres aligned to the input and output gratings from the top side of the photonics chip was previously implemented on our silicon photonics chips by Putrino [67]. That approach consists of two optical fibres aligned at the designed grating coupling angle (about 10° from vertical) as shown in Figure 3.6(a). The distance between the optical fibre and the chip surface also needs to be minimised to reduce light losses. The position on the photonics chip surface and the angle of optical fibres can be tuned during the X-Y-Z fibre alignment process using micro-manipulators. For an AFM probe, physical contact between cantilever probe and the measured sample is required and the
sample also needs X-Y-Z manipulation over the tip and, accordingly, the region around the tip needs to be free from obstructions, which the original coupling technique did not allow. The potential solutions to the obstruction issue are presented below and include light coupling from the back-side of the silicon photonics chip and optical fibres mounted horizontally on the chip.

### 3.3.2 Optical fibre from the substrate side

When a reflector is placed above the gratings, light is efficiently coupled into and out of grating couplers from the substrate side [72] (see Figure 3.6(b)). This approach can be realised in two ways: through the silicon wafer as shown in Figure 3.7(a) which is effectively transparent at 1550 nm; or with a section of the silicon wafer removed, leaving gratings suspended inside the silicon oxide membrane as shown in Figure 3.7(b). Light coupling through the silicon wafer requires fabrication of carefully aligned microlenses at the back-side of the silicon photonics wafer, or the use of optics at the end of the fibre, such as graded index (GRIN) collimators or lenses, to focus light onto the gratings through
3.3. Light coupling to silicon photonics chip

![Diagram showing light coupling through the back side of a silicon photonics chip.](image)

**Figure 3.7:** Light coupling through the back side of silicon photonics chip using (a) focused/collimated light from the optical fibre at the input and a large area photodetector at the output or (b) using two optical fibres located in the openings etched through the silicon substrate.

The relatively thick silicon wafer. The output light can be captured using a large area photodetector attached directly to the back-side of the silicon wafer.

The first approach, with the lens or collimator at the input and large area photodetector at the output was not further investigated in this work, as it is challenging to align the input optical fibre to the photonics chip from the substrate side without any alignment features. Additionally, optical fibre lenses and light collimators may be difficult to permanently attach to the chip after alignment, which is also difficult without alignment features.

The second approach with the opening in the silicon substrate was more thoroughly investigated. When the silicon wafer below the input and output gratings is removed, we can couple light to the photonics chip from the back-side, using optical fibres similar to the approach described above for coupling from the top side of the wafer. That approach requires precise lithography allowing mask alignment to the back-side of the wafer. Then, the silicon can be removed using a deep silicon processes (e.g. Bosch process [79–81] or wet silicon etching [82]). However, the most significant risk is related to the alignment and gluing of the optical fibres inside the openings. The fibres need to be located in close proximity to the fragile silicon oxide membrane. Any vertical misalignment of the optical fibre may cause perforation of the membrane and destroy the silicon photonics chip. In addition, the fabricated silicon photonics wafer consists of several layers, which are developed to compensate substrate intrinsic stress developed during the chip fabrication. This balance no longer exists when the silicon substrate is removed. The suspended membrane needs to have neutral or tensile stress to avoid buckling after release and hence it may be necessary to tune the resultant stress of the remaining membrane by the deposition of a compensation layer on the top surface of the membrane before removal of the silicon substrate. As it was not possible in our facility to align the back-side etch mask necessary for locating the substrate openings, coupling through the back of the silicon wafer was also not further investigated as a part of this work.

For the final device, we used an alternative approach, with angle polished optical fibres attached horizontally to the top side of the silicon photonics chip.
Figure 3.8: Schematic cross-section of the angle polished optical fibre used to couple light to the silicon photonics chip after Snyder and O'Brien [83]. The optical fibre is placed on the silicon photonics chip surface and aligned to the input grating. Laser light guided inside the optical fibre core is reflected from the angle polished fibre face towards the grating at an angle of 10° from vertical and coupled to the silicon photonics planar waveguide. The same effect occurs in reverse at the output grating from the planar waveguide.

3.3.3 Angle-polished optical fibres

Light coupling from angle polished optical fibres to a silicon photonics chip was extensively investigated for the packaging of silicon photonics chips [75, 83, 84]. The approach presented in Figure 3.6(c) requires two optical fibres, one for the input and one for the output, with the fibre end-face polished at an angle of approximately 40° for gratings designed for a 10° coupling. Light transmitted through the optical fibre core is totally internally reflected from the polished face of the optical fibre towards the input grating and coupled to the planar waveguide at the optimal angle of 10° from vertical, as shown in Figure 3.8. The same process occurs at the output from the silicon photonics chip. After the polished fibres are aligned to the gratings, they can be permanently glued in place using index matching glue.

The current design of the photonics chip used to demonstrate a working prototype, utilises 5 mm long straight waveguides between input and output gratings with an interrogating grating in the middle. Optical fibres of 250 µm in diameter are attached to the front side of the photonics chip and aligned to the ends of that waveguide. This effectively limits the sample size that can be imaged using the current device. However, the sample access can be further improved by changing the design of the silicon photonics chip and placing input and output gratings near one edge of the chip, far from the interrogating grating and the AFM tip. Additionally, using an optimised waveguide geometry and/or a slight angle during the imaging process, between the cantilever and the sample surface planes, removes the current limitation on the sample size as shown in Figure 3.9.

Other methods of coupling light into the waveguides are possible, such as tapered waveguides, but required corresponding tapers on the silicon waveguides which were not incorporated on the photonics chip.
3.4 Mechanical design of the cantilever

The aim of this design is an AFM cantilever probe, which will be able to perform sample imaging in contact mode. It is known that AFM cantilevers designed for contact mode operation should have a spring constant up to 1 N/m [85]. We decided to design an AFM probe with a spring constant in the range of 0.6 N/m to 0.8 N/m, allowing for process variations during the fabrication process. We use a rectangular cantilever made of silicon nitride and covered on both sides with a gold thin film (50 nm each layer). The gold thin films have three functions: (1) bottom-side thin gold film facing the interrogating grating acts as a mirror for the read-out (reflectance for gold at 1550 nm is 97.5% [86]); (2) the top-side thin gold film provides a conductive surface for subsequent FIB processing (tip placement: see Section 5.2.3); (3) the symmetrical structure also provides temperature related stress compensation of the cantilever. The spring constant of a rectangular cantilever can be calculated using the following equation, which is valid for deflections negligible with respect to the cantilever length [85]:

$$k = \frac{Ewtf^3}{4l^3} \quad (3.11)$$

where $E$ is Young’s modulus of the cantilever material, and $w$ is width, $l$ is length, $t$ is thickness of the cantilever beam. The Young’s modulus of silicon nitride can vary between 95 GPa [87] and 230 GPa [88], depending on the deposition parameters. Silicon nitride deposited in our facility using a standard in-house recipe has a Young’s modulus of 180 GPa, and that value is used in all calculations. However, the cantilever is coated with gold thin films, which also influence the spring constant. For the composite cantilever beam, which consists of more than one material, the effective Young’s modulus needs to be used. The effective Young’s modulus allows calculation of an ”average” value of Young’s modulus for the cantilever made with multiple materials (e.g. gold and silicon nitride). The effective Young’s modulus for our structure can be calculated using the following equation [89]:

$$E_0 = \frac{(E_1h_1^2 - E_2h_2^2)^2 + 4h_1h_2(h_1 + h_2)^2 E_1E_2}{(E_1h_1 + E_2h_2)(h_1 + h_2)^3} \quad (3.12)$$
where the silicon nitride Young’s modulus is $E_1 = 180$ GPa, and its thickness is $h_1 = 2$ µm, and the gold Young’s modulus is $E_2 = 79$ GPa [90], with a combined gold thickness of $h_2 = 100$ nm. This gives 165 GPa as the effective Young’s modulus of our beam, which allows calculation of the first resonant frequency of the cantilever using an equation given by Naeli and Brand [91]:

$$f_0 = \frac{1.875^2 \ t}{2\pi \sqrt{12 \ E_0}} \sqrt{\frac{E}{\rho}}$$

(3.13)

where $t$ is cantilever thickness, $l$ is cantilever length, $E = 165$ GPa is the effective Young’s modulus and $\rho = 3957$ kg/m$^3$ is the effective material density [90, 92]. The 1st resonant frequency is an important parameter of the cantilever, which is used later during Brownian motion calculations.

The dimensions of our cantilever are determined by three requirements: (1) spring constant of the cantilever, which should be lower than 1 N/m; (2) squeezed film damping (damping is proportional to the inverse square of the cantilever width [93, 94]); (3) size of the reflector over the grating and surface area for subsequent tip welding. The cantilever width needs to be larger than the grating length to reflect all the light back into the cavity. For our silicon photonics chip, the minimum cantilever width was 20 µm, providing complete coverage of the whole grating area, and allowing a small overhang for fabrication tolerances. We found that the width of 20 µm was also adequate for tip welding. Using the spring constant equation (3.11), we found that the dimensions of the cantilever of width 20 µm, length 205 µm and thickness 2.1 µm, gives a spring constant value of 0.89 N/m, as well as a resonant frequency of 52 kHz, which fulfils all three requirements presented above.

### 3.5 Out of plane cantilever deflection

The cantilevers presented in previous work [95, 96] do not allow the cantilever mirrors to be parallel to the interrogating grating surface after device release due to deflection of the cantilever at the anchor. The initial deflection of the cantilever is caused by: (i) spring effect of the anchors [97]; and additional; (ii) upwards bending due to the bimorph structure as shown in Figure 3.10. The spring effect of the anchor was demonstrated on two test structures. The first test cantilever was fabricated using a 700 nm thick silicon nitride thin film structural layer whereas the second test cantilever was fabricated with 1700 nm thick silicon nitride thin film as shown in Figure 3.10. Both structures demonstrated identical deflection angle at the anchoring point of the cantilever, which indicated that the deflection was caused by the spring effect of the anchor. There are several solutions available to address this issue.

Gill et al. [97] proposed a design which uses a reinforcement hump fabricated behind the cantilever anchor as shown in Figure 3.11. Their solution worked well for cantilevers fabricated using fully conformal polysilicon deposited using a low pressure chemical-vapour deposition (LPCVD) process where the initial deflection of the cantilever was reduced from 3% to 0.1%. The structural material for our cantilevers is silicon nitride deposited
3.5. Out of plane cantilever deflection

**Figure 3.10:** The cross-sectional profiles along the cantilever (A–A') with the anchor effect causing cantilever offset after device release demonstrated for the two thicknesses of the silicon nitride structural layer. The profiles were recorded using a Zygo NewView 7300 white light optical surface profilometer. The inset shows an SEM image of the measured cantilever.

**Figure 3.11:** The side view of the cantilevers with (a) standard step anchor and (b) enforced step anchor to minimise anchor effect proposed by Gill et al. [97].

Using an inductively-coupled plasma chemical-vapour deposition (ICPCVD) process which exhibits reduced deposition conformity, which would make it challenging to replicate results presented by Gill et al. [97].

To eliminate the spring effect of the anchor and resulting initial deflection of the cantilever, we adopted a design approach presented by Plaza et al. [98] using a T-shaped double anchor design. The T-shaped anchor consists of two large cylindrical top-hat anchors connected together with a large stiff microbeam and a cantilever attached to its side as shown in Figure 3.12(b). The microbeam was designed to have a high resonant frequency, significantly higher than the first and second order resonant frequency of the cantilever, thereby avoiding the influence of the beam vibration, and a wide microbeam was used to reduce beam torsional flexure modes on the AFM measurement results.
Figure 3.12: Models used for FEM simulations of the anchor influence on the cantilever properties. (a) Cantilever with perfect anchor, and (b) cantilever with T-shaped anchor.

Figure 3.12(a) shows the perfect anchor, while Figure 3.12(b) shows the simplified model of our double anchor used for FEM simulations. As presented by Plaza et al. [98], the T-shaped anchor has a significant influence on the mechanical parameters of the fabricated cantilever, and accordingly, we performed FEM simulations to estimate the resonant frequency and spring constant of our T-shaped anchor cantilever, and compared it with a FEM simulation on the same cantilever with a perfect anchor. The differences between the two simulations allow an estimation of the influence of the microbeam anchor on the spring constant of the cantilever. The simulations show that the double anchor results in a decreased resonant frequency of the cantilever from $f_{0S} = 52$ kHz to $f_{0T} = 45$ kHz and also causes a decrease in the spring constant. The resulting spring constant value for the double anchor structure is $k = 0.63$ N/m. Additionally, the further decrease of the resonant frequency is expected from due to the added mass of AFM tip installed on the free end of the cantilever, reducing the estimated value by approximately 3 kHz, and achieving a final value of 42 kHz. The values of the cantilever resonant frequency and spring constant for the T-shaped anchor cantilever estimated using FEM simulations vary from the analytically estimated value since numerical simulation includes the influence of the anchor structure on those parameters.

3.6 Noise analysis of our optical readout

As described in the literature review, minimum detectable deflection (MDD) and its derivative deflection noise density (DND), are metrics used to quantify the performance of AFMs, and they are related to the signal-to-noise ratio (SNR) of the optical interferometric measurement system. The sources of noise for the interferometric readout are: shot noise arising in the photodetector; receiver electronic noise; and Brownian motion of the mechanical structure, and these are considered in turn below. However, first we need to consider the expected optical power (the signal) in the photodetector.
3.6. Noise analysis of our optical readout

3.6.1 Optical path losses

Our optical path consists of the laser source, polarisation controller, silicon photonics chip and detector. Single mode optical fibres (SMF-28) with FC/PC connectors are used to connect all elements together. The optical system requires a minimum of three FC/PC or FC/APC connectors, with typical losses of 0.3 dB each, or 0.9 dB total. The fibre-to-fibre insertion losses (not including losses originating from the interrogating grating, which are considered in the next paragraph) for horizontally glued optical fibres are expected to be up to 10 dB, which includes 9 dB for perfect fibre coupling losses [83] and 1 dB for the silicon photonics waveguide losses [99]. Although the insertion losses for the grating couplers can be minimised [74], this was not further investigated.

Our readout should operate at approximately mid-way on the interferometric slope to achieve linearity and maximum dynamic range of cantilever deflection, which means that the interrogating readout will introduce an additional 3 dB loss to the optical path, with an additional 3 dB loss due to device operation with a separation greater than 6 μm between interrogating grating and cantilever, as shown in Figure 3.5. The minimum total losses of the system are then 17 dB using the optimal point of operation for AFM measurements. The maximum power of our laser source was about 11 mW, which then gives a possible maximum of 220 μW at the output from the chip for an ideal device, with additional losses likely due to less than perfect fibre polishing and alignment.

3.6.2 Readout noise

In the following noise treatment, all of the noise sources are referred to the optical receiver input to allow comparison with the photodetector current, and hence the calculation of the signal to noise ratio.

There are two approaches commonly used in the literature to calculate signal-to-noise ratio in an AFM. Both approaches produce the same relationships between noise signals originating from particular sources. The first approach to describing signal-to-noise ratios of optical signals measured by a photodetector, in this thesis will be called the optical signal-to-noise ratio (OSNR). This approach, presented by Kocabas and Aydinli [100], considers a signal as an RMS value of the current at the output of the photodetector \( \langle i_{\text{out}} \rangle \), which is equal to the optical power \( P_t \) at the output of the silicon photonics chip captured by the photodetector multiplied by the detector responsivity \( \gamma \). The optical signal-to-noise ratio (OSNR) can be calculated from the following equation:

\[
\text{OSNR} = \frac{\langle i_{\text{out}} \rangle^2}{\langle i_{\text{noise}} \rangle^2}
\]  

(3.14)

where \( \langle i_{\text{noise}} \rangle \) is RMS value of the considered noise. The second approach was presented by Yaralioglu et al. [24], where the signal is defined as a change of the photodetector current \( \Delta i_{\text{out}} \) with respect to the cantilever-grating separation \( \Delta z \). Then, the signal-to-noise ratio
Chapter 3. Design of AFM readout module

(SNR) can be calculated from the following equation:

\[
\text{SNR} = \frac{(\Delta i_{\text{out}}/\Delta z)^2}{\langle i_{\text{noise}} \rangle^2} \left[ \frac{1}{\text{m}^2} \right]
\] (3.15)

This approach is desirable for an AFM system as it includes the sensitivity of the system, and considers small signals directly related to the cantilever displacement which is relevant for evaluating AFM measurements. The investigation of SNR presented in this chapter is based on the approach presented by Yaralioglu et al. [24]. However, the results shown in Figure 3.13 present values calculated using both approaches by plotting signal-to-noise ratio (SNR) and optical signal-to-noise ratio (OSNR) for comparison on the same graph.

To calculate the SNR values, first we evaluate the transfer function of the cantilever to find the maximum slope using the derivative of the Equations 3.4 and 3.6 over the \( \delta \) as

\[
\frac{d}{d\delta} \left( \frac{P_t}{P_i} \right) = \frac{d}{d\delta} \left( \frac{1}{1 + F \sin^2 \frac{\delta}{2}} \right)
\] (3.16)

and using reflectivity \( R = 0.16 \) to obtain a good fit to the numerically simulated values for a gap greater than 6 \( \mu \)m (see Figure 3.5), the maximum slope of the transfer function corresponding to the maximum value of the derivative is found near the mid-point of the slope, giving efficiency of \( \eta = \frac{P_t}{P_i} = 0.26 \), which defines point of the maximum slope of the transfer function (optimal working point). Then, the readout sensitivity, \( S \), can be calculated for this maximum slope of the transfer function, defined of a change of efficiency, \( \Delta \eta \), over change of the gap, \( \Delta z \) [25] using Equation 3.3:

\[
\frac{\Delta P_t}{P_i} = \eta \cdot \Delta \delta = \eta \frac{4\pi n_{\text{gap}} \Delta z}{\lambda}
\] (3.17)

\[
S = \frac{\Delta \eta}{\Delta z} = \frac{1}{P_i} \frac{\Delta P_t}{\Delta z} = \eta \frac{4\pi n_{\text{gap}}}{\lambda} \left[ \frac{1}{\text{m}} \right]
\] (3.18)

where \( P_i \) is incident power, \( \Delta P_t \) is a change of the transmitted power, \( \eta \) is efficiency, \( n_{\text{gap}} \) is refractive index of the medium between the cantilever and the interrogating grating, and \( \lambda \) is wavelength of the light source in vacuum. For our readout operating at optimal working point, the readout sensitivity can be estimated using the efficiency \( \eta = 0.26 \), refraction index of the air \( n_{\text{gap}} = 1 \), and wavelength of the light source \( \lambda = 1550 \text{ nm} \), giving \( S = 2.11 \cdot 10^6 \text{m}^{-1} \). The output current \( i_{\text{out}} \), input power \( P_i \) and output power \( P_t \) are connected as follow:

\[
i_{\text{out}}(z) = \gamma P_i(z) = \gamma P_t \eta(z)
\] (3.19)

where \( \gamma = 1.0 \text{ A/W} \) is the detector responsivity and \( \eta \) is interrogating grating efficiency. The signal investigated in this section is defined as a change of the photodiode current.
\[ \frac{\Delta i_{\text{out}}}{\Delta z} = \gamma P_i \frac{\Delta \eta}{\Delta z} \]  

(3.20)

Using the definition of readout sensitivity \( S \), from Equation 3.18, the investigated signal is defined as follow:

\[ \frac{\Delta i_{\text{out}}}{\Delta z} = S P_i \gamma = \frac{S P_i \gamma}{\eta} \]  

(3.21)

Note that for a responsivity of unity, optical power and photodetector current can be interchanged magnitude-wise.

Shot noise arises in the photodetector and has a component directly related to the optical signal power. The shot noise of the photodetector, \( \langle i_s \rangle \), is given by [101]:

\[ \langle i_s \rangle = \sqrt{2e P_i \gamma \Delta f} \]  

(3.22)

where \( e = 1.602 \cdot 10^{-19} \) C is the elementary charge, \( P_i \) is the optical power on the detector, \( \gamma = 1.0 \) A/W is the detector responsivity, and \( \Delta f \) is the noise bandwidth. Then, the SNR of shot noise, \( \text{SNR}_s \), is given:

\[ \text{SNR}_s = \frac{(\Delta i_{\text{out}}/\Delta z)^2}{\langle i_s \rangle^2} = \frac{(SP_i \gamma)^2}{2e P_i \gamma \Delta f} = \frac{S^2 P_i \gamma}{2e \eta^2 \Delta f} \left[ \frac{1}{\text{m}^2} \right] \]  

(3.23)

The SNR of shot noise is plotted as a function of photodetector current in Figure 3.13.

The photodetector current requires amplification using a low noise receiver, and a transimpedance feedback amplifier is a good choice since it can offer both a low noise and a high dynamic range. For a well designed JFET input transimpedance amplifier operating in a narrow bandwidth, the input referred noise is dominated by the receiver transimpedance (feedback resistor) [102], and therefore we need to consider its thermal noise contribution. Johnson current noise, which represents the thermal noise current in the resistor, \( \langle i_{jn} \rangle \), is given by [103]:

\[ \langle i_{jn} \rangle = \sqrt{\frac{4k_B T \Delta f}{R_t}} \]  

(3.24)

where \( k_B \) is the Boltzmann constant, \( T \) is temperature, \( R_t \) is the feedback resistance (transimpedance) and \( \Delta f \) is the noise bandwidth. A transimpedance value commensurate with the range of expected output optical powers is \( R_t = 100 \) kΩ, giving a current sensitivity of \( 10^{-5} \) A/V. Then, the SNR of Johnson noise, \( \text{SNR}_{jn} \), is given:

\[ \text{SNR}_{jn} = \frac{(\Delta i_{\text{out}}/\Delta z)^2}{\langle i_{jn} \rangle^2} = \frac{R_t (SP_i \gamma)^2}{4k_B T \Delta f} = \frac{R_t (SP_i \gamma)^2}{4\eta^2 k_B T \Delta f} \left[ \frac{1}{\text{m}^2} \right] \]  

(3.25)

The signal-to-noise ratio (SNR) of Johnson noise is plotted as a function of photodetector current in Figure 3.13.
In our laboratory, it was often convenient to use the flexibility of a commercial, variable gain transimpedance amplifiers such as the Stanford Research Systems SR570 [104] and Femto DLPCA-200 [105], which covers a wide current sensitivity range and has noise performance as defined in the specification as noise current density of $i_{amp} = 2 \text{ pA/}\sqrt{\text{Hz}}$ for SR570 [104] and $i_{amp} = 0.45 \text{ pA/}\sqrt{\text{Hz}}$ for DLPCA-200 [105], both operating with current sensitivity of $10^{-5} \text{ A/V}$. The RMS value of amplifier current for a noise bandwidth $\Delta f$ is given:

$$
\langle i_{amp} \rangle = i_{amp} \cdot \sqrt{\Delta f}
$$

(3.26)

Then, the SNR of electronic noise of the amplifier, $SNR_{amp}$, is given:

$$
SNR_{amp} = \frac{(\Delta i_{out}/\Delta z)^2}{i_{amp}^2 \Delta f} = \frac{(SP_\gamma)^2}{i_{amp}^2 \eta^2 \Delta f} = \frac{(SP_\gamma)^2}{i_{amp}^2 \eta^2 \Delta f} \left[ \frac{1}{\text{m}^2} \right]
$$

(3.27)

The SNR for the Stanford Research Systems SR570 and Femto DLPCA-200 transimpedance amplifiers over a range of photodetector currents is shown in Figure 3.13.

Cantilevers exhibit thermomechanical (Brownian) noise, also called "random walk" noise caused by the random and unbalanced impact of molecules on the cantilever [106]. Furthermore, the Brownian noise exhibits a resonant response due to the mechanical second

---

**Figure 3.13:** Signal-to-noise ratio (SNR) and optical optical signal-to-noise ratio (OSNR) for the shot noise of the photodetector, Johnson noise of the amplifier with transimpedance of 100 kΩ, electronic noise of the Femto DLPCA-200 and the Stanford Research Systems SR570 transimpedance amplifiers for a current sensitivity of $10^{-5}$ as a function of photodetector current, within a bandwidth of 200 kHz, and the Brownian motion SNR spectrums for a noise bandwidth of 200 kHz is presented. The Brownian motion was calculated for a cantilever with a resonant frequency, $f_0 = 50$ kHz, quality factor, $Q = 50$, and spring constant $k = 0.63 \text{ N/m}$. 

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3.6. Noise analysis of our optical readout

order response of the cantilever [107]. During AFM imaging in contact mode, if the free end of the cantilever is supported on a hard surface, the Brownian motion is suppressed at the supported end of the cantilever [108]. However, Brownian motion frequency response can provide a convenient way to measure both the resonant frequency and quality factor, from which the spring constant of the structure can be found, and therefore we are interested in estimating the relative contribution of Brownian noise for the free-standing structure. The frequency spectrum of cantilever deflection caused by thermomechanical (Brownian) motion near the 1\textsuperscript{st} resonant frequency, \( n_{2B}(f) \), representing the Brownian motion Z-direction cantilever deflection noise spectrum density, can be calculated using the following equation [109, 110]:

\[
 n_{2B}(f) = \sqrt{\frac{2k_BT}{\pi f_0 kQ \left[ 1 - \left(\frac{f}{f_0}\right)^2\right]^2 + \frac{[f/(f_0Q)]^2}{\sqrt{Hz}}} \left[ \frac{m}{\sqrt{Hz}} \right]}
\]  

(3.28)

where \( k_B \) is Boltzmann constant, \( T = 300 \) K is the room temperature, \( f_0 \) is the resonant frequency, \( k \) is the spring constant and \( Q \) is the quality factor of the cantilever. The calculated spectrum of the Brownian motion signal \( n_{2B}(f) \) with assumed resonant frequency of the cantilever, \( f_0 = 50 \) kHz, spring constant, \( k = 0.63 \) N/m, and quality factor, \( Q = 50 \), is shown in Figure 3.14. A notable characteristic of the Equation 3.28 is that the Brownian motion deflection noise density floor level at \( f \ll f_0 \) is Q-times lower than the peak \( (f = f_0) \).

![Figure 3.14: Defection noise density spectrum due to the cantilever Brownian motion in Z-direction calculated for a cantilever with spring constant, \( k = 0.63 \) N/m, resonant frequency \( f_0 = 50 \) kHz, and a quality factor \( Q = 50 \).](image)

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The mean value of Brownian motion signal calculated for a frequency range between 0 and \( f_x \), gives the mean cantilever deflection in the Z-direction, \( \sqrt{z_B^2} \), and can be calculated as:
\[
\sqrt{z_B^2} = \int_0^{f_x} n_z B^2(f) df \tag{3.29}
\]

The SNR for Brownian motion is often considered for two conditions: (1) while the bandwidth, \( \Delta f \), is significantly lower than resonant frequency, \( f_0 \); and (2) while bandwidth is higher than resonant frequency. The use of bandwidth significantly lower than resonant frequency of the cantilever allows to minimise the influence of the Brownian motion during AFM measurement while it affect measurement results, i.e. in systems with OBD readout or during contact mode AFM imaging of a soft samples, while the Brownian motion is not fully suppressed.

For \( \Delta f \ll f_0 \) the mean cantilever deflection along the Z-axis, \( \sqrt{z_B(1)^2} \), depends on the temperature, \( T \), spring constant, \( k \), resonant frequency, \( f_0 \), bandwidth, \( \Delta f \), and quality factor of the cantilever, \( Q \), and can be calculated from the simplified equation [100]:
\[
\sqrt{z_B(1)^2} = \sqrt{\frac{2k_B T \Delta f}{\pi Q k f_0}} \tag{3.30}
\]
giving the SNR of Brownian motion
\[
SNR_{B(1)} = \frac{1}{\sqrt{z_B(1)^2}} = \frac{\pi Q k f_0}{2k_B T \Delta f} \tag{3.31}
\]

For \( \Delta f \gg f_0 \) the mean cantilever deflection along the Z-axis, \( \sqrt{z_B(2)^2} \), depends only on the temperature, \( T \), and spring constant of the cantilever, \( k \), and can be also calculated from the equipartition theorem [108]:
\[
\sqrt{z_B(2)^2} = \sqrt{\frac{k_B T}{k}} \tag{3.32}
\]
giving the SNR of Brownian motion
\[
SNR_{B(2)} = \frac{1}{\sqrt{z_B(2)^2}} = \frac{k}{k_B T} \tag{3.33}
\]
where \( k_B = 1.38 \cdot 10^{-23} \) J/K is Boltzmann constant, \( T = 300 \) K is a room temperature, and \( k = 0.63 \) N/m is the spring constant of the cantilever. That gives a deflection of the cantilever in the Z-axis of \( \sqrt{z^2} = 81 \) pm. This value is the average deflection noise over all frequencies and matches the value calculated using Equation 3.29 integrated over a wide frequency range. The SNR values calculated for bandwidth of 200 kHz are presented in Figure 3.13. The Brownian SNR (being the lowest) clearly dominates the other sources of
3.6. Noise analysis of our optical readout

noise, and hence measurement of the resonant frequency and quality factor of the cantilever from the Brownian noise should be feasible.

Brownian motion noise can limit OBD readout, even if the free end of the cantilever is supported on a hard surface, due to the indirect measurement of cantilever deflection (e.g. OBD measures cantilever deflection angle) [108]. However, it has no significant influence on the interferometric readout described here, where the cantilever tip displacement is measured directly [108]. Consequently, our readout should be shot noise limited if it operates in the photodetector current range where the electronic noise of the transimpedance amplifier is lower than the photodetector shot noise, as shown in Figure 3.13. The Figure 3.13 shows that SNR of our interferometric readout can be improved by increasing the optical power available for the measurement by improvement of light coupling efficiency between the chip and optical fibres, and increasing the power of laser source. Additionally the noise performance can be improved by improving the readout sensitivity $S$ (see Equation 3.18), which can be achieved with redesigned interrogating grating with improved light coupling angle $\theta$ (see Equations 3.3, 3.4 and 3.10).
Chapter 4

Materials and methods

This chapter investigates materials suitable for device fabrication using surface micromachining techniques, and describes processing methods utilised for deposition and patterning of those materials. For successful device fabrication and to achieve desired performance, the investigation of material properties and development of fabrication processes is necessarily undertaken prior to AFM sensor fabrication. In this chapter, we investigate three materials: amorphous silicon, Brewer Science PI-2611 polyimide, and silicon nitride. The amorphous silicon and HD Microsystems PI-2611 polyimide are examined as sacrificial layers, while the silicon nitride is examined to be used as a structural layer (see Figure 4.1).

![Simplified schematic of surface micromachined MEMS device](image)

**Figure 4.1:** Simplified schematic of surface micromachined MEMS device. The device structural layer is deposited on the substrate covered with a sacrificial layer.

A crucial step in the fabrication of surface-MEMS is the release of a free-standing structure that relies on the selective removal of the sacrificial layer underlying the structural layer [111]. Furthermore, potential structural materials require the availability of process compatible sacrificial materials, as well as release processes with high etch-selectivity between the structural and sacrificial layers. In addition to process compatibility, the sacrificial layer must not negatively impact on the stress of the deposited structural layer in order to avoid unwanted deformation of the free-standing structure [112, 113]. To achieve this, the sacrificial layer must exhibit thickness uniformity, be smooth, chemically stable, non-outgassing [114], have a controllable etch profile, and must match both the intrinsic stress and thermal expansion coefficient of any underlying and/or overlying materials. The etch profile is an important factor, since it has, in conjunction with the structural layer
conformality, a direct impact on the structural integrity of the anchors used to support the cantilevers on the substrate.

For the proposed design of the sensing cantilever, we considered two alternative sacrificial layer materials: amorphous silicon and HD Microsystems PI2611 polyimide. Amorphous silicon sacrificial layers allow the use of organic materials in the fabricated devices, which remain unaffected during the release process thereby allowing, for example, the fabrication of AFM cantilevers with organic tips [115]. In addition, amorphous silicon can be deposited at high deposition rate (lowering the processing time), and has well-controlled thickness with high thickness uniformity. The most significant risk in using amorphous silicon as a sacrificial layer is the limited etch selectivity with both silicon oxide and silicon nitride thin films, which may result in unwanted film damage during the MEMS release process (removal of the sacrificial layer). For a silicon sacrificial layer, we extensively investigated silicon deposition processes to achieve the desired parameters of our thin films. Next, we developed dry etching recipes with controllable etch profiles and, finally, we investigated available release methods for a fabricated device.

An alternative material considered as a sacrificial layer was HD Microsystems PI2611 polyimide. We describe the deposition process used to fabricate uniformly thick polyimide films. Subsequently, we investigated in more details dry etching processes to control the etch profile as there was no available solution in the literature that met our requirements. The additional investigation of a release process was not necessary, as polyimide sacrificial layers allow for easy device release using well-known high-pressure oxygen plasmas.

The fabrication of application specific MEMS utilising surface micromachining relies on the ability to select and use a variety of thin-film materials as the structural layer to achieve the desired electrical, mechanical and/or optical properties. For a micromachined AFM cantilever, the structural layer (in conjunction with the cantilever shape) must be compatible with the range of spring constants \( k \) typically used in AFM measurements, exhibit good thickness conformality to provide strength to the anchors, and allow the tensile stress to be controllable in the range 30 MPa to 120 MPa. AFM cantilevers are typically fabricated from single crystal silicon, however, the amorphous silicon thin films grown in our facility exhibit significantly lower Young’s modulus and poor conformality, excluding that material from consideration for the structural layer. On the other hand, silicon nitride grown in our facility has high Young’s modulus, controllable tensile stress within the optimum range, and excellent conformality, making it a suitable choice for the structural layer. Accordingly, the last part of this chapter characterises the deposition and etching of silicon nitride thin films for a structural layer. A silicon nitride deposition process was developed using ICPCVD, thus realising stable thin films. The process allows fine-tuning of the intrinsic stress of deposited thin films to achieve the desired material parameters. We detail the deposition process used to prepare the SiNx structural layer, followed by the details of the dry etching process used to pattern the silicon nitride structural layer and define the geometry of the suspended structures.
4.1 ICPCVD amorphous silicon as a sacrificial layer

Amorphous [116], polycrystalline [117] and single crystal silicon [118], and more recently porous-silicon [119], have all been used as thin film structural layers to create suspended structures. Amorphous silicon [120] and porous silicon [121, 122] can also be used as sacrificial layers, for example, in surface-micromachining of complementary metal-oxide-semiconductor (CMOS) compatible tunable radio frequency (RF) capacitors, where the sacrificial layer is a sputtered amorphous silicon film [123]. An important requirement of sacrificial layers in certain applications is the ability to achieve large suspension gaps. In resonant operation, squeezed film damping limits the quality factor, thus requiring either high vacuum or suspension gaps greater than 10 μm [93]. For bio-sensing applications, where sensing requires infiltration of cells of up to 10 μm in size, large gaps are also required [124]. For our AFM, a large gap between the sensing surface and the cantilever allows for higher deflection of the cantilever, without risking stiction of the cantilever to the silicon photonics chip and, furthermore, minimises squeezed film damping.

We present a process for high deposition rate amorphous silicon for use as a thick sacrificial layer as depicted in Figure 4.2, using an ICPCVD process with a heated substrate. A significant advantage of this process is the high deposition rate (>120 nm/min), which is higher than previously reported ICPCVD processes (up to 90 nm/min) [125–129]. It is possible to achieve significantly higher deposition rates using microwave chemical-vapour deposition processes (MWCVD), up to 380 nm/min [130, 131], or hot wire chemical-vapour deposition (HWCVD), up to 780 nm/min [132]. However, these alternative processes require deposition temperatures of at least 250 °C. The high deposition rate using ICPCVD enables deposition of nominally up to 10 μm thick sacrificial layers within 2 hours, which is well within the typical processing times for surface-MEMS structures containing multiple layers. The proposed silicon sacrificial layer is ideal for fabrication of MEMS devices on small samples, where spin-coating techniques for sacrificial layers tend to produce a significant edge bead which cannot be easily removed. In addition, MEMS devices fabricated with a thick ICPCVD silicon sacrificial layer can be released using simple wet chemical etching in tetramethyl ammonium hydroxide (TMAH) or potassium hydroxide (KOH) solutions, as well as using dry etching via XeF₂ vapour [133–135] or SF₆ plasma.

![Figure 4.2: Simplified schematic of surface micromachined MEMS device with the fast deposited ICPCVD silicon as an option for an inorganic sacrificial layer.](image-url)

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Figure 4.3: (a) Schematic view of Oxford Instruments Plasmalab System 80+ deposition chamber, (b) The adopted general arrangement of the test sample with 90 µm thick silicon shadow mask and marked points for measurements of thickness uniformity of film deposited on the 25×25 mm² underlying silicon sample area.

[136]. The variety of release methods, which can be used with the proposed sacrificial layer material, allows high selectivity to most modern structural layer materials including silicon oxide, silicon nitride and most metals.

The next section will describe our preparation process for amorphous silicon, giving the deposition recipes and the experimental process adopted for the characterisation of the resulting thin films.

4.1.1 Experimental

The deposition of amorphous silicon thin films was performed using an Oxford Instruments Plasmalab System 80+, which uses two 13.56 MHz RF generators. The deposition process recipe is shown in Table 4.1 and contains four main steps. The process commences with temperature stabilisation of the sample (30 min) followed by a gas flow step (30 sec) to stabilise the chamber pressure and gas composition. Two gasses are metered into the chamber; SiH₄ is delivered through a bottom gas ring, while argon is delivered through a gas shower above the inductively-coupled plasma (ICP) antenna, as depicted in Figure 4.3(a). Argon is used to assist in striking the plasma and is switched off during deposition [137]. A 3-phase plasma strike procedure is required to start and stabilise the low-pressure pure silane plasma.

During deposition, the ion energy (DC bias) depends on the RF generator power connected to the heated table, whereas the ion current (plasma density) is controlled by the
4.1. ICPCVD amorphous silicon as a sacrificial layer

RF generator power connected to the inductively coupled antenna [138]. During the main stage of the silicon deposition, only one RF generator was used, with the RF generator connected to the heated table being switched off to minimise the ion energy, since low ion energy helps in avoiding formation of thin films with significant in-build compressive stress. The high deposition rate can be obtained by adjusting a few variables: gas composition and flow, ICP power, and power to the RF generator connected to the heated table (DC bias). To achieve high deposition rate, we maximised the ICP [139] and minimised the RF power connected to the heated table (DC bias) [140]. For this experiment, we used the maximum value of ICP power and the maximum flow of SiH₄ available in the tool in order to maximise the deposition rate [141].

All depositions were conducted on 300 µm thick, 25×25 mm² silicon <100> substrates coated with a 100 nm thick SiOₓ film, which was used to separate the deposited silicon from the substrate. Before deposition, a 90 µm thick strip of silicon, 3×25 mm² in size, was placed on the substrate as a shadow mask as shown in Figure 4.3(b). The resulting step in the film deposited on the 300 µm thick substrate was used to measure the thickness of the layer, and the 90 µm thick silicon strip with the deposited film was used for stress measurement of the deposited silicon film via stress-induced substrate curvature [142]. All depositions were of 30 min duration. In order to investigate the effect of substrate temperature on the film properties, the heated table temperature was controlled to a fixed value between 25 °C and 300 °C during deposition.

The characterised properties of the deposited silicon thin films are presented in the next section.

4.1.2 Material properties

The deposited silicon thin films were characterised to assess material parameters relevant to the film being used as a sacrificial layer. The properties to be evaluated included deposition rate, film thickness and uniformity, stress, Young’s modulus, hardness, surface roughness and chemical resistance to positive photoresist developer. In all cases, no visible defects were observed in the deposited thin films using optical microscopy. As the proposed recipe is intended to be used as a thin film for sacrificial layers in MEMS devices, the void density of the deposited films was not extensively investigated.

The next subsection of this chapter will present measurements of the deposition rate and thickness uniformity of the amorphous silicon thin films, which are basic parameters of any thin film deposition process.

Deposition rate and thickness uniformity

Film thickness (ranging from 4 µm to 6 µm) was measured using a Veeco Dektak 150 stylus profilometer. Measurements were made in three regions, distributed over the area of the 25×25 mm² sample as indicated in Figure 4.3(b), and used to assess both deposition rate and thickness uniformity. These parameters are shown in Figures 4.4(a, b) as a function of deposition temperature, where the average deposition rate is seen to monotonically
Table 4.1: Process steps in the silicon deposition recipe [APC valve = Automatic Pressure Control valve, which can be set in the range of 0 – 100%; RF power = power of the RF generator driving the heated table; ICP power = power of the RF generator driving the inductively coupled antenna]. Temperature was kept constant during each process, and it was set in a range from 25 to 300 °C.

<table>
<thead>
<tr>
<th>Step name</th>
<th>SiH$_4$ flow</th>
<th>Ar flow</th>
<th>Chamber pressure</th>
<th>APC valve</th>
<th>RF power</th>
<th>ICP power</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>1: Temperature stabilization</td>
<td>0 sccm</td>
<td>0 sccm</td>
<td>0 mTorr</td>
<td>OPEN</td>
<td>0 W</td>
<td>0 W</td>
<td>30 min</td>
</tr>
<tr>
<td>2: Gas flow</td>
<td>30 sccm</td>
<td>30 sccm</td>
<td>12 mTorr</td>
<td>48 %</td>
<td>0 W</td>
<td>0 W</td>
<td>30 sec</td>
</tr>
<tr>
<td>3: Plasma strike, phase 1</td>
<td>30 sccm</td>
<td>30 sccm</td>
<td>12 mTorr</td>
<td>48 %</td>
<td>10 W</td>
<td>50 W</td>
<td>5 sec</td>
</tr>
<tr>
<td>3: Plasma strike, phase 2</td>
<td>30 sccm</td>
<td>0 sccm</td>
<td>5 mTorr</td>
<td>OPEN</td>
<td>10 W</td>
<td>150 W</td>
<td>2 sec</td>
</tr>
<tr>
<td>3: Plasma strike, phase 3</td>
<td>30 sccm</td>
<td>0 sccm</td>
<td>5 mTorr</td>
<td>OPEN</td>
<td>0 W</td>
<td>200 W</td>
<td>2 sec</td>
</tr>
<tr>
<td>4: Silicon deposition</td>
<td>30 sccm</td>
<td>0 sccm</td>
<td>5 mTorr</td>
<td>OPEN</td>
<td>0 W</td>
<td>300 W</td>
<td>30 min</td>
</tr>
</tbody>
</table>
4.1. ICPCVD amorphous silicon as a sacrificial layer

decrease with increasing substrate temperature from a value of 201 nm/min observed for
the film deposited at 25 °C to 129 nm/min for the film deposited at 300 °C, as shown
in Figure 4.4(a). It is a common observation for low-pressure ICPCVD processes in this
temperature range that as the deposition temperature increases the film density also
increases. This can explain the noticeable decrease in deposition rate with increasing
substrate temperature, given that the deposition process is consuming a similar amount
of silicon radicals at all deposition temperatures, but results in progressively denser, and
therefore thinner layers with increasing deposition temperature. Similar behaviour has
been observed previously by Nominanda and Kuo [143] and Jeong et al. [144] for plasma
enhanced chemical-vapour deposition (PECVD) processes.

In the low-temperature regime below 100 °C, thickness variation decreased with increas-
ing temperature and was measured via Dektak to always be better than ±1.1%. An abrupt
change in thickness uniformity is observed at a substrate deposition temperature of 150 °C
and, as such, we use this temperature to define the transition from a low-temperature
process to a high-temperature process. Low-temperature recipes produced silicon thin
films that could be etched in positive photoresist developer and achieved high deposition
rates, allowing a 15 µm thick sacrificial layer to be deposited in 75 minutes at 25 °C.
High-temperature recipes produced silicon films that were stable in positive photoresist
developer but with lower deposition rates, requiring 2 hours to achieve the same 15 µm thick
sacrificial layer at 300 °C. Relative to all other deposited films, the film thickness variation
for 150 °C depositions was found to be the highest (±1.8%) and was consistently lower for
thin films deposited above 150 °C. The reported change in the thickness uniformity is as we
observed in our measured data (one sample, three separate measurements). The variation
of the thickness is within the specification of the tool of <6% over a 2” diameter sample.
This change may not be fully associated with the change in deposition temperature but
may also reflect tool run-to-run variation.

The ICPCVD silicon deposition process can be limited by surface-reactions [111],
and/or by the density of available radicals in the plasma [145]. For a process that is limited
by surface-reactions excess radicals are present in the chamber which can result in the
formation of powder-like polymerised disilane (SiH$_2$) particles [145]. This is a common
observation for higher pressure silicon deposition processes [146]. The deposition recipes
investigated in this work were specifically chosen so as to avoid powder formation within
the chamber, suggesting that the deposition rates were limited by the available radicals in
the plasma. Once the process conditions resulting in no powder formation were achieved,
the density of available radicals in the plasma was kept constant by ensuring the pressure,
gas flow, and RF power were all kept constant.

The following section will introduce stress estimation of the deposited thin films.
Process related stress is one of the most important parameters of thin films used in MEMS
fabrication.
Figure 4.4: Thin film (a) deposition rate, (b) thickness uniformity, (c) stress estimation, (d) Young's modulus, (e) hardness, and (f) RMS surface roughness for amorphous silicon thin films as a function of substrate temperature during deposition.
Thin film stress estimation

In many applications, the stress of released thin films needs to be minimised. This necessitates matching the stress of the sacrificial layer and the structural thin film allowing the deformation of any free-standing structures to be minimised during the release process [112, 113]. In this work, we measured stress of the amorphous silicon sacrificial layer as deposited on silicon substrates, and later in this chapter we used the same method to estimate and tune the stress of the silicon nitride structural layers.

The stress of thin film amorphous silicon layers studied in this work was estimated using thin film stress induced substrate bending and Stoney’s formula [142]. The curvatures of the 90 µm thick silicon strips with dimensions of 3×25 mm² were measured before and after depositions of the amorphous silicon thin films using a Zygo NewView 7300 white light optical surface profilometer. Figure 4.4(c) shows the resulting stress estimation as a function of substrate deposition temperature. It should be noted that in all cases a relatively low value of intrinsic tensile stress was found (<70 MPa). Not only is tensile stress preferred over compressive stress whenever it is desirable to create flat suspended beams and plates, but the observed magnitude of the stress is considerably lower than for hydrogenated amorphous silicon films deposited using PECVD or RF glow discharge. Yin et al. [147] reported compressive stress values in the range from 300 MPa to 800 MPa for hydrogenated amorphous silicon prepared via PECVD. Harbison et al. [148] reported compressive stress values in the range from 50 MPa to 800 MPa for hydrogenated amorphous silicon thin films deposited using RF glow discharge.

The subsequent section will present measurement results of Young’s modulus and hardness, parameters used to estimate the quality of the deposited thin film.

Young’s modulus and hardness

The Young’s modulus and indentation hardness of the samples were estimated via the nanoindentation technique [149] using a Hysitron TI 950 TriboIndenter instrument and a Berkovich indentation tip [150]. Each amorphous silicon thin film sample was indented with 5 sets of 30 indents with loading progressively increasing from 0.5 mN to 10 mN. The Young’s modulus and hardness of the material were calculated using the Oliver and Pharr method [151], where the penetration depth exceeded 50 nm [152, 153]. During the calculations we assumed Poisson’s ratio $\nu_{\text{silicon}}=0.278$ [154], $\nu_{\text{indenter}}=0.07$, and Young’s modulus $E_{\text{indenter}}=1140$ GPa [152]. The obtained Young’s modulus and hardness values are shown in Figures 4.4(d, e) as a function of substrate deposition temperature.

The generally high Young’s modulus and hardness values observed, especially for deposition at the highest temperature (300 °C), are comparable with values reported in the literature for high-quality thin film silicon layers. Jiang et al. [155] reported values of Young’s modulus up to 100 GPa and hardness up to 10 GPa for sputtered amorphous hydrogenated silicon. In comparison, Bhushan and Li [156] measured a Young’s modulus of 179 GPa and a hardness for undoped crystalline silicon <100> of 13 GPa.
Figure 4.5: AFM images of surfaces of amorphous silicon thin films deposited at different substrate temperatures. The indicated scale is common to all images.

The next subsection will present another important parameter for our sacrificial layer – surface roughness, which needs to be sufficiently low since the roughness of subsequent layers deposited on top of sacrificial layer will be the same or higher, duplicating the surface roughness of the sacrificial layer.

Surface roughness

The RMS surface roughness of the samples was calculated from $5 \times 5 \, \mu m^2$ surface profile images of the silicon films acquired using a WITec alpha 300RA+ AFM, and calculated using WITec Project FOUR software, version 4.0. Figure 4.5 shows the AFM images of the amorphous silicon thin film surfaces obtained for films prepared at the investigated deposition temperatures, and the RMS surface roughness values are shown in Figure 4.4(f) as a function of deposition temperature. The surface roughness was found to decrease with increasing substrate deposition temperature, with the highest surface roughness (6.9 nm) observed for a deposition temperature of 75 °C, and the lowest value (3.1 nm) occurred for a substrate temperature of 300 °C. The results indicate that more robust films (lower RMS surface roughness, higher Young’s modulus and higher indentation hardness) are formed at higher substrate deposition temperatures. For use as a sacrificial layer, the most important parameter can often be the RMS surface roughness, since any subsequently deposited layers will conform to the same degree of roughness [157]. Thus, surface roughness needs to be minimised, especially for MEMS optical surfaces, in order to achieve adequate performance. For optical MEMS, the maximum acceptable level of surface roughness is often given as $\lambda/20$ [158–162]. For example, for devices operating at a wavelength of 1550 nm, the surface roughness value needs to be <78 nm. All surface roughness values observed in this study are significantly below this limit.
The following section reports on the resistance of the silicon thin films to the positive photoresist developer. This is necessary requirement for most sacrificial layers, as it allows for subsequent processing on top of the sacrificial layer.

**Chemical resistance to positive photoresist developer**

The amorphous silicon thin films deposited at low temperatures were found to be readily etched in a positive photoresist developer. Chemical resistance to positive photoresist developer is important if any post-deposition photolithography process is required directly on the sacrificial layer. Chemical resistance to developer was determined by immersing samples in Microchem AZ326, an aqueous solution of 2.38% TMAH. Initially, samples were immersed in the developer for 5 minutes, and the thickness change of the amorphous silicon layer was measured using a Veeco Dektak 150 stylus profilometer. Next, the immersion was repeated twice, each time for an additional 10 minutes. The amorphous silicon samples deposited at temperatures up to 100 °C were found to be completely etched away during the first 5 minute immersion. The sample with a deposition temperature of 150 °C was partially etched over the same 5-minute duration, and had a resulting extremely rough surface. In contrast, amorphous silicon samples deposited at temperatures of 200 °C and above were found to be chemically stable and not etched in positive photoresist developer, even after a total immersion time of 30 minutes. Not surprisingly, samples with higher hardness were found to be more resistant to etching in positive photoresist developer.

**4.1.3 Deposition process summary**

It was found by Haller et al. [163] that hydrogenated amorphous silicon etch rate can be directly and positively correlated with hydrogen content in the deposited material. It is expected that total hydrogen content in films deposited at lower temperatures (up to 100 °C) is significantly higher in comparison to films deposited at 150 °C and above. Moravej et al. [164] reported a change from 6.0 at% to 3.0 at% total hydrogen content in amorphous silicon thin films for a deposition temperature change from 100 °C to 150 °C; and the hydrogen content in films prepared in the temperature range from 25 °C to 100 °C is expected to be even larger. For temperatures above 150 °C, Moravej et al. [164] found that the density of the films increases significantly as the deposition temperature is increased, and this may be due to a decrease in hydrogen content in the undesirable form of SiH₂ with increasing deposition temperature. Based on this, we correlate the expected excessive hydrogen concentration of films deposited in the low-temperature regime with their ability to be etched in a positive photoresist developer.

It was found by Jiang et al. [155] that hydrogen content in silicon thin films can be directly correlated with Young’s modulus and indentation hardness. That can be further explained by the work of Moravej et al. [164], who reported that films deposited at lower temperatures contain a significantly higher void density. The presence of voids can imply lower values of both Young’s modulus and indentation hardness observed for films deposited in the low deposition temperature regime as compared to the high deposition
Table 4.2: Amorphous silicon etching process divided into three steps: Step A consists two SF\textsubscript{6}/O\textsubscript{2} etching recipes for fast silicon patterning, each of them with different etch rate and undercut; Step B consists of photoreist removal recipe, and Step C consists of CF\textsubscript{4} etching recipe for improvement of the sidewall profile.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Step A</th>
<th>Step B</th>
<th>Step C</th>
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<tbody>
<tr>
<td></td>
<td>Recipe 1</td>
<td>Recipe 2</td>
<td></td>
</tr>
<tr>
<td>CF\textsubscript{4} flow [sccm]</td>
<td>–</td>
<td>–</td>
<td>34</td>
</tr>
<tr>
<td>SF\textsubscript{6} flow [sccm]</td>
<td>80</td>
<td>80</td>
<td>–</td>
</tr>
<tr>
<td>O\textsubscript{2} flow [sccm]</td>
<td>5</td>
<td>5</td>
<td>75</td>
</tr>
<tr>
<td>RF power [W]</td>
<td>15</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>ICP power [W]</td>
<td>600</td>
<td>150</td>
<td>100</td>
</tr>
<tr>
<td>Pressure [mTorr]</td>
<td>60</td>
<td>5</td>
<td>1000</td>
</tr>
<tr>
<td>Time [min]</td>
<td>6</td>
<td>9</td>
<td>5</td>
</tr>
<tr>
<td>Etch rate [um/min]</td>
<td>1.42</td>
<td>0.94</td>
<td>–</td>
</tr>
<tr>
<td>Undercut [% of depth]</td>
<td>69</td>
<td>26</td>
<td>–</td>
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</tbody>
</table>

This subsection concludes the reports on material characterisation of amorphous silicon, and the following subsection reports the developed silicon etching process to be used for removal of the silicon sacrificial layer and release of free-standing structures.

4.1.4 Etching process for thin film patterning

During patterning of the sacrificial layer, we need to produce positive sidewall slopes in order not to jeopardise the conformality of subsequently deposited overlayers. This is a very important requirement since any overhang of the sacrificial layer sidewall can cause discontinuity of the subsequently deposited structural thin films. Any layer discontinuities may lead to structural collapse during the release process. In addition, the sacrificial layer patterning process needs the use of a mask that can be easily removed after patterning, without damaging the surface of the sacrificial layer. The amorphous silicon layer patterning was conducted using inductively-coupled plasma reactive-ion etching (ICPRIE) via a three-stage process at room temperature as presented in Table 4.2. The silicon sacrificial layer was protected by a spin-coated photoresist mask used to define the pattern. In the first
Table 4.3: Summary of the measured material properties for ICPCVD silicon thin films prepared using low- and high-temperature recipes.

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</thead>
<tbody>
<tr>
<td>Thickness uniformity [±%]</td>
<td>177 – 201</td>
<td>128 – 144</td>
<td>0.4 – 1.1</td>
<td>0.5 – 1.4</td>
<td>±6% over 50 nm area [165]</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Young's modulus [GPa]</td>
<td>79 – 94</td>
<td>111 – 123</td>
<td>low pressure CVD [155]</td>
<td>up to 100 GPa [155]</td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>Indentation hardness [GPa]</td>
<td>6.9 – 8.3</td>
<td>9.9 – 10.5</td>
<td>low pressure CVD [155]</td>
<td>up to 10 GPa [155]</td>
<td></td>
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<td></td>
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<tr>
<td>RMS surface roughness [nm]</td>
<td>5.5 – 6.9</td>
<td>3.1 – 3.8</td>
<td>–</td>
<td>–</td>
<td></td>
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<tr>
<td>Chemical resistance to positive photoresist developer</td>
<td>No</td>
<td>Yes</td>
<td>–</td>
<td>–</td>
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</table>
step (Step A) the silicon sacrificial layer was etched using an SF$_6$/O$_2$ ICPRIE recipe. Here we considered two versions of SF$_6$/O$_2$ etching, each with different outcomes. Etching with recipe #1 is mainly isotropic (see Figure 4.6), with minimum ion directional bombardment, and provides exceptionally high selectivity to the photoresist mask. We did not observe any change in mask thickness after the relatively long etching process taking 9 minutes. However, recipe #1 produces significant undercutting of the overlying mask. The outcome of the second recipe is characterised by significantly lower undercut, but the selectivity to the photoresist mask is compromised (see Figure 4.7). We removed about 0.25 µm of the mask thickness for a 1 µm deep etch of the sacrificial layer. Due to this low silicon to photoresist etch selectivity, recipe #2 is limited when used for our amorphous silicon sacrificial layers of up to about 12 µm in thickness due to the maximum photoresist mask of 3.5 µm thick used in this work. Since we were aiming to use a sacrificial layer between 8 µm and 10 µm in thickness, we adopted recipe #2 for further device fabrication to ensure process compatibility. Subsequent to etching with SF$_6$/O$_2$ chemistry, the photoresist mask was removed with dry etching in O$_2$ plasma (step B). Alternatively, photoresist can also be dissolved in an acetone bath. After photoresist mask removal, a positive sidewall slope is achieved during CF$_4$ plasma exposure as shown in Figure 4.8. This step partially removes the exposed silicon sacrificial layer material and sidewall overhang created during previous etching steps. The process can be timed to fine-tune its thickness. For this work, by trial-and-error, we established that a 5 minutes long etch was sufficient to achieve the desired sidewall profile. The thickness of the sacrificial layer was reduced by about 1.7 µm, which can be compensated for by deposition of an initially thicker silicon layer.

The next section introduces the removal processes used for our amorphous silicon sacrificial layers for the release of the overlying structural layers.

4.1.5 Etching process for device release

All of the amorphous silicon thin films investigated in this work can be etched using: (i) dry chemical etching in XeF$_2$ vapour, with etch rates of up to 2 µm/min in the vertical direction and up to 4 µm/min in the lateral direction [133–135]; (ii) SF$_6$ dry plasma with etch rates up to 15 µm/min [136]; (iii) heavily concentrated and heated TMAH and KOH solutions, with etch rates up to 1.4 µm/min (e.g., Tabata et al. [166] used a solution of 22 wt% TMAH at 90 °C, and Shikida et al. [167] used a solution of 34.0 wt% KOH at 71 °C).

Since forming a suitable AFM tip using surface micromachining was beyond the scope of this work, we considered tip attachment after deposition of the sacrificial layer and device patterning. Using a standard AFM silicon tip would compromise the release process if silicon were used as a sacrificial layer, however a platinum tip can be fabricated. To allow the possibility of using a standard AFM tip, an alternative organic sacrificial layer was investigated, and is described in the following section.
Figure 4.6: Silicon sacrificial layer profile after first etching step using recipe #1 in Table 4.2. The undercut caused by this recipe is about 69% of thickness of the etched sacrificial layer. During the etching process, a significant overhang on the silicon sidewall was created.

Figure 4.7: Silicon sacrificial layer profile after first etching step using recipe #2 in Table 4.2. The undercut caused by this recipe is about 26% of the thickness of the etched sacrificial layer. The overhang of the silicon sidewall is still visible, but it was reduced in comparison to recipe 1. The etch rate of the photoresist mask was significantly higher as the thickness of photoresist mask was reduced by half.
4.2 Polyimide as an organic sacrificial layer

Organic sacrificial layers are commonly used to support structural layers during surface micromachining of MEMS as depicted in Figure 4.9. They provide controllable layer thickness using a simple spin-on process, can survive a wide variety of surface micromachining processes, and can easily be removed through a dry release method in order to avoid the stiction issues associated with wet release processes. Organic polymer sacrificial layers include standard lithography photoresists [168, 169], polyimides (e.g., HD Microsystems PI-2610, PI-2611 or Brewer Science ProLIFT) [170, 171], epoxy-based photoresists (e.g., SU-8) [172], and water-soluble polymers [173]. Of these, HD Microsystems polyimides are of considerable importance for micromachining applications since, in addition to being able
to be removed with a pure oxygen plasma, they can withstand processing temperatures of up to 400 °C. Another advantage of organic sacrificial layers is that sidewall control during process is readily achievable as described below.

Dry etching of polyimides has been extensively explored with the aim of controlling the sidewall angle of the anchors used to support suspended structural layers. Mimoun et al. [174] developed an etch recipe which combined isotropic and anisotropic plasma etching to obtain a wine glass shaped sidewall profile in polyimide vias using sequential etching in two separate chambers. In comparison, the recipes developed in this paper use only a single etch process in a single chamber to achieve tapered sidewalls.

Deschler and Balk [175] investigated the influence of O₂/CF₄ gas composition on the sidewall profile of etched structures in 2 µm thick polyimide layers using photoresist as a mask. Their approach was similar to that of Till et al. [176], who developed a recipe for reactive-ion etching (RIE) etching of polyimide (Hitachi PIQ) in a pure oxygen plasma to control the sidewall slope. Till et al. [176] achieved control of the sidewall profile through the use of a photoresist erodible mask on a 1.5 µm thick polyimide layer by careful control of the etching conditions, although the presented process is limited by the thickness of the photoresist mask. However, for thick polyimide films (>5 µm) a thicker photoresist masking layer is required, and the process that provides high selectivity to the photoresist mask is characterised by a low polyimide etch rate (maximum about 180 nm/min). Pham et al. [177] presented an etch recipe based on pure oxygen plasma for relatively thin 1–2.5 µm HD Microsystems PI-2610 polyimide sacrificial layers. However, their process leads to the formation of unwanted residues, which is a major issue. They also investigated the use of O₂/He gas mixtures to reduce the residue level, but those mixtures affected the anisotropy of the etching profiles. Chen et al. [178] developed several polyimide etch recipes to investigate the influence of O₂/SF₆ gas ratio on the etch rate and sidewall profile. They investigated relatively high-pressure RIE recipes (600 mTorr) in which the ratio of O₂ to SF₆ varied from 50% to 100%, and reported on the resultant unusual sidewall profiles. Lee et al. [179] investigated similar polyimide dry etch recipes for the fabrication of thin film transistors (TFT), based on an O₂/SF₆ chemistry, but with the addition of Ar gas. Etching was performed using capacitively-coupled plasma reactive-ion etching (CCPRIE) at a pressure of 100 mTorr and RF power of 100 W, whereas Chen et al. [178] used 300 W. In addition, Lee et al. [179] investigated the details of the plasma chemistry during the etching process using optical emission spectroscopy (OES).

The avoidance of any post-processing residues is an important issue during polyimide etching [180] which, in general, can be avoided by using gas mixtures other than pure oxygen. Various gas mixtures have been proposed to avoid the formation of contaminant residues. Turban and Rapeaux [181] used O₂/CF₄ and O₂/SF₆ plasmas to etch polyimide for a desmearing process during printed circuit board production, and analysed the influence of different gas mixtures on the etching process. Bliznetsov et al. [182] also considered different gas mixtures at various plasma power levels to achieve a vertical polyimide sidewall profile, based on a high-throughput polyimide etching process using O₂/CF₄ chemistry in
Chapter 4. Materials and methods

a dual frequency RIE tool. In addition, Ma et al. [183] investigated the influence of the bias plate power on the residues remaining after etching in pure O\textsubscript{2} plasma.

Dry etching of thick polyimide films with a standard photoresist mask is not feasible due to the lack of durability of photoresist in a plasma environment, and thus other masking materials with higher etch selectivity to polyimide are required. Bagolini et al. [114] investigated O\textsubscript{2}/CF\textsubscript{4} dry etching of HD Microsystems PI-2610 polyimide (2–4 µm layer thickness) using several different materials as a masking layer, including aluminium, silicon carbide, and silicon oxide. In their study, silicon oxide was selected as the preferred option due to its better adhesion to polyimide compared to aluminium, and because it is easier to remove than silicon carbide. Additionally, they investigated the chemical resistance of polyimide to the various solvents used for cleaning the wafers between processing steps. Arevalo et al. [184] presented a surface micromachining process that used a gold structural layer as a hard mask for dry polyimide etching. For application in which gold is suitable to serve as the structural layer, their approach simplifies the fabrication process by avoiding the need for a separate hard mask.

Zulfiqar et al. [185] presented etching processes for 10 µm to 30 µm thick polyimide layers based on two different hard mask materials (SiNx and Al). Their investigation included the influence of plasma chemistry, RF and ICP power, and chamber pressure on the etch rate of polyimide. Their analysis covered plasmas with pure O\textsubscript{2}, O\textsubscript{2}/Ar, O\textsubscript{2}/CF\textsubscript{4} and O\textsubscript{2}/SF\textsubscript{6} mixtures. Vertyanov et al. [186] presented etching processes for 25 µm and 76 µm thick polyimide films, where they examined the influence of the gas composition (O\textsubscript{2}, Ar and SF\textsubscript{6}) and RF power on the process etch rate. However, it needs to be noted that the influence of chamber pressure on the etching of polyimide layers thicker than 5 µm and at elevated process temperature (in our case, 100 °C) has not been previously documented in the open literature. Such thick sacrificial layers are often necessary in order to reduce squeezed film damping of the fabricated free-standing structures, which may be suspended several micrometres above the substrate surface [187]. For example, high structures with tapered sidewalls are essential for MEMS ionisation gas sensors [188]. The features of the proposed polyimide etch recipe can also be used for fabrication of polyimide-based variable focus microlenses [189] as well as for angled mirrors for planar silicon photonics [190, 191] where tapered sidewalls are essential.

In the next two sections, we will describe a residue-free anisotropic plasma etching process for thick polyimide films, with the ability to precisely control the sidewall profile. The sidewall angle is a key parameter required for fabricating free-standing structures based on thin films deposited with limited conformality using thermal evaporation and/or PECVD techniques. A steep slope of the polyimide sidewall will limit the layer thickness deposited on the sidewalls significantly. Thus, a controlled profile can be used to improve sidewall coverage to some extent.

In what follows we present details of polyimide deposition and patterning processes used during the characterisation of sidewall etch profile of polyimide. We report on the influence of chamber pressure on the polyimide etching process at elevated processing
temperatures, and we investigate the mechanisms which determine the sidewall angle. In addition, we introduce a hard mask lift-off process based on an interfacial polymer layer for removal of the overlying hard mask.

4.2.1 Experimental

The polyimide etching experiments were performed using an ICPRIE Plasmalab System 100 tool from Oxford Instruments. In this system, a 4-inch diameter, 300 µm thick, <100> silicon carrier wafer is mechanically clamped to the heated sample table and back-side helium is introduced to provide good thermal contact between the table and carrier wafer. Before commencing the etching process, the sample table temperature was set at 100 °C and allowed to stabilise. The experiments were performed using polished square <100> silicon substrates (2×2 cm²) of 300 µm in thickness. The experiments were also repeated on 2-inch diameter silicon <100> substrates with the same thickness as the 2×2 cm² samples in order to assess the influence of loading effects on the etching process. A schematic of the adopted process flow is shown in Figure 4.10. The bare silicon substrates were covered with Brewer Science APX-K1 adhesion promoter, which was selected in preference to the VM-651 recommended by HD MicroSystems (manufacturer of the PI-2611 polyimide) due to easier storage and usage specifications. The APX-K1 adhesion promoter is premixed and ready to use, whereas VM-651 needs to be diluted before each use. The adhesion promoter was found to be necessary between each layer in the procedure in order to avoid delamination of the polyimide from the substrate, as well as delamination between subsequently deposited layers.

The substrates were spin-coated with a thick polyimide layer (HD MicroSystems PI-2611) in a two-step spinning cycle. Firstly, a rotation speed of 500 rpm for 5 seconds was used to spread the dispensed material on the surface, followed by 2000 rpm for 40 seconds to achieve the desired thickness, typically in the range of 8 µm – 9 µm thick. After spin-coating with PI-2611, the samples were soft baked in ambient laboratory conditions on a hotplate for 90 seconds at 90 °C, followed by another 90 seconds at 150 °C. Subsequent curing of the polyimide was completed in a nitrogen atmosphere on a hot plate using the heating/cooling profile shown in Figure 4.11, which indicates a curing segment at 400 °C for one hour. The initial soft bake at 90 °C and 150 °C allows stacking of multiple layers of polyimide in order to achieve very thick sacrificial layers [192], whereas the final high-temperature curing process given by the temperature profile in Figure 4.11 produces a stable film with the desired parameters.

A layer of Brewer Science APX-K1 adhesion promoter was applied on top of the PI-2611 polyimide layer to obtain adequate adhesion of the overlying Brewer Science ProLIFT 100 layer, and thus avoid the risk of delamination at the interface, as schematically indicated in Figure 4.10 by the thick red interfaces. The etching mask consisted of the ProLIFT 100 polyimide layer (about 3.5 µm thick) and an overlying silicon oxide hard mask (about 1 µm thick). The ProLIFT 100 was spin-coated on the surface of the PI-2611 polyimide, soft baked at 135 °C for 90 seconds and then cured at 300 °C for 30 minutes. Next, a layer
of APX-K1 adhesion promoter was deposited just before silicon oxide deposition in an ICP-CVD system at a substrate temperature of 275 °C (see Figure 4.10). Brewer Science ProLiFT 100 [171] are polyimides that are soluble in positive resist (alkaline) developers after imidization by baking, but retain high resistance to acid etchants, organic solvents, and high process temperatures. PI-2611 is a non-photodefineable, dry etch polyimide which, in contrast to ProLiFT 100, is not soluble in positive resist developers after full curing. The solubility of ProLiFT 100 in positive resist developers allows lift-off of the silicon oxide hard mask in a wet process after dry etching of PI-2611 polyimide has been completed, without affecting the PI-2611 sacrificial layer.

The silicon oxide hard mask was patterned using a photoresist mask and etched in CF₄ using an Oxford Plasmalab System 100 ICPIRE system. The developed process flow does not require a separate step to remove the photoresist used to pattern the silicon oxide hard mask before commencing the polyimide etching process, since the polyimide etch process will also rapidly remove the photoresist and leave a residue-free top surface. The etch rate of the SiOₓ hard mask was determined to be between 15 nm/min and 20 nm/min, and the
Figure 4.11: Temperature profile for curing polyimide in a nitrogen atmosphere. A ramp rate of 3 °C/min was used for the heating and cooling steps. The hold times consist of 5 minutes at 170 °C for solvent evaporation, and 1 hour at 400 °C for complete polyimide curing.

thickness was set to 1 μm in order to ensure good edge definition in subsequent imaging processes. For routine processing, the thickness of the hard mask can be reduced, for example, to 400 nm of SiO₂ for a 9 μm thick polyimide film. A summary of all processing steps, preceding polyimide etching, is listed in Table 4.4.

The adopted polyimide etching chemistry was based on the work of Mimoun et al. [174], which consisted of 80% O₂, 13% N₂, and 7% CF₄. In this work, the desired etch

### Table 4.4: Detailed fabrication procedure for polyimide samples prior to polyimide etching.

<table>
<thead>
<tr>
<th>Step Description</th>
<th>Conditions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clean silicon substrate wafer</td>
<td></td>
</tr>
<tr>
<td>Spin adhesion promoter Brewer Science APX-K1 (4000 rpm/40 s)</td>
<td></td>
</tr>
<tr>
<td>Bake APX-K1 on the hotplate (175 °C/40 s)</td>
<td></td>
</tr>
<tr>
<td>Spin HD Microsystems PI-2611 (500 rpm/5 s followed by 2000 rpm/40 s)</td>
<td></td>
</tr>
<tr>
<td>Soft-bake PI-2611 on the hotplate (90 °C/90 s, followed by 150 °C/90 s)</td>
<td></td>
</tr>
<tr>
<td>Cure PI-2611 on the hotplate (400 °C/1 hour, heating/cooling ramp rate 3 °C/min: see Figure 4.11)</td>
<td></td>
</tr>
<tr>
<td>Spin adhesion promoter Brewer Science APX-K1 (4000 rpm/40 s)</td>
<td></td>
</tr>
<tr>
<td>Bake APX-K1 on the hotplate (175 °C/40 s)</td>
<td></td>
</tr>
<tr>
<td>Spin Brewer Science ProLIFT 100-24 (500 rpm/5 s, next 4000 rpm/90 s)</td>
<td></td>
</tr>
<tr>
<td>Soft-bake ProLIFT 100-24 on the hotplate (135 °C/90 s)</td>
<td></td>
</tr>
<tr>
<td>Cure ProLIFT 100-24 on the hotplate (300 °C/30 min)</td>
<td></td>
</tr>
<tr>
<td>Spin adhesion promoter Brewer Science APX-K1 (4000 rpm/40 s)</td>
<td></td>
</tr>
<tr>
<td>Bake APX-K1 on the hotplate (175 °C/40 s)</td>
<td></td>
</tr>
<tr>
<td>Deposit SiO₂ hard mask using ICP-CVD deposition tool (275 °C/1 hour)</td>
<td></td>
</tr>
<tr>
<td>gas flow: SiH₄ = 6.5 sccm, He = 123 sccm, Ar = 126 sccm, N₂O = 70 sccm; pressure: 2 Pa; ICP power: 450 W.</td>
<td></td>
</tr>
<tr>
<td>Sample at this stage corresponds to that shown in Figure 4.10(a)</td>
<td></td>
</tr>
<tr>
<td>Pattern SiO₂ hard mask using ICP-RIE etching tool with photoresist mask (20 °C/26 min)</td>
<td></td>
</tr>
<tr>
<td>gas flow: CF₄ = 34 sccm; pressure: 10 Pa; RF power: 100 W; ICP power: 400 W.</td>
<td></td>
</tr>
<tr>
<td>Sample at this stage corresponds to that shown in Figure 4.10(b)</td>
<td></td>
</tr>
</tbody>
</table>
characteristics were achieved by setting the substrate stage temperature to 100 °C and varying the chamber pressure between 5 mTorr and 40 mTorr to tune the sidewall profile. The temperature of 100 °C was chosen experimentally as a minimum value to achieve stable and repeatable etching results. Prior to commencing the etching process, the samples were placed on top of the carrier wafer. The additional thermally conductive material was not introduced between the samples and the carrier wafer since trial runs indicated adequate process stability and performance. The carrier wafer was loaded into the etching chamber and mechanically clamped to the heated table. Helium gas was introduced between the carrier wafer and the heated table to achieve good thermal contact and allow efficient heating of the carrier wafer. The samples remained in that condition for 15 minutes for temperature equilibration before processing commenced. During the etching process the temperature on the surface of the sample may increase due to plasma heating, which can be an advantageous characteristic since the localised increase in temperature may locally speed up the etching reactions. This phenomenon could be explored further to achieve local sidewall tapering just under the hard mask, if desired. The ICP and RF power were chosen experimentally in order to achieve a stable and repeatable etch profile with relatively high etch rates. The chamber pressure during the etching process has a significant influence on the resulting polyimide pedestal slope [193]. Mimoun et al. [174] did not provide any additional details on their recipe or the equipment they used. The details of the plasma etch recipes developed in the present study are summarised in Table 4.5.

Table 4.5: Polyimide plasma etch recipe details.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>O&lt;sub&gt;2&lt;/sub&gt; flow</td>
<td>60.0 sccm</td>
</tr>
<tr>
<td>N&lt;sub&gt;2&lt;/sub&gt; flow</td>
<td>9.8 sccm</td>
</tr>
<tr>
<td>CF&lt;sub&gt;4&lt;/sub&gt; flow</td>
<td>5.2 sccm</td>
</tr>
<tr>
<td>ICP Power</td>
<td>200 W</td>
</tr>
<tr>
<td>RF Power</td>
<td>100 W</td>
</tr>
<tr>
<td>Stage temperature</td>
<td>100 °C</td>
</tr>
<tr>
<td>Pressure</td>
<td>5 – 40 mTorr</td>
</tr>
<tr>
<td>He backing pressure</td>
<td>10 Torr</td>
</tr>
</tbody>
</table>

After the polyimide etching experiments, the samples were placed in an AZ326 photore sist developer bath (an aqueous solution of 2.38% TMAH) in order to dissolve the ProLIFT 100 layer and lift-off the silicon oxide hard mask. This process required up to a few hours to complete when polyimide etching was performed through small openings in the hard mask. However, it is noted that the process consistently resulted in a residue-free and undamaged surface of the PI-2611 polyimide, and guaranteed removal of the hard mask.

The following section will report details of performed experiments and then present sidewall profile time-evolution recorded during the etching process.
4.2.2 Etching process for thin film patterning

To evaluate the etching process, two series of experiments were performed, each having multiple samples that were prepared and etched in a separate fabrication process, and the cross-section of each sidewall profile was obtained using a focused ion beam (FIB) and a scanning electron microscope (SEM). During the first series of experiments, the samples were etched with a common recipe but with the etching time being varied between 1 and 20 minutes. This allowed the progress of the etching of the polyimide sacrificial layer to be evaluated. During the second series of experiments, each sample was etched until all the polyimide was removed under different chamber pressure in the range between 5 and 40 mTorr for individual samples.

The following paragraph will report on the series of experiments which allow the time evolution of the etch profile to be characterised.

Etch profile time evolution

The first series of experiments consisted of ten samples, each etched for a different etching time, with the first sample being etched for 1 minute. The etching time for the remainder of the samples was progressively increased from 4 to 20 minutes in steps of 2 minutes. Figures 4.12(a)-(c) show SEM images of the cross-sectional etch profiles for three selected samples. The profiles of the etched structures shown in Figure 4.12(d) were extracted from the SEM images by an edge detection filter using ImageJ 1.50i software in order to allow for a precise comparison between the samples. The obtained profiles were stacked together on one image to show the time-evolution of the etching profile (see Figure 4.12(d)).

As evident from Figure 4.12(d), two distinct slopes \( \alpha \) and \( \beta \) can be distinguished on the sidewall of the PI-2611 polyimide layer. The ICPRIE etching process consists of two components: physical and chemical etching. The physical etching component is associated with close to vertical ion bombardment of the exposed part of the polyimide and is mainly responsible for obtaining straight vertical sidewalls. The chemical etching component is associated with the reactive species of the plasma chemically etching the polyimide material equally in all directions, and is primarily responsible for the horizontal etching of polyimide since the sidewalls are partially protected from ion bombardment by hard mask shadowing. Both physical and chemical etching components are associated with vertical etch of polyimide. As the polyimide is etched vertically, an increasing amount of sidewall depth is exposed which then allows horizontal chemical etching to occur. Since both the horizontal and vertical etching occur simultaneously, the horizontal etching of the sidewall creates sloped sidewalls as the upper parts of the polyimide sidewall are progressively exposed and etched for longer than the lower sidewall parts (angle \( \alpha \) for PI-2611 polyimide). The same reasoning can be used to explain the angle of the main sidewall slope of the overlaying ProLIFT layer (lower portion of the ProLIFT sidewall). Note that the ion bombardment enhanced vertical component of the etching process dominates the horizontal component more prominently for ProLIFT than for PI-2611, which results in a steeper sidewall profile for the ProLIFT layer. The upper sidewall slope of the PI-2611 polyimide
Figure 4.12: (a)–(c) SEM images of FIB-etched cross-section of the etched sample structures using the same recipe (see Table 4.5, with pressure = 10 mTorr) for different etch times. The indicated scale is common to (a)–(c) images. (d) The stacked outline plots of cross-section of ten separate samples with etch time starting with 1 minute and then from 4 minutes up to 20 minutes in 2-minute increments. The outlines were extracted from the images such as (a)–(c) using edge detection of the profiles and projected into the x-y plane. The outlines of measured profiles were aligned to the bottom surface of the SiO\(_x\) hard mask. The angle \(\alpha\) stays constant as all profiles were acquired at the same chamber pressure, while the angle \(\beta\) stays constant as etch rate difference between polyimide and ProLIFT is constant. The underetch under the SiO\(_x\) hard mask commences due to heating of hard mask.

layer (angle \(\beta\) in Figure 4.12(d)), is a consequence of the difference in the etch rates between the ProLIFT and the underlying PI-2611 polyimide. Since the horizontal etch rate of the ProLIFT is significantly higher than the PI-2611 polyimide layer, progressively more PI-2611 top surface is exposed for chemical etching. This allows chemical etching of the exposed upper surface of the PI-2611 to progress at the same time as etching in the horizontal direction, which results in the formation of a slope of angle \(\beta\).

The slight undercut which is present just below the hard mask in the ProLIFT 100 is likely due to localised heating of the SiO\(_x\) hard mask caused by ion bombardment during the long etching process. Although the ion energy does not cause significant etching of the hard mask, it can increase the temperature of the masking layer. Since this heat is not dissipated efficiently to the substrate due to low thermal conductivity of the thick underlying polymer layers, there is likely to be a significant increase in the local SiO\(_x\) temperature [194, 195]. Thus, in comparison to the bulk of the ProLIFT, the increased temperature just under the SiO\(_x\) hard mask will result in faster chemical etching of the ProLIFT surface [196, 197], resulting in an increased lateral etch rate directly under the mask. The faster etch rate directly under the mask on the cross-sectional profiles presented in Figure 4.12(d) is observed to commence after a few minutes of etching once the temperature of the hard mask has increased sufficiently.
4.2. Polyimide as an organic sacrificial layer

The next section will report on the influence of the chamber pressure on the polyimide sidewall profile.

**Etch profile slope control**

The second series of experiments investigated the influence of the chamber gas pressure on the sidewall profile of the etched polyimide layers, with the chamber pressure being varied between 5 and 40 mTorr. The SEM images showing the cross-sections of the etched structures are presented in Figure 4.13, which indicate that pressure has a significant influence on the sidewall profile. The \( \alpha \) angle for the lower part of the PI-2611 polyimide slope varies significantly as a function of chamber pressure, and this relationship is shown in Figure 4.14(a). Our observations indicate that an overall increase in the etch rate of PI-2611 is associated with increasing process pressure (see Figure 4.14(b)), and that the \( \alpha \) angle of the PI-2611 polyimide can be modified by chamber pressure control (see Figure 4.14(a)).

![Figure 4.13: SEM images of cross-sections of etched patterns of ProLIFT/PI-2611 polyimide stacks with SiO\(_x\) hard mask on top for various etching chamber pressures. Note that there are no organic residues on the silicon substrate after polyimide etching. The white layer visible on the images on the underside of the SiO\(_x\) hard mask as well as on the substrate, is gallium residue deposited during FIB milling used to prepare the cross-sections of the etched profiles. The indicated scale is common to all images.](image)

The \( \alpha \) angle of the PI-2611 sidewall profile was found to vary from 8° from the vertical for 5 mTorr pressure to about 25° for pressures of 30 mTorr and above. In contrast, the angle of the upper portion of the PI-2611 sidewall was found to be independent of the chamber pressure (angle \( \beta \) as defined in Figure 4.12(d)), and found to be close to 40° from the vertical.

In addition, and as presented in Figure 4.14(b), within the range of the investigated process pressures, we observed a strong positive correlation between the etch rate and chamber pressure. The lowest etch rate of PI-2611 polyimide (570 nm/min) was achieved
for the lowest process pressure (5 mTorr), and the highest etch rate (860 nm/min) was achieved for the highest process pressure (40 mTorr).

To verify the influence of loading effects on the etch rate, we repeated selected experiments on 2-inch diameter silicon wafers. The etch rates and sidewall profiles of the polyimide measured on 2-inch wafers were found not to be noticeably different in comparison to the smaller samples examined in this work (2x2 cm²). While isotropic etching of larger samples may give rise to edge effects, our observations agree with the findings of Turban and Rapeaux [181], where no noticeable loading effects were observed in the low-pressure regime.

The overall positive correlation between the $\alpha$ angle and process pressure can be attributed to increasing chamber pressure enhancing the chemical etch rate component, whereas the ion bombardment enhanced vertical component of the etch rate remains unaffected or increases at a lower rate than the chemical etch rate [198]. These differences appear to be the principal reasons for sidewall profile variability with chamber pressure.

The following section will describe the last segment of experimental polyimide etching work, which presents our innovative hard mask for polyimide sacrificial layer etching.

**Hard mask**

The purpose of the hard mask is to protect the sacrificial layer during RIE etching process. It also needs to be transparent to aid photolithographic alignment. Suitable materials for the hard mask in our facility were therefore limited to SiN$_x$, SiO$_x$, and thin-film silicon (up to 400 nm). SiO$_x$ was chosen due to its simple deposition process.
During our process development, the SiO$_x$ hard mask was liable to delaminate causing under-etch around the mask pattern. This phenomenon was similar to that previously reported with the usage of metal hard masks [114]. Such delamination is often attributed to residual stress in the deposited masking thin film, as well as to any differences in thermal expansion coefficients between the layers, which can lead to cracks and delamination near the etch holes [199]. All of these issues were eliminated by using the adhesion promoter APX-K1, which was applied between all interlayers. Application of an adhesion promoter is recommended by the PI-2611 polyimide manufacturer [170].

The last part of the polyimide etching investigation is a process of hard mask lift-off used to remove the SiO$_x$ hard mask subsequent to the etching of the polyimide. This process allows patterning of the polyimide sacrificial layer and removal of the silicon oxide hard mask in a positive photoresist developer, i.e. a weak base solution. We avoided the use of hydrofluoric acid solutions to remove the oxide mask due to the fact that any exposed oxide layers that may be required to remain elsewhere on the chip would also have been etched.

The subsequent paragraph will present a short description of the release process for overlying structural layers, where polyimide thin film is removed in an oxygen plasma.

### 4.2.3 Etching process for device release

The devices fabricated with polyimide sacrificial layers can be released in an oxygen plasma. Oxygen plasma allows for relatively fast etching of polyimide without influencing any other structural materials, like metals, silicon, silicon oxide and silicon nitride. That allows for a clean and reliable release process. The rate of the release process can be increased by exposing fabricated devices to UV radiation before etching [200]. UV light will destroy some bonds in the polyimide thin film, which will lead to increased etching rates in an oxygen plasma. For dry release of the polyimide sacrificial layer we used an oxygen plasma in a March Instruments PM600 barrel asher at 1000 mTorr pressure and 150 W RF power.

The next section introduces a new material for the realisation of our devices – silicon nitride, which is used as the device structural layer.

### 4.3 Silicon nitride as a structural layer

Silicon nitride was selected to serve as the structural layer (see Figure 4.15) due to its excellent mechanical properties (Young’s modulus and intrinsic stress) which can be easily controlled during the deposition process [201, 202], and the high etch selectivity to both amorphous silicon and polyimide sacrificial layer materials considered in this work. Thin film SiN$_x$ layers were deposited using a Sentech SI500 ICPCVD system, with planar ICP antenna allowing quality material with tunable stress to be achieved. The adopted deposition recipe is presented in Table 4.6. The tensile stress can be tuned by adjusting the process parameter space, in particular by changing the SiH$_4$ to NH$_3$ ratio and the deposition temperature.
Figure 4.15: Simplified schematic of surface micromachined MEMS device with the silicon nitride as a structural layer.

Table 4.6: Silicon nitride deposition recipe using Sentech ICPCVD tool which is compatible with our device design regarding residual thin film tensile stress value.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiH4 flow</td>
<td>8.8 sccm</td>
</tr>
<tr>
<td>NH3 flow</td>
<td>10.2 sccm</td>
</tr>
<tr>
<td>He flow</td>
<td>167.2 sccm</td>
</tr>
<tr>
<td>Ar flow</td>
<td>542.4 sccm</td>
</tr>
<tr>
<td>Pressure</td>
<td>19.4 sccm</td>
</tr>
<tr>
<td>RF Power</td>
<td>0 W</td>
</tr>
<tr>
<td>ICP Power</td>
<td>300 W</td>
</tr>
<tr>
<td>Temperature</td>
<td>250 °C</td>
</tr>
<tr>
<td>Deposition rate</td>
<td>16.7 nm/min</td>
</tr>
<tr>
<td>Tensile stress</td>
<td>80 MPa</td>
</tr>
</tbody>
</table>

4.3.1 Etching process for thin film patterning

The silicon nitride structural thin film layers were patterned using a CF$_4$/O$_2$ plasma in an ICPRIE process. The adapted recipe is presented in Table 4.7 and provides sufficient etch selectivity to the photoresist mask for room temperature etching. For the adopted thickness of 2 μm for the SiN$_x$ structural layer, we use a 7 μm thick photoresist mask, which can survive the 22 min long dry etching process.

Table 4.7: Silicon nitride etching recipe using Plasamlab 100 ICPRIE tool.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CF4 flow</td>
<td>34.0 sccm</td>
</tr>
<tr>
<td>O2 flow</td>
<td>1.0 sccm</td>
</tr>
<tr>
<td>Pressure</td>
<td>2.0 Pa</td>
</tr>
<tr>
<td>Temperature</td>
<td>20 °C</td>
</tr>
<tr>
<td>RF power</td>
<td>100 W</td>
</tr>
<tr>
<td>ICP power</td>
<td>150 W</td>
</tr>
<tr>
<td>Etch rate</td>
<td>50 nm/min</td>
</tr>
</tbody>
</table>
4.4 Summary

In this chapter, we extensively investigated the properties and processing methods of two materials (HD Microsystems PI-2611 polyimide and amorphous silicon) suitable to serve as the sacrificial layer, as well as silicon nitride material for the structural layer. We have presented the complete fabrication process flow for each sacrificial layer material, from deposition, through patterning to final removal. Then, we presented the process flow for silicon nitride used as a structural material in our device fabrication process. All these processes are necessary for device fabrication, as described in the Chapter 5.
Chapter 5

Device fabrication, assembly and measurement set-up

This chapter describes the fabrication process of our AFM probe module using surface micromachining, a method compatible with standard CMOS processing. To compare and contrast, first, we describe the fabrication process of a commercial AFM cantilever probe, which uses bulk silicon micromachining. Then, we present an overview of the whole fabrication process followed by a description of each step in detail. The fabrication is performed on planarised silicon photonics chips with buried waveguides, and we begin with the outline of the silicon photonics fabrication process, which was undertaken in an external facility. Subsequently, we describe our approach for mounting of the AFM tip on the surface micromachined cantilevers using FIB cutting, transfer, and welding.

5.1 Standard AFM cantilever probe fabrication process

Standard AFM probes are fabricated using bulk silicon micromachining, a technique where devices are fabricated by removing bulk material from the processed substrate. The fabrication process starts with a silicon wafer of $<100>$ crystallographic orientation. Initially, the wafer is thermally oxidised on either side in order to create a reliable hard mask for wet etching of silicon, as shown in Figure 5.1(a). The thermal silicon oxide is patterned on both sides with a photoresist mask and wet etched in hydrofluoric acid (HF), as shown in Figure 5.1(b). The, created openings in the silicon oxide hard mask allow wet anisotropic silicon etching forming sidewalls with an angle of $54.74^\circ$ to the surface due the $<100>$ orientation. In this way, sidewalls are etched from all sides in the wafer forming a sharp silicon tip during the wet anisotropic etch. Silicon can be etched in two solutions: KOH or TMAH. TMAH solution is preferable due to better selectivity to the silicon oxide hard mask. As depicted schematically in Figure 5.1(c), the directional etching process defines both trenches and a sharp tip depending on the mask. Subsequently, the oxide hard mask is removed in a wet HF process, and the sample is covered with silicon nitride ($\text{SiN}_x$) from the top tip side, protecting the sharp tip fabricated in the previous step, as shown in Figure 5.1(d). Finally, the exposed silicon is etched from the unprotected back-side of the
Figure 5.1: Fabrication process of standard AFM cantilever probes after Russell [203]. The process starts with (a) thermal oxidation of a $\langle 100 \rangle$ silicon wafer, and (b) thermally patterning on both sides of the wafer. Subsequently, the uncovered silicon oxide is removed by a wet chemical etch, followed by (c) silicon anisotropic etching to form a sharp tip. (d) In the next step, the cantilever and tip are covered with silicon nitride on the top tip side, serving as a protective layer during the additional wet silicon etch to thin the cantilever to the desired thickness. Finally, the cantilever is released by removing the silicon nitride in a wet etch.

wafer to create a cantilever as shown in Figure 5.1(e). The cantilever probe is ready to use after a wet etch of the silicon nitride, thus releasing a suspended cantilever with a sharp tip.

The fabricated silicon tips can be additionally sharpened by thermal oxidation, and subsequent etch of the grown oxide in HF acid. The next section will introduce our fabrication process, which uses surface micromachining.

5.2 LumiMEMS™ based AFM probe fabrication process

In contrast to the bulk machining approach described above, our MEMS cantilever with an integrated on-chip optical readout was fabricated using surface micromachining. Fabrication of the AFM probe was performed in four main processing steps.

The first step was the fabrication of planarised photonics chips with buried waveguides. The silicon photonics chips were fabricated by Laboratoire délectronique des technologies de linformation (LETI) using standard silicon photonics industry techniques [61]. The second step was the fabrication of an unreleased MEMS cantilever using the planarised silicon photonics chip as a substrate. This step consisted of thin metal layer deposition, patterning and registration to define the metal electrodes, the sacrificial layer, and the structural materials of the cantilever (gold, silicon nitride and again gold). Subsequently, in the third step, a standard commercial AFM silicon tip was transferred and welded to our cantilever using an FIB approach. Finally, the fourth step consisted of releasing the fabricated device via sacrificial layer removal in an oxygen plasma. The MEMS fabrication process is shown in Figure 5.2.
5.2. LumiMEMS™ based AFM probe fabrication process

Figure 5.2: The main steps of cantilever fabrication process. Fabrication starts on top of (a) a planarized silicon photonics chip with (b) the electrode metallisation. Subsequently, (c) the sample is covered with polyimide sacrificial layer, which is cured and patterned. Successive steps consist of (d) metal mirror deposition and patterning, as well as (e) deposition and patterning of silicon nitride structural layer and the metallisation. Prior to (g) the device release, (f) a silicon sharp tip is installed using FIB.

5.2.1 Silicon photonics chip

Silicon photonics chips were designed with an array of separate waveguides designed to be single mode at 1550 nm. Each silicon waveguide included three gratings: an input, interrogating, and output grating as shown in Figure 5.3. All three gratings were connected in series using adiabatic tapers within the straight silicon waveguide.

The fabrication process to produce the buried waveguides is depicted in Figure 5.4. The silicon photonics chips were fabricated using a SOI substrate, which consisted of a 700 μm thick silicon wafer covered with 2 μm thick silicon oxide (SiO₂) and an overlying 220 nm thick epitaxial layer of silicon (see Figure 5.4(a)). The waveguides were formed in the top epitaxial silicon layer (see Figure 5.4(b)) with the pattern transfer defined using lithography. The top silicon layer was etched through its entire thickness using RIE to form the waveguide. Subsequently, using deep-UV (DUV) lithography, the grating pattern was etched into the waveguides to a depth of 70 nm using RIE (see Figure 5.4(c)). This single process step defined all the gratings within the silicon waveguide. Subsequently, the whole wafer was covered with a top silicon oxide thin film using chemical-vapour deposition (CVD) (see Figure 5.4(d)). This layer was then planarised using chemo-mechanical polishing (CMP) to achieve a final thickness of 100 nm above the silicon waveguide (see Figure 5.4(e)). The silicon strip between the top and bottom silicon oxide layers forms the waveguiding
structure. The fabricated wafer with buried silicon photonics waveguides was diced into chips with dimensions of 15×15 mm², which served as substrates for the subsequent MEMS fabrication described in detail in the next section.

5.2.2 MEMS cantilever fabrication

Cantilever fabrication commenced on top of the planarised silicon photonics chip (see Figure 5.2(a)) beginning with deposition of the bottom metallisation layer (see Figure 5.2(b)). Metallic electrodes are necessary for cantilever actuation and can be used for electrostatic actuation during tapping mode AFM operation or device characterisation. Metal pads were defined using a lift-off technique, which allows for precise patterning of the thin films with limited deposition conformity. First, the substrate was spin-coated with a 3.5 μm thick layer of negative photoresist which, after patterning, included a negative sidewall angle leading to non-continuity in the subsequently deposited layer of metal, as shown in Figure 5.5. The metallisation, which included a 5 nm chromium adhesion layer and 100 nm of gold, was deposited using thermal evaporation. After metal deposition, the photoresist was dissolved in acetone, thus removing the overlying metal thin film and leaving the desired pattern on the sample.

The deposition of the sacrificial layer followed the definition of the electrodes (see Figure 5.2(c)). The thickness of the sacrificial layer defines the gap between the substrate and the suspended cantilever. The mechanical parameters of the sacrificial layer may have a significant influence on the parameters of subsequently deposited structural layers. We
5.2. LumiMEMS™ based AFM probe fabrication process

![fabrication process diagram]

**Figure 5.4:** Fabrication process of the silicon photonics chip with buried waveguides after [67]. Process starts with (a) blank silicon-on-insulator (SOI) wafer with (b) silicon etching process to define the waveguides. Subsequently, (c) gratings are created with 70 nm deep silicon etch and (d) the whole wafer is covered with silicon oxide thin film. Finally, (e) the silicon oxide is polished to a thickness of 100 nm above the waveguides. Figure (e) shows the final cross-section of grating and waveguide along A–A' as shown in Figure 5.3.

Independently considered polyimide and amorphous silicon to serve as the sacrificial layer. Both of these layers were thoroughly investigated as described in Chapter 4. We have selected polyimide, and the decision was triggered by the use of a commercial silicon AFM tip and its installation before device release. Using a silicon tip eliminated the possibility of using a silicon sacrificial layer as both would be removed when the sacrificial layer is removed during the device release process. The silicon sacrificial layer can be considered.

![directional deposition diagram]

**Figure 5.5:** Photoresist with negative sidewall slope, covered with overlying gold thin film after a directional deposition process in a thermal evaporator. The sample is ready for the lift-off process with photoresist edge exposed to the solvent, which removes the photoresist layer along with the overlying gold thin film, thus leaving the patterned gold deposited directly on the substrate.
for devices utilising metal or dielectric tips, or for devices where the tip will be installed after device release. Silicon oxide, although a popular MEMS sacrificial layer, was not considered as a sacrificial layer for our devices, since the top surface of the silicon photonics chip is planarised using silicon oxide.

Polyimide PI-2611 from the HD Microsystems was spin-coated on the samples and cured. The anchor holes were defined via dry etching using a silicon hard mask through a process carefully tailored to provide an appropriate sidewall profile. On the patterned sacrificial layer, the metal mirrors on the underside of the cantilevers were fabricated (see Figure 5.2(d)). The metal mirrors are used as reflectors for the light diffracted out of the plane of the interrogating grating and towards the cantilevers. In addition, the metal mirrors functioned as the top electrode for electrostatic actuation. As shown in Figure 5.6, the metal mirrors consisted of a 50 nm layer of gold with a 5 nm chromium stiction layer that were deposited using thermal evaporation and patterned using lift-off.

![Figure 5.6: Schematic cross-section of the cantilever fabricated on top of the sacrificial layer using a bottom-up micromachining technique consists of the following layers, gold (50 nm), chromium (5 nm), silicon nitride (2 μm), chromium (5 nm), gold (50 nm). The chromium layer provides adhesion of gold to the silicon nitride structural layer.](image)

Subsequently, a 2 μm thick silicon nitride structural layer was deposited using ICPCVD. It was followed by thermal evaporation and lift-off patterning of a top metal layer consisting of 5/50 nm of chromium/gold and being a mirror image of the metal reflective layer under the silicon nitride structural layer (see Figures 5.6 and 5.2(e)). The top metal thin film had two functions: it provided a conductive surface for subsequent FIB processing; and it served to provide a symmetric cross-sectional geometry for the suspended cantilever, minimising any initial deflection due to stress imbalance and reducing sensitivity of the cantilever to temperature changes. After top metal deposition and patterning, the silicon nitride thin film structural layer was etched using the ICPRIE technique with a thick photoresist mask defining the cantilever geometry. Importantly, the removal of the sacrificial layer and cantilever release (see Figure 5.2(g)) is delayed until after the installation of a sharp silicon tip via the process described in the following section (see Figure 5.2(f)).

5.2.3 Tip attachment for AFM imaging

There are several methods of tip fabrication for AFM probes. The most common are silicon tips fabricated by wet etching of bulk silicon as described previously in Section 5.1, and shown in Figure 5.7. These AFM tips are cheap, reliable and provide high yield
5.2. LumiMEMS™ based AFM probe fabrication process

![Figure 5.7: Commercial AFM silicon probe fabricated using standard bulk micromachining process. The SEM images show: (a) the AFM cantilever holder with a cantilever, (b) a magnified view of the free end of the cantilever with a sharp tip, (c) a magnified view of the AFM tip.](image)

...during fabrication. However, an adaptation of this fabrication process for realisation of cantilevers with our integrated on-chip optical readout can be problematic using available equipment, as photonics chips are incompatible with processes used for commercial AFM probe fabrication. Most commercial AFM probes are fabricated using bulk micromachining, that involves thermal silicon oxide growth and wet etching processes. These traditional processes used to form the cantilever probe may negatively impact the silicon photonics features. However, the fabrication process can be decoupled by fabricating the cantilevers separately from the silicon photonics chip and subsequent bonding.

An alternative approach is tip fabrication on top of a tipless cantilever. Folch et al. [204] presented a method for fabrication of silicon tips on top of silicon nitride cantilevers. The fabrication process was based on isotropic etch of silicon from a thick silicon layer deposited on top of a silicon nitride cantilever. The tip was created due to under-etch of the thick silicon layer during the etching process. Schneider et al. [13] presented a process which allowed the fabrication of SU-8 cantilevers with a sharp tip using silicon moulds, which achieved their desired performance. However, organic AFM tips have generally about an order of magnitude larger contact area of the tip than silicon tips (apex radius 300 nm or more), which is a significant limitation of that technology. Lee et al. [115] presented hydrogel AFM tips with tip radius below 20 nm and significantly better performance than the SU-8 tips. The small tip area was achieved by introducing compression to a polydimethylsiloxane (PDMS) mould during tip fabrication. Ximen and Russell [205]
Chapter 5. Device fabrication, assembly and measurement set-up

Figure 5.8: Tungsten test tips fabricated in this work using FIB on a silicon cantilever. The tungsten tips were deposited as high aspect-ratio pillars and subsequently milled to achieve a sharp tip at the end of the pillar.

presented a technique based on FIB etching of silicon nitride cantilevers deposited on top of a previously tip-shaped sacrificial layer mould. The subsequent etching process allows fabrication of a sharp tip made from the same material as the cantilever. Additionally, they presented FIB-deposited high aspect ratio tips that can be fabricated on top of cantilevers or broken tips. Recently, Ageev et al. [206] presented similar high aspect ratio AFM tips made of tungsten, instead of the most popular FIB-used material platinum. Tungsten tips have better mechanical properties than platinum tips and presented lower wear over time.

The FIB allows fabrication of sharp tips using platinum or tungsten material. Platinum tips are easy to fabricate, but they generally have a limited lifetime due to low hardness, especially in contact mode. The use of tungsten allows fabricating high aspect ratio tips of excellent mechanical properties. We performed several attempts to fabricate platinum and tungsten tips achieving significant outcomes. Our fabricated AFM tips are shown in Figure 5.8. We found that the primary limiting factor of this approach is localised heating of the cantilever surface during material deposition and milling. Since our cantilevers were made mainly of SiN$_x$, supported by a polyimide sacrificial layer, they had limited thermal conductivity. The heating during the FIB deposition process led to significant temperature increase and thermal decomposition of the underlying polyimide. The heating was directly related to the deposition rate and processing time. We found that it would have been possible to use platinum and tungsten deposited tips if silicon was used as the sacrificial layer. However, we decided to proceed with the use of polyimide as the sacrificial layer and to adopt an alternative approach for tip formation on the surface micromachined cantilevers.

Our adopted method was similar to the method presented by Wang and Butte [207], who presented the process of AFM tip transfer to a tipless cantilever. The aim of their work was fabrication of a high aspect ratio tip at the end of a long cantilever. The silicon tip was shaped to the desired geometry while attached to a firm substrate and subsequently
5.2. LumiMEMS™ based AFM probe fabrication process

Figure 5.9: AFM tip transfer from commercial cantilever to a fabricated device undertaken in this work. (a) Top view image of the U-shape milled pattern around the silicon tip still attached to the commercial AFM cantilever. The micromanipulator needle is attached to the silicon tip. (b) Side view image of our unreleased cantilever with attached silicon tip welded using platinum.

transferred to a suspended cantilever using a micromanipulator. The tip was attached to a micromanipulator and removed from the substrate by cutting it at the angle of 52° from vertical with a FIB. Subsequently, the tip was transferred to the suspended cantilever and attached by localised platinum deposition filling the gap between the cantilever and the tip. This method allowed tuning of the angle of the fabricated tip.

However, we used a standard commercial AFM cantilever that was FIB milled around the tip in a U-shape, leaving a tip attached to the host cantilever by a small piece of silicon, as shown in Figure 5.9(a). Next, the micromanipulator needle was temporarily welded to the partially removed tip (see Figure 5.9(a)) supporting it for the full release and allowing its transport to our surface micromachined cantilever. The U-shape pre-cut was needed prior to welding of the transfer needle as the needle interfered with FIB cutting. Subsequently, the transferred tip was platinum welded around the edges to attach it accurately to our pre-surface micromachined cantilever. This approach minimised FIB local heating during the process. The described process rendered an AFM probe with a sharp tip ready for release via the removal of the sacrificial layer that is described in the following section.

5.2.4 AFM probe release

The last step of the AFM probe fabrication process is the removal of the sacrificial layer and device release (see Figure 5.2(g)). We used an oxygen plasma to remove the polyimide sacrificial layer. The release process depends on the sacrificial layer used. If silicon rather than polyimide was adopted as the sacrificial layer, we could have used a wet etch in KOH or TMAH solutions, dry etch in SF$_6$ plasma, or a dry chemical etch in the XeF$_2$ vapour.
5.3 Overview of the fabrication process

The main elements encompassing the fabricated probe are the suspended cantilever and the underlying waveguide with an interrogating grating etched into the waveguide underneath the tip. The 220 μm long and 20 μm wide cantilever attached to the middle of 120 μm long and 50 μm wide double supported beam formed a T-shaped geometry as illustrated in Figure 5.10. The suspended structure was fabricated as a tri-layer symmetrical stack of gold (50 nm), silicon nitride (2 μm) and gold (50 nm) thin films. The gold layer facing the interrogating grating has two purposes: (i) it was used as a suspended electrode for electrostatic actuation of the cantilever, and (ii) as the layer reflecting the light diffracted by the interrogating grating from the underlying waveguide.

The gold layer on the opposite side of the cantilever was used as a stress compensation layer to balance the residual stress gradients across the structure. This keeps the cantilever relatively flat. However, both gold thin film layers (each 50 nm thick) were in electrical contact to reduce the charging effects during FIB welding of the silicon tip. The adopted geometry allowed us to minimise any significant undesired deflections often associated with the anchor effect and deflections associated with the differences in the thermal expansion coefficients of the used materials (silicon nitride and gold). Figure 5.11 shows a comparison of cantilever deflections obtained for cantilevers with a single anchor and our T-shape design, which clearly demonstrates its benefits.

Electrostatic actuation was performed by means of electrostatic attraction between the cantilever gold layers and a gold electrode deposited on the silicon photonics chip directly under the cantilever. During operation the electrostatic attraction towards the photonics chip due to the voltage potential difference between the electrodes was balanced by the mechanical restoration force. This allowed AC and DC actuation for characterisation of the readout response. It may also be useful during AFM imaging (i.e. tapping mode), however, this was not performed during this thesis work.
Figure 5.11: Cross-sectional profiles along the cantilever with a single anchor geometry depicted by the SEM micrograph presented in inset (A) along the cross-section (A–A'); and along the cantilever of a T-shaped double anchor geometry presented in inset (B) along the cross-section (B–B'). The profiles were recorded using a Zygo NewView 7300 white light optical surface profilometer.

5.4 Sensing module assembly

The laser light was coupled in and out of the chip using identical input/output gratings and single mode optical fibres (SMF-28) [83, 84], as shown in Figure 3.8. The facets of the optical fibres were polished at an angle of 40° to achieve total internal reflection at the end facet for light propagating in the core of the fibre. Using index matched UV curable adhesive the fibres were glued to the chip parallel to the buried waveguide with the 40° polished ends aligned over the grating couplers. In this position, the polished fibre facets reflected the light out of the fibre core at an angle of 10° from vertical to suit the coupling angle of the grating couplers [75, 208].

5.5 Measurement set-up

5.5.1 Readout module characterisation and calibration set-up

Electrostatic actuation of the cantilever was performed using the fabricated on-chip electrodes, which allowed characterisation of the read-out response as a function of cantilever height above the interrogating grating. The electrodes were connected to a signal generator, through a high voltage amplifier to perform DC actuation of the device in the range of 0–135 V. As the cantilever was actuated, both the optical output power from the output waveguide grating and the gap between the cantilever and substrate were independently measured.

The interrogating light was provided by a 1.55 μm single mode, low noise, laser source through SMF-28 optical fibres and a polarisation controller, as the grating couplers used
in our interferometric readout are polarisation sensitive. After being modulated by the cantilever motion the output power from the waveguide was coupled into an indium gallium arsenide (InGaAs) photodetector (Thorlabs DET08CFC/M), amplified using a transimpedance amplifier (Femto DLPCA-200), and logged using a National Instruments USB-6009 analog-to-digital converter (ADC). Simultaneous to the measurement of the transmitted light, the deflection of the cantilever was measured independently using a white light optical ZYGO NewView 7300 profilometer.

5.5.2 AFM imaging set-up

The integrated measurement set-up is schematically presented in Figure 5.12. The fabricated AFM probe was integrated into a commercial AFM system (Digital Instruments D3000 with DMLSG piezoelectrical scanner, manufactured in 1995) in an inverted configuration by placing the on-chip readout probe on the sample stage and having a calibrated test sample attached upside down to the piezo scanner, where a standard AFM cantilever is normally placed. We adopted this arrangement in order to simplify the AFM probe holder, since it has optical fibres (SMF-28) glued to the silicon photonics chip. The standard AFM cantilever holder, designed to hold the scanning cantilever with a tilt angle of 11° to the horizontal was replaced by a glass microscope slide with the reference sample attached using Crystalbond wax. The AFM probe chip was attached to the sample table with Kapton tape.

The optical connections were set similarly as previously described for the electrostatic actuation described in a Section 5.5.1. During AFM imaging the cantilever response signal from the Stanford Research Systems SR570 was interfaced to the DI D3000 using a Digital Instruments breakout box, and connected as an input to the feedback loop signal. All imaging in this work was performed in contact mode, with both an open and closed feedback loop. The open feedback loop is not commonly used in AFM imaging. However, imaging with open feedback loop allows us to identify the influence of noise from the Z-axis piezo-scanner on the experiment results.

5.5.3 Noise spectrum measurement set-up

The measurements of the noise spectrum were performed in a different configuration, where the Thorlabs DET08CFC/M photodetector and Stanford Research Systems SR570 transimpedance amplifier were replaced by a photodetector module with build-in low dark current InGaAs PIN photodiode and a fixed gain low noise transimpedance amplifier, which had significantly lower amplifier electronic noise than the Stanford Research Systems SR570. However, it had limited functionality (no gain variability and DC offset) which hampered its integration with the D3000 AFM system.

The signal spectrum measurements were obtained using an Agilent 89410A vector signal analyser (VSA) recording the photodetected and transimpedance amplified signal from the AFM probe. Additionally, the spectrum response from the laser vibrometer (Polytec OFV-5000) was also measured. The laser vibrometer measures the displacement of the
Figure 5.12: Schematic view of the measurement set-up. The LumiMEMSTM probe is installed on the sample stage of a Digital Instruments D3000 AFM, and the sample is attached to the piezo-scanner. 1550 nm laser light is connected to the input optical fibre of the LumiMEMSTM probe, and after being modulated by cantilever motion, it is detected by an InGaAs photodiode via an output optical fibre. The detecting photodiode output electrical signal was amplified with a transimpedance amplifier.

cantilever with a sensitivity of 50 nm/V, which allowed us to confirm the value of the resonant frequency and quality factor of the fabricated cantilever, as well as verify the calibration and sensitivity slope of the integrated readout.

5.6 Data processing

The AFM images were acquired using Digital Instruments D3000 software and then processed using a Gwyddion 2.48 software package [209] using standard algorithms for data levelling by mean-plane subtraction, alignment of the rows using the polynomial method, and shifting minimum (or average) data point to zero. The height distributions and RMS surface roughness were calculated using built-in statistical functions. For additional analysis, we used Fityk software [210], which allowed us to fit all recorded probability density noise graphs with a Gaussian function and calculate peak parameters.

5.7 Summary

In this chapter, we presented the LumiMEMSTM AFM probe fabrication process. We have described the fabrication of the silicon photonics chip performed in an external facility.
using standard fabrication process, followed by the fabrication of a cantilever structure on top of an 8 µm thick polyimide sacrificial layer. The fabrication process was completed with the installation of the AFM tip, transferred from a commercial AFM cantilever using focused ion beam (FIB) and subsequent device release in oxygen plasma. In this chapter we presented a few methods available for tip fabrication, including bulk and surface micromachined silicon tips, moulded organic (SU-8 and hydrogel) tips, FIB etched silicon nitride tips, FIB deposited platinum and tungsten tips, and our adopted approach of tip transfer from a commercial silicon AFM cantilever, which further allows the use of a tip comparable with those typically used in the AFM industry. The fabrication process is followed by device assembly, describing the process of gluing angle-polished optical fibres to the surface of the silicon photonics chip.

The last part of this chapter presented the measurement set-up, and described the equipment used for each step of device characterisation. Finally, integration of the LumiMEMS™ probe with the Digital Instruments D3000 AFM imaging system was described, and software for capturing AFM images of a reference sample were presented.
Chapter 6

AFM imaging and probe performance demonstration

To demonstrate the developed AFM LumiMEMS™ sensor module, we performed four independent experiments: (i) characterisation of cantilever motion in response to electrostatic actuation, (ii) the acquisition of AFM images representing surface topology of a known reference sample and comparison with a modern commercial AFM system (Bruker Dimension ICON), (iii) measurement of the noise response of the non-scanning cantilever tip in contact with the sample as part of a complete AFM system, and (iv) measurement of unactuated Brownian motion of the free-standing cantilever.

6.1 Cantilever response to electrostatic actuation

The electrostatic actuation of the AFM cantilever probe was performed to characterise the position of the cantilever in terms of the amplitude of the optical signal transmitted through the underlying waveguide. As the actuation voltage was increased and the cantilever was attracted to the substrate, we simultaneously measured the waveguide output power and the cantilever position above the interrogating grating independently using a Zygo NewView 7300 optical profiler. Figure 6.1 presents the transmitted LumiMEMS™ signal as a function of cantilever downward deflection from its original undeflected position about 8.6 µm above the substrate. We observe a periodic response, which confirms the interferometric effect taking place as the gap decreases with increasing actuation voltage. As expected from the analysis in Section 3.2, the peaks and nulls were found to be repeated for a change in cavity height between the cantilever and grating of approximately $\frac{\lambda}{2 \cos \theta}$, where $\lambda$ is the wavelength of the laser source of $\lambda = 1.55 \mu m$ [15, 17] and $\theta = 10.6^\circ$ is the coupling angle of our grating. The slope between the consecutive minima and maxima relates to the change in the transmitted optical signal, $\Delta P$, that is associated with the change in the cantilever position, $\Delta z$. Detailed knowledge of this measured transfer function was subsequently used to calibrate the probe for AFM measurements, and is inversely related to the displacement sensitivity. The positive slope of the first peak is characterised as $\Delta P/\Delta z = 67 \mu W/\mu m$ giving displacement sensitivity $S = 3.9 \mu m^{-1}$, whereas the negative slope of the first peak is
Chapter 6. AFM imaging and probe performance demonstration

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure6_1.png}
\caption{Interferometric output of the measured AFM probe. The optical power transmitted through the AFM module as a function of the downward deflection of the cantilever from its rest position of 8.6 \( \mu \)m above the substrate and the corresponding electrostatic actuation voltage. Cantilever deflection was measured independently using a surface profiler. The two linear regions of positive and negative slope are marked on the plot.}
\end{figure}

characterised as \( \Delta P/\Delta z = -93 \ \mu \text{W}/\mu \text{m} \) giving displacement sensitivity of \( S = -5.4 \ \mu \text{m}^{-1} \). The asymmetry between slopes originates from the asymmetrical properties of the optical reflection in an Fabry-Pérot interferometer (FPI), which has been investigated in detail by Bruce and Clothier [211], Monzón et al. [212, 213], and Giust et al. [214]. The linear part of both positive and negative slopes of the interferometric response can be used unambiguously. The readout system provides up to 300 nm of unambiguous linear dynamic range, which can be extended using two or more laser sources with different wavelengths [16, 17]. It can be observed from Figure 6.1 that the peak transmission values of the optical signal for successive signal maxima increase as the cantilever deflection increases with increasing voltage. This occurs due to the increase of the finesse of the optical cavity. However, for the performed AFM measurements this change in the peak transmission values is not relevant as all measurements were performed on the first positive slope as indicated in the Figure 6.1.

6.2 AFM imaging of a reference sample

Characterisation of surface topography imaging using a LumiMEMS™ readout was performed and directly compared with a commercial AFM system. Figure 6.2 presents AFM images of the reference sample recorded using a commercial (OBD) AFM tool (see Fig-
ure 6.2(a)) and using the interferometric readout approach acquired in both open (see Figure 6.2(b)) and closed feedback loop (see Figure 6.2(c)) configuration. During the measurements, the LumiMEMS probe was integrated with Digital Instruments D3000 AFM as shown in Figure 5.12, and the performance comparison is made with a Bruker Dimension Icon, which is the successor of the D3000. For this experiment, we used a silicon carbide reference sample, 6H-SiC (0001) supplied by TedPella. The silicon carbide sample has characteristic steps of 0.75 nm in height fabricated on its top surface. The step height corresponds to half of the lattice constant. The average roughness of the terraces between steps is claimed to be 90 pm. Figures 6.2(a-c) present 2×2 µm² area surface topography images of the sample consisting of several steps. Each image consists of 6 to 7 terraces with a vertical increment of 0.75 nm between the consecutive terraces. Figures 6.2(d-f) show the probability density function (PDF) of the height distribution as measured on the reference sample. Each peak on a height distribution profile represents the level of single terrace, starting from the lowest to the highest within the measured area. The distances between peaks represent step heights, and can be used to verify the linearity of the measured readout response. The height and full width at half maximum (FWHM) of the peak are related to the measured surface roughness (noise) of each terrace. Narrower

![AFM images of a SiC-6H (0001) reference sample consisting of 0.75 nm high steps between consecutive terraces. The images cover an area of 2×2 µm². (a) The image was acquired using a Bruker Dimension Icon AFM system with the settings set to achieve the best quality image. (b) The image was acquired using our integrated on-chip interferometric readout with an open feedback loop, which means that the piezo-scanner along the Z-axis remained rigid. (c) The image was acquired using our integrated on-chip interferometric readout with a closed feedback loop, which means that the piezo-scanner was modulated along Z-axis to keep the scanning cantilever deflection constant. (d-f) The graphs show height distribution profiles for each AFM image.](image-url)
and higher peaks represent lower surface roughness of the terrace and Figures 6.2(d-f) also list the values obtained for the average distance between peaks and the FWHM for each of the measurements.

The AFM image measurement shows that the out-dated Digital Instruments D3000 AFM with a LumiMEMS™ readout is able to measure sub-nanometre features. The results shown in Figure 6.2 indicate that by using our interferometric readout we measure for the terraces of the reference sample an RMS roughness value that is about a half of the value measured using a commercial AFM (37 pm vs 62 pm). Based on the comparison of the RMS roughness values measured for the terraces using a modern Bruker Dimension Icon system equipped with OBD readout and our approach, we can infer that our readout achieves a noise level about half the magnitude of that of the tested OBD readout. The noise levels for images acquired with open and closed feedback loop are nearly identical. For this measurement, therefore, the feedback loop does not significantly contribute to the noise. It must be noted, however, that the presented surface roughness measurements do not have to accurately reflect the AFM apparatus noise characteristics, since measurements may be impacted by differences in the sharpness of the AFM tips used. The presented experiment was conducted primarily to confirm calibration of the tool, verify linear response and demonstrate sample imaging. Static noise characterisation, which is not impacted by the sharpness of the tip is performed in the following section, presenting the difference in performance between the original Digital Instruments D3000 tool and our readout.

6.3 Non-scanning AFM static noise

To investigate the effective noise characteristics for the LumiMEMS™ probe installed in an AFM system, we performed a static non-scanning system noise measurement. The measurement was conducted with the tip in contact with the sample, while the AFM probe remained stationary with the X-Y and Z-piezo scanners in static fixed positions. This experiment provided information about the noise level during AFM imaging by measuring the signal in conditions closest to surface imaging. Additionally, this experiment allowed for a direct comparison of noise performance between the LumiMEMS™ readout attached to the D3000 and the host D3000 OBD readout.

The results were logged as a stationery AFM image with the noise data logged line-by-line. Each line consisted of 512 points recorded with a scan rate of 1 Hz, and each image consisted of 512 lines. After images were collected, the PDF and RMS noise values were calculated.

Figure 6.3 shows the PDF and the calculated RMS noise values of the measurements performed using OBD and LumiMEMS™ readouts. We can see a significant difference in the noise PDF between the standard Digital Instruments D3000 AFM setup and our LumiMEMS™ readout. We obtained an RMS noise of 19 pm using the LumiMEMS™ readout, which was significantly lower than the 51 pm measured with the OBD readout of the Digital Instruments D3000. The reference noise measurements using the standard configuration of the Digital Instruments D3000 were recorded for three different commercial
AFM cantilevers. The MikroMasch silicon cantilevers had spring constants of 0.18 N/m (HQ:CSC17), 2.8 N/m (HQ:NSC18) and 40 N/m (HQ:NSC15). There was no difference between the noise images and the obtained PDF plots measured using the three different cantilevers, which means that there is no cantilever influence on the noise level (Brownian motion noise is suppressed).

These results confirm the relationship between RMS values from static AFM noise measurements for the OBD and our readout from the previous experiment, as shown in Figure 6.3. In both situations, the integrated interferometric readout achieved significantly better noise performance than the OBD readout. Our measurement of the static AFM noise shows an RMS noise level of 19 pm, which was lower than the lowest values reported recently by Dukic et al. [37], which was 25 pm for OBD readout and 32 pm for the piezo-resistive readout. The RMS noise level from the LumiMEMS™ probe is also significantly lower than the noise value achieved with the default configuration of Digital Instruments D3000 AFM tool, which was equal to 51 pm.

6.4 Deflection noise density of the cantilever

Characterisation of the deflection noise density (DND) of the cantilever [25], introduced in Section 2.3, was performed to investigate noise sources for our AFM readout approach, as
well as to measure the resonant frequency and the quality factor of the fabricated suspended cantilever.

The measurement required the acquisition of the frequency spectrum of the output optical signal transmitted through the LumiMEMS™ readout with the suspended cantilever unactuated above it. The cantilever vibration being characterised is stimulated by the ambient laboratory environment. This measurement is related to the ultimate noise floor achievable by an AFM system utilising our approach and operating in standard laboratory environment. Figure 6.4 presents the DND of the cantilever motion as a function of frequency measured using LumiMEMS™, as well as using an external vibrometer under the same conditions. The spectrum measurements were performed for the frequency range of 0 – 400 kHz, however during AFM imaging the Stanford Research Systems SR570 transimpedance amplifier was used since it provided more flexible interface options to the D3000 system, and the measured 3 dB measurement bandwidth of the Stanford Research Systems SR570 at the gain setting used, was 87 kHz and that value was used for minimum detectable deflection (MDD) calculations. The DND was measured using a vector signal analyser (VSA) and recorded as the RMS voltage density spectrum averaged over 100 trials. Averaging reduces the random noise of the measurement system by $\sqrt{100}$, assuming that our measured signal is stationary over a long period of time [215]. Measured values were converted to current density using a transimpedance amplifier with current sensitivity of $10^{-5}$ A/V, and finally to power density using the photodetector responsivity factor, $\gamma = 1.0$ A/W. The power density was then converted to deflection noise density using the displacement sensitivity measured of $\Delta P/\Delta z = 30 \mu W/\mu m$, which is the initial slope of the response shown in Figure 6.1 near zero deflection.

We can observe from Figure 6.4 that the Brownian motion signal dominates the measured frequency spectrum near the first resonant frequency of the cantilever, which is identified by the distinct peak in the frequency spectrum as predicted in Section 3.6. Additionally, the peak caused by second resonant frequency was identified. The Brownian motion of the cantilever, also known as thermomechanical motion, is not dependent on the readout technique, but is a function of the cantilever design. Using the DND measurements presented in Figure 6.4, we have found the first resonant frequency of our cantilever to be $f_0 = 46$ kHz with a quality factor $Q = 46$, and the second resonant frequency to be $f_1 = 280$ kHz. The second resonant peak is not visible in the vibrometer measured data as it is below the noise floor for this tool as observed in Figure 6.4. The measured first resonant frequency is higher than expected in Chapter 3 due to fabrication tolerances (i.e. higher thickness of the structural layer).

The values obtained for the resonance centre frequency peak amplitude are in agreement for both LumiMEMS™ and vibrometer measurements. The vibrometer has a calibrated displacement sensitivity of $50$ nm/V, which means that an output signal change of 1 V represents a displacement of 50 nm. Since the peak amplitudes as measured using LumiMEMS™ and the vibrometer are in agreement, the positive slope displacement sensitivity of our system presented in Figure 6.1 is validated, and that value was used for DND
Figure 6.4: Deflection noise density spectrum derived from vector signal analyser (VSA) current density measurements. The green trace shows Brownian motion signal measured using an optical vibrometer and the pink trace our interferometric readout. The effective measurement noise shown in the blue trace (which includes: dark current noise, photodetector shot noise, transimpedance amplifier electronics noise and noise of laser source) is dominated by shot noise. The effective measurement noise was acquired using an optical attenuator fitted in place of the silicon photonics chip and set to give the same optical power at the receiver. The solid black line shows the square root sum of squares of the calculated spectrum of the Brownian motion signal and the shot noise. The shot noise was estimated based on measured input power to the photodetector, $P_i = 3.7 \mu W$, using Equation 6.2.

calculations. This validation of the previously measured transfer parameters as presented in Figure 6.4 gives us confidence in the measured displacement sensitivity for our method.

Subsequently, we measured the effective measurement noise, consisting of all noise sources excluding the Brownian noise originating from the LumiMEMS™ probe. During this experiment, we replaced the LumiMEMS™ probe with a variable optical signal attenuator (JDS Fidel) and adjusted the attenuation to achieve the same level of signal transmission as observed for the set-up including the LumiMEMS™ probe. The attenuation was set to a value of 34.3 dB, which represents the signal losses of the input and output coupling between the optical fibre and silicon photonics chip, plus the signal losses resulting from the specific position of the cantilever. The losses are significantly higher than previously discussed in Chapter 3 due to misalignment during gluing of the optical fibres to the silicon photonics chip. Figure 6.4 shows that the effective measurement noise signal level is indistinguishable from the LumiMEMS™ measurement noise in the frequency range above the first resonant frequency peak. However, there is a noticeable difference for the low-frequency range
(below the first resonant frequency peak) between effective measurement noise and the LumiMEMS™ Brownian motion response, which is expected due to the different levels for shot noise and Brownian motion \((f \ll f_0)\) signals as can be seen in Figure 3.13 for our photodiode current of \(i_{\text{out}} = 3.7 \mu A\). This gap between shot noise level and Brownian motion response is expected to increase with increasing transmitted power \(P_t\). The average effective measurement noise was found to be about \(i_{\text{eff}} = 1.2 \text{ pA}/\sqrt{\text{Hz}}\), which can be represented as \(DND_{\text{eff}} = 36 \text{ fm}/\sqrt{\text{Hz}}\). This figure can be compared to the theoretical noise values originating at various points of our measurement set-up and calculated using manufacturer provided technical specifications. In our measurement system, the primary noise sources are: (i) electronic noise of the transimpedance amplifier; and (ii) shot noise of the photodetector. The square root sum of the squared noise values gives us the total calculated noise of the measurement system.

The electronic noise of the transimpedance amplifier for the Stanford Research Systems SR570, is specified by the manufacturer for the used current sensitivity of \(10^{-5} \text{ A/V}\) as an noise current density of \(i_e = 2 \text{ pA}/\sqrt{\text{Hz}}\) [104] and a \(DND_e = 61 \text{ fm}/\sqrt{\text{Hz}},\) while for the Femto DLPCA-200 the noise current density was defined as \(i_e = 0.45 \text{ pA}/\sqrt{\text{Hz}}\) [105] and a \(DND_e = 14 \text{ fm}/\sqrt{\text{Hz}}\). This noise level is only slightly worse than the Johnson noise level of transimpedance amplifier, dominated at low frequencies by the transimpedance, \(R_t = 100 \text{ k}\Omega\) (see Section 3.6). The Johnson noise was calculated using a modification of Equation 3.24 for Johnson noise current [102], and results in a thermal noise current density

\[
i_{jn} = \frac{\langle i_{jn} \rangle}{\sqrt{\Delta f}} = \sqrt{\frac{4k_B T \Delta f}{R_t}} \times \frac{1}{\sqrt{\Delta f}} = \sqrt{\frac{4k_B T}{R_t}} \tag{6.1}
\]

where \(k_B = 1.38 \cdot 10^{-23} \text{ J/K}\) is the Boltzmann constant, \(T = 300 \text{ K}\) is the temperature, and \(\Delta f\) is noise bandwidth, giving a Johnson noise current density \(i_{jn} = 0.41 \text{ pA}/\sqrt{\text{Hz}},\) which gives us \(DND_{jn} = 12 \text{ fm}/\sqrt{\text{Hz}}\).

The RMS value of the shot noise current density of the photodetector was calculated using a modification of Equation 3.22

\[
i_s = \frac{\langle i_s \rangle}{\sqrt{\Delta f}} = \sqrt{2e P_t \gamma \Delta f} \times \frac{1}{\sqrt{\Delta f}} = \sqrt{2e P_t \gamma} \tag{6.2}
\]

where \(e = 1.602 \cdot 10^{-19} \text{ C}\) is the electronic charge, \(P_t = 3.7 \mu W\) is the optical power on the detector, \(\gamma = 1.0 \text{ A/W}\) is the detector responsivity, and \(\Delta f\) is noise bandwidth. The shot noise current density was equal to \(i_s = 1.1 \text{ pA}/\sqrt{\text{Hz}},\) which can be represented as \(DND_s = 33 \text{ fm}/\sqrt{\text{Hz}}\).

The value of observed effective measurement noise \(DND_{\text{eff}} = 36 \text{ fm}/\sqrt{\text{Hz}},\) is approximately equal to the squared root of sum of squares of the shot noise and electronic noise of Femto DLPCA-200 amplifier used during this experiment. Consequently, they are the main contributing noise sources in our measurement presented in Figure 6.4. Since the value estimated for the shot noise \((DND_s = 33 \text{ fm}/\sqrt{\text{Hz}})\) is significantly higher than value of the electronic noise \((DND_e = 14 \text{ fm}/\sqrt{\text{Hz}})\) and nearly reaches the observed effective measurement noise value \((DND_{\text{eff}} = 36 \text{ fm}/\sqrt{\text{Hz}}),\) the readout is shot noise.
6.4. Deflection noise density of the cantilever

limited. However, during the AFM measurements presented in this section, the Stanford Research Systems SR570 amplifier was used in place of Femto DLPCA-200, limiting noise performance to its electronic noise level near the estimated $DND_e = 61 \text{ fm}/\sqrt{\text{Hz}}$.

The amplitude of the cantilever Brownian motion peak is found to be significantly greater than the level for shot noise and electrical noise sources. The frequency spectrum of the cantilever vibration near the first resonant frequency, $n_zB(f)$, was calculated using Equation 3.28. We used measured values of the resonant frequency, $f_0 = 46 \text{ kHz}$, quality factor, $Q = 46$ and the spring constant of $k = 1.2 \text{ N/m}$. The measured spring constant exceed the designed value due to fabrication tolerances (i.e. higher thickness of the structural layer). However, it is still acceptable for our experiments. Figure 6.4 shows the square root of the sum of squares of the calculated theoretical Brownian motion DND and the shot noise DND presented in Figure 6.4. This signal compares favourably with the measured Brownian motion signal using LumiMEMS™. The mean squared error function was used to determine value of spring constant from measured spectrum.

The DND allows us to calculate the MDD for our AFM probe. Using the average of our measured $DND = 36 \text{ fm}/\sqrt{\text{Hz}}$, we find that the MDD value in 87 kHz bandwidth used in the AFM imaging is equal to 11 pm. This MDD value is significantly lower than the value for a similar optical readout reported by Noh et al. [216], where they achieved an MDD of 54 pm with a bandwidth of 250 Hz. The $DND = 36 \text{ fm}/\sqrt{\text{Hz}}$ is the ultimate value demonstrated in this thesis for the LumiMEMS™ readout. This value can be achieved if Brownian motion noise is not significant, for example by mechanical damping of the Brownian motion during contact mode imaging.

Mechanical vibration of the cantilever can be stimulated with electrostatic actuation and thereby enable tapping mode measurements, although such measurements were beyond the scope of this work.
Chapter 7

Summary, conclusions and future work

In this thesis, we have presented an AFM probe using an integrated on-chip cantilever deflection readout, based on silicon photonics, merging the high sensitivity of an interferometric optical readout with on-chip miniaturisation. The realised AFM probe, being characterised by a deflection noise density (DND) of $36 \text{ fm/}\sqrt{\text{Hz}}$, achieved measurement noise levels surpassing present day optical beam deflection (OBD) and piezoelectric readouts.

The underlying concept of the adopted cantilever displacement detection methodology is based on the optical interferometric effect between an underlying on-chip interrogating waveguide and a suspended sensing cantilever. Using finite-difference time-domain (FDTD) simulations, we found that the silicon photonics-based probe sensitivity decreased with increasing gap between the interrogating grating etched in the waveguide and the cantilever reflecting surface. On the other hand, a relatively small gap limits the maximum travel distance of the probe, increases squeezed film damping \[76\], which lowers the quality factor of the cantilever, and increases the risk of cantilever snap-down. We selected a gap around $8 \mu\text{m}$ for our devices as it provides a good compromise between sensitivity and available travel distance.

The fabrication process of the sensing microcantilever probe was decoupled from the fabrication of the silicon photonics chip, which forms a planar surface for subsequent MEMS processing. The fabrication of the industry standard silicon photonics waveguides was outsourced and performed at LETI using SOI wafers. As such, the choice of the sacrificial material for subsequent MEMS micromachining was of significant importance as it is etched away in the final processing step. Silicon and polyimide were chosen as potential sacrificial layer materials due to their compatibility with the photonics chip substrate for subsequent MEMS fabrication. However, only polyimide was compatible with commercially available common silicon tips that were chosen for the AFM probe and which further allowed relevant performance comparison of our probe with commercial AFM instruments. Silicon sacrificial material could be used, for example, if the tips were formed using FIB-deposited platinum or tungsten.
Chapter 7. Summary, conclusions and future work

The MEMS cantilever fabricated for the AFM probe was surface micromachined and is anchored to the photonics substrate. Typical MEMS anchors can exhibit some non-ideal characteristics that cause undesired deformation of the suspended cantilever after release. Solutions to this effect were examined with a T-shaped anchor, which consisted of two anchors connected with a microbeam, and the cantilever was attached at the centre of this microbeam. The dimensions of the structure were based on the required spring constant near 1 N/m for contact mode measurements. Simulations confirmed the cantilever anchor performance, agreeing well with subsequent measurements.

The silicon photonics chip was connected with the laser light source and photodetector using single mode optical fibres to couple light into and out of the chip, via waveguide grating couplers. Several approaches were considered, and the lateral coupling of light through total internal reflection (TIR) of an angle polished fibre glued to the chip was chosen. The choice was based on the flexibility of this method necessary for integration of the AFM probe into an existing AFM system, thus allowing measurement of large-area samples. This method was characterised to have relatively low insertion loss, allowing the use of a low power laser source. It was found that the insertion loss can be significantly minimised, but was not investigated further [74].

The noise characteristics of the realised sensing probe module were determined to be influenced mainly by photodetector shot noise, with the electronic noise of the photodetector amplifier being significantly lower. The shot noise was found to dominate during characterisation of noise performance of the device for frequencies away from the cantilever natural mechanical resonance, where Brownian motion noise was found to dominate.

We have presented AFM imaging of reference samples using the developed probe, and demonstrated a significant reduction of the measured surface roughness compared to a modern commercial AFM tool (Bruker Dimension ICON). We achieved an AFM static image RMS noise floor of 19 pm, thus surpassing the performance of OBD (25 pm) and piezoresistive readouts (32 pm) [37]. A detailed analysis of the noise sources affecting the measurement methodology indicates that our readout approach is shot noise limited. Our system achieved deflection noise density $D_{\text{ND}} = 36 \text{ fm/}\sqrt{\text{Hz}}$, thus allowing higher resolution imaging with potentially higher imaging rates.

7.1 Future work

This thesis covered the initial work on the silicon photonics readout for AFM systems, which establishes the foundation for further improvement to the readout technique for increased sensitivity and additional functionality of the system. A clear trend in AFM imaging systems is increased accuracy and imaging speed along with simultaneous miniaturisation of the measurement systems. We believe our approach can fulfil these requirement, with further refinement.
7.1.1 Silicon photonics chip

The most significant limitation of our current AFM probe is due to the layout of the silicon photonics chip, which was designed for research into the readout technology, with application towards chemical sensing. Any high-profile elements, such as optical fibres, will limit the imaging area. U-shaped waveguides can be designed on the silicon photonics chip so that optical access can be located along one edge of the silicon photonics chip far from the cantilever. Furthermore, to be commercially viable, the AFM probe assembly must be low cost and easily replaced.

Current silicon photonics fabrication processes allow integrating an on-chip laser source and detector. Integration of the laser source and detector can reduce the AFM probe assembly process while making a complete device capable of supporting many AFM cantilevers [217], thereby reducing sample imaging times. However, the MEMS fabrication process needs to be conducted on top of these active components, which may constrain the processing conditions, for example limiting the processing temperature range. Perhaps a more viable approach is to have the consumable AFM probes as part of a passive photonics device, which can be easily exchanged.

One possible solution is to incorporate arrays of input and output gratings that are designed to overlap with a corresponding array built into the AFM instrument, where arrays of low cost vertical-cavity surface-emitting lasers (VCSELs) and photodetectors are housed. Such overlapping gratings have been shown to exhibit less than 1 dB insertion loss, when suitably aligned using a well-designed probe holder [218]. The other approach is to incorporate an additional silicon photonics chip that acts as an interconnector between the optical fibres and the AFM probe using grating-to-grating coupling of light [218].

7.1.2 Device fabrication

The current fabrication process uses a surface micromachined cantilever on top of the silicon photonics chip. This approach allows easy prototyping of AFM probes and can be used for high volume AFM probe fabrication. Moreover, bulk micromachining allows fabrication of a cantilever with a tip, which can then be bonded to the silicon photonics chip [217]. This process allows maintaining full compatibility with standard silicon AFM probes and the associated fabrication processes, since they are usually fabricated with bulk micromachining.

7.1.3 Operation in liquids

Operation in liquids is becoming an increasingly essential part of AFM measurements. For our interferometric readout, operation in liquids reduces the cantilever quality factor, which also applies to any readout approach, and causes a small change in the readout optical coupling angles, depending on the refractive index of the fluid. We do not anticipate that this would have a significant effect on the readout performance. An additional factor for
any optical readout is optical attenuation due to the fluid although, for our readout, the optical path is very small and so the additional attenuation should be acceptable.

Currently, a major limitation to the LumiMEMS™ readout, when used in a liquid, is the light coupling method utilising angle polished optical fibres to couple light to and from the silicon photonics chip. The angle polished fibres use total internal reflection (TIR) at the interface of the optical fibre face and air. Since the refractive index of water is significantly higher than air, total internal reflection will not occur. However, a solution is to use metal mirrors deposited on the angle polished face of the optical fibre, or use a completely different method of light coupling to the silicon photonics chip, such as tapered waveguide couplers or grating-to-grating couplers (see Section 7.1.1).

### 7.1.4 Tapping mode operation

Tapping mode AFM measurement is a commonly used AFM technique as it reduces the risk of damage to the imaged sample. Tapping mode can be realised using the existing AFM probes fabricated in this work. However, it requires a dedicated control system for built-in electrostatic actuation. Also, the readout noise can be reduced significantly, since tapping mode measurements can be performed with a very narrow signal bandwidth.

### 7.1.5 Imaging of challenging samples

Further development of our AFM readout can benefit from the demonstration of imaging more challenging samples, e.g. those requiring high resolution, or imaging of soft samples. Progressing along this path will require imaging using several different AFM cantilevers with different spring constants suitable for samples being investigated. Additionally, the use of tapping mode can be beneficial for surface characterisation of soft and fragile samples since it minimises the risk of sample damage during the imaging process.
Bibliography


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Appendix A

Author’s publication list arising from the PhD program

A.1 Journal Articles


A.2 Conference Papers
