Manufacture of Micro-Optical Elements for Imaging and Light-Guidance

by

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Declaration of Authorship

I, Mark Langridge, declare that this thesis titled, ‘Manufacture of Micro-Optical Elements for Imaging and Light-Guidance’ and the work presented in it are my own. I confirm that:

- This work was done wholly or mainly while in candidature for a research degree at this University.
- Where any part of this thesis has previously been submitted for a degree or any other qualification at this University or any other institution, this has been clearly stated.
- Where I have consulted the published work of others, this is always clearly attributed.
- Where I have quoted from the work of others, the source is always given. With the exception of such quotations, this thesis is entirely my own work.
- I have acknowledged all main sources of help.
- Where the thesis is based on work done by myself jointly with others, I have made clear exactly what was done by others and what I have contributed myself.

Signed: 

Date:
Many are the ions
Gallium falls like rain drops
The surface erodes

Bold ions strive forth
Kicking debris to the sky
And burrowing in

A surface wasteland
Sputtered surface and ions
The damage is done

Myself
Abstract

In this thesis we discuss the manufacture and characterisation of micro-optical elements, for guiding light into sub-wavelength beams & spots, and for use in super-resolution imaging.

A physical limit exists in microscopy where it is impossible to view object smaller than half the illuminating wavelength, via conventional means. In white light microscopy this creates an resolution limit of 321nm ($\lambda=500\text{nm}$, in air). This places a limit on the smallest objects a researcher can study using optical microscopy.

We present a method for fabricating plano-convex lenses which, when placed in near proximity to the samples, boost magnification of conventional microscopes by up-to 2.5x and resolve features below 200nm, with white light illumination. We also demonstrate a curved axicon Bessel-beam former, that produces long (>17$\mu$m) non-diffracting beams of light, that can be sub-wavelength in width ($\frac{2}{\pi}\lambda$).

In this thesis we contribute the following to current knowledge:

We describe a focused ion-beam milling technique to form bespoke geometry of parabolic & spherical curvature, including reflective dishes, of diameter 1-10$\mu$m, with a surface roughness of 4.0-4.1 nm. As part of this work, we calculate the efficiency of a new technique for removing ion-beam induced damage, using wet-chemical etching. Here we show that increasing the ion-dose above 3000 $\mu$C/cm$^2$ allows a higher percentage of the implantation and amorphisation damage to be removed, and leaves less than 0.5% of the gallium remaining in the surface.

We use the ion-milled dishes to form lens moulds; we double-replicate the brittle silicon mould, to create a hard wearing rubber mould. As multiple rubber moulds can be created per silicon mould the process becomes industrially scalable. A thin-film of polymer lenses is then formed from the mould.

We characterise these lenses, demonstrating 1.2-2.5x magnification and resolution of 200 nm. We demonstrate their use by imaging two biological samples, one fixed & stained, and one unlabelled in water.

Additionally, using computer simulations alongside the focused ion-beam manufacturing technique, we demonstrate a curved axicon lens structure, that forms long, non-diffracting beams of intense light. We model and experimentally analyse how the lens profile and high-to-low refractive index change forms the beam, and show that increasing the refractive index change decreases the beam width but at a loss of light transmission.
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Publications


Patents

2014 Patent application no: GB1415763.0 *Optical Device for Producing Micro-Jets*

2014 Patent application no: GB1418180.4 *Super-Resolution Lenses*

Posters

2014 *Microscience and Microscopy Congress* *Micro-Optical Manufacture for White-Light Microscopy*

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2013 *Institute of Physics Advanced Photonic Techniques in Soft-Matter and Biology* *Bespoke Manufacture of Elastomer Microlenses by Focused Ion Beam Lithography*

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2012 *University of Surrey Postgraduate Research Conference* *Manufacture of Optical Components Near the Diffraction Limit*
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<td>$\theta_c$</td>
<td>critical angle of TIR</td>
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<td>$M$</td>
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<tr>
<td>$p$ &amp; $q$</td>
<td>Distance from lens to object and image, respectively</td>
<td>m</td>
</tr>
<tr>
<td>$f$</td>
<td>focal length of a lens</td>
<td>m</td>
</tr>
<tr>
<td>$f_d$</td>
<td>focal length of a parabolic dish</td>
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<td>$T$</td>
<td>Thickness of a lens</td>
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<td>$h_o$ &amp; $h_i$</td>
<td>height of the object and image, respectively</td>
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<tr>
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<td>Lens/sphere radius</td>
<td>m</td>
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<tr>
<td>$k$</td>
<td>wavevector</td>
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<tr>
<td>$\omega$</td>
<td>angular frequency</td>
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<td>Incident Gaussian beam width</td>
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<td>Axicon angle</td>
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<td>Frequency</td>
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<td>$D_A$</td>
<td>Area Dose</td>
<td>$\mu C/cm^2$</td>
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<tr>
<td>$q$</td>
<td>Charge (in the beam)</td>
<td>C</td>
</tr>
<tr>
<td>$f_I$</td>
<td>Ion Fluence</td>
<td>cm$^{-2}$</td>
</tr>
<tr>
<td>$e$</td>
<td>Fundamental charge on an electron</td>
<td>C</td>
</tr>
<tr>
<td>$r_s$</td>
<td>Sputter ratio</td>
<td>(atoms/ion)</td>
</tr>
<tr>
<td>$D_T$</td>
<td>Total Dose (in a pattern)</td>
<td>$\mu C/cm^2$</td>
</tr>
<tr>
<td>$a$</td>
<td>Depth/Dose ratio</td>
<td>$\mu m / \mu C/cm^2$</td>
</tr>
<tr>
<td>$TIS$</td>
<td>Total Integrated Scatter</td>
<td>%</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>RMS-roughness</td>
<td>nm</td>
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Dedicated to my wife, who loves me, despite everything...

To my mother, who gave me my love of science.
To my father, who gave me my love of numbers.
To my brother, who gave me my love of computers.
To my aunt, who reminds me being crazy is fine.
And to all my friends, who are just... the weirdest.
Chapter 1

Introduction & Journal Review

1.1 Introduction

In this thesis we design, manufacture and characterise micro-optic devices for sub-diffraction viewing and forming sub-wavelength light-beams.

In 1874 Abbe first defined the *Diffraction Limit*, a physical limit to the smallest spot size to which light can be gathered, based on the wavelength of the light, $\lambda$ [1]. He described it by the following the equation:

$$d_A = \frac{\lambda}{2n \sin(\theta)} \quad (1.1)$$

Where the refractive index $n$, and the converging half-angle of the light, $\theta$, also play a role. Lord Rayleigh improved upon this formula to describe the minimum distance between two objects that can be considered resolved by a lens [2]. This set a limit to the spacing between two objects that a microscope could resolve:

$$d_R = \frac{0.61\lambda}{n \sin(\theta)} \quad (1.2)$$

When two objects are separated by a distance smaller than the *diffraction limit*, the two objects cannot be resolved. To view smaller objects, ever shorter wavelengths are required. In the visible regime, the wavelength of light is between 400-700 nm. This places the absolute minimum resolvable distance at 257 nm for deep-blue light (for a glass lens in air, $\theta \approx 70^\circ$).

The earliest microscopes that were made from a simple pair of glass lenses, working together to magnify the object of interest, could not reach this limit, due to *aberrations* of the lenses. As the lenses were often imperfectly lathed, they did not conform to the
perfect shape, creating *spherical aberration* which enlarged the focal spot. This set the fundamental limit for early optics.

Modern day optical microscopes are made of a series of ultra-high quality compound lenses, making reaching the diffraction limit a function of how well made, and well maintained the microscope is.

Surpassing the diffraction limit is of general interest to microscopists everywhere. The area has been coined *super-resolution* imaging, to denote that the resolution is beyond the Rayleigh criterion. In biology, where almost all viruses and many sub-cellular processes are below 200 nm in size, there is a huge push to view beyond the limits of light. The most common biological technique is the use of fluorescence microscopy, where small light-emitting molecules are attached to specific parts of the cell and forced to emit. A sub-wavelength imaging technique for such molecules known as STED (Stimulated Emission Depletion microscopy), recently won the 2014 Nobel prize for chemistry[3]. The downside with these techniques is that high intensity light can kill live cells, whilst fluorescent dyes can interfere with biological process that the researcher wishes to observe[4]

The simplest and most recent development in the field of super-resolution imaging is *Micro-sphere Optical Nanoscopy* (MON), first published in 2011[5]. In MON, a micron-scale optically-transparent sphere is placed directly onto the surface of a substrate and viewed through a microscope. A magnified image of surface, with sub-diffraction resolution, can then be seen within the area of the sphere. This technique has been used to view 50 nm gold features on glass under a white light microscope[5], and pushed to resolve 25 nm features when confocal microscopy was performed through the sphere[6]. The greatest downsides of this technique are the inability to easily manoeuvre the spheres onto an area of interest, as well as a lack of control over the precise optics.

In this thesis, we discuss work performed to use this effect in an improved form of Micro-scale Optical Nanoscopy. We discuss a method for manufacturing high-quality micron-scale lenses from focused ion-beam milled moulds, giving exact control over the lens profile. We also describe a manipulation setup designed to move the lenses freely with respect to the sample, that fits under a conventional microscope.

As part of this work, a replication technique for manufacturing enduring, polymer moulds from silicon master moulds is described, allowing the ion-beam manufacture of lenses to become a scalable process. Lenses made from these moulds are then characterised, showing excellent properties in terms of super-resolution optics.

We also described a structure we discovered that forms long, intense, non-diffracting beams of light. We computationally model, and experimentally verify the beam-forming properties of a set of novel micro-optic structures. We show that the structure can form
sub-wavelength beams that propagate non-diffractively for microns in length. These intense beams have potential uses in optical tweezing\cite{7}, particle counting and optical injection\cite{8}

To summarise the thesis by chapter:

In Chapter 1 we begin with a short journal review, covering the field of super-resolution imaging, highlighting *Microsphere Optical Nanoscopy*.

In Chapter 2 we introduce the underpinning theory behind several areas we discuss during this thesis. This includes an introduction to Geometric Optics, describing the key features of lenses, along with Bessel beams, an unusual non-diffracting beam of light. We continue by introducing an optical modelling software used in later chapters, known as *MEEP*, which runs *finite-difference time domain* simulations of optical geometry. The final area we introduce in this section steps away from the subject of light and optics, and moves on to manufacturing. We describe *Focused Ion-Beam Microscopy* in detail, preparing the reader for the terminology used for our manufacturing method, in Chapter 3.

In Chapter 3 we introduce the various aspects of our manufacturing method. This includes a *focused ion-beam milling* technique for creating three-dimensional geometry, used to make lens moulds. We also introduce a double-replication technique for creating a polymer-rubber copy of the delicate milled master mould, enabling larger volume lens production. During this chapter we detail a wet-chemical etching technique for removing induced ion-beam damage, which we use for reducing adhesion during the replication process. Some of the work detailed in this chapter has been published in 2014 in the journal *Micron*\cite{9}, and is subject to one patent (WO2010112827 A2), and one patent application (GB1418180.4).

In Chapter 4 we characterise our micro-lenses, and compare them to micro-sphere lenses. Looking at different diameters of lenses on Blu-ray discs, whose tracks are below the diffraction limit of white light (centred at a wavelength of 550 nm), along with *lacy carbon grid* and ion-milled chequerboard samples. We view our lenses through various filters, looking at the effect of wavelength and polarisation, and use confocal laser scanning microscopy to directly observe the path of light through the lenses and the sphere samples. During this work, the manipulation rigs, described in Chapter 3, play an essential role in manoeuvring the sample and lenses, to allow characterisation. We finish the chapter by viewing two biological samples, live yoghurt bacteria and fixed & stained yeast.

In Chapter 5 we use *MEEP* to simulate a novel micro-optical structure used to form non-diffracting beams of light. By placing two curved-troughs in close proximity,
we show how the light passing through the structure interferes, forming a beam. We describe how the beam is similar in ways to a Bessel beam, but with some differences. We describe how the properties of the beam change when the profile of the troughs is modified, as well as the refractive indices of structure and surrounding medium varied. The described work in this chapter and the next is subject to a patent application (GB1415763.0).

In Chapter 6 we experimentally verify the structure shown in Chapter 5, using optical and confocal microscopy. We confirm several of the findings from our simulations in Chapter 5, including how the refractive index alters the beam profile. Having directly observed the optical jets, we then show how our three-dimensional milling technique allows us to form curved and circular light sheets, along with fine single point beams.

To conclude, in Chapter 7 we bring together our work, highlighting the important findings, and noting where future work could be used to take our findings further.
Chapter 1. Introduction

1.2 Journal Review

A focusing lens is defined as an optical element that converges the light that passes through it to a single focal point. The property of a curved interface causing light to converge has been studied since the ancient Greeks. The earliest known lenses date back to the Assyrians, ancient Egyptians and Babylonians, with the earliest surviving piece being the Nimrud lens, discovered by A. Layard in the mid 19th century[10]. These early lenses were made from polished crystal, and lacked the optical quality we seek today. The ancient Greeks sought the secrets of optics, having noted how polished crystal, glass and water could bend the path of light. Euclid wrote the first book on geometrical optics, describing several important features that still underpin the subject to this day[11]. The Romans were known to have studied the concentration of light in glass globes filled with water[12], but the pure glass lenses we see today did not appear until hundreds of years later.

Ibn Sahl, a mathematician who lived in Baghdad in the late 10th century was the first to accurately describe what we now call Snell’s law, the law of light refraction that governs general optics, in his treatise On Burning Mirrors & Lenses[13]. Highly accurate lenses from around this period have been found in northern Europe among Viking treasure hoards. These pure glass elliptical lenses have been found to have very little spherical aberration, and appear to have been made by lathing, suggesting they were stolen from southern Europe and the middle east[14]. In this early period, before the development of the microscope, the science of biology mostly involved the study of anatomy [15] and fauna & flora[16][17].

The microscope itself was first developed in the late 16th century, although there is some debate on whether it was Hans Lippershey or Zacharias Janssen who first invented it[10]. With the invention of the microscope, the world of the very small was finally opened up for viewing. In 1655 Robert Hook published his paper first describing the structure of the cells of a cork tree, in his book of observations made using a microscope, titled Micrographia[18]. From that point forward, optical microscopy has been used to study cell structure, microbes and bacteria, along with all the small scale processes of the body. Outside of biology, optical microscopes are regularly used in almost every scientific and engineering discipline, from material science to modern day electronic engineering.

The limits of optical microscopy were first penned by Ernst Abbe in 1874, when he wrote about the diffraction limit[1]. This limit defines that the smallest size resolvable by light passing through a circular aperture, and is around one-half of the illuminating wavelength. In 1879 Lord Rayleigh updated this theory to define the Rayleigh Criterion
for the minimum resolvable distance by a microscope[2]. We discuss both their ideas on
the diffraction limit further in our theory of optics, in Chapter 2.2.7.

As we highlighted in our introduction, ever since the discovery of the diffraction limit,
scientists have been testing different methods to circumvent it: Metamaterials, Flu-
orescence microscopy, Near-field Optical Microscopy, and finally Micro-sphere Optical
Nanoscopy. We will touch on each of these techniques, before describing in detail MONs,
the field in which this thesis belongs.

1.2.1 Optical Super-Resolution

1.2.1.1 Metamaterials

One major area in super-resolution imaging is the use of meta-materials, specifically,
materials with negative permittivity and permeability, leading to an overall negative
refractive index. As Victor Veselago first theorised in 1968, light passing from a positive
to a negative refractive index material is refracted on the same side of the normal to
the material boundary as it entered, quite the opposite to the usual case [19]. Diagrams
comparing regular to negative refraction are shown in Figure 1.1 a) and b). When
looking at optical systems using such a material, a convex lens would cause light to
diverge, whilst a concave lens would gather light. The most interesting case is looking
at light from a point source entering a flat slab of material. When light from the point
first passes the air/material boundary of the slab it is gathered towards a point (rather
then diverging), as indicated in Figure 1.1 c). Upon hitting the second material/air
boundary, it is diverted to a longer focus, but still gathers to a point. This allows a flat
surface to act as a lens, with the thickness tuning the focal length.

Pendry predicted in 2000 that a metamaterial lens would gather all Fourier components
of light passing through the lens to a single focal point, without decaying [20]. Normally,
the high frequency components that carry information on small structures decay expo-
nentially within the near-field, a distance of around one-wavelength from the structure.
A negative-refracting lens allows these components to gain exponentially whilst inside
the lens medium, allowing them to survive into the far field.

Whilst metamaterials do, in theory, make a perfect lens, producing such an ideal lens
is technically difficult. The first experimental demonstration of a negative refractive
index material was in 2001, when a microwave signal was shown to negatively refract
though a 2D array of copper strips and split ring resonators [21]. In the visible spectrum,
alternating stacks of thin metal and dielectric layers were theorised to allow the formation
of a resolved sub-wavelength image [22]. This was practically demonstrated by Liu et.
Figure 1.1: Diagram of Negative Refraction, with Superlens. Figure a) demonstrates Snell’s law for a regular, positive refractive index material, whilst b) shows a negative refractive index material. The effect of a negative refractive index is to refract light on the same side of the surface normal (green dotted line) as the incident beam. Figure c) shows a negative refractive “super-lens”, an effect where all the light from a point source is gathered into a focal point within, and then outside of a flat plane of negative refractive material.

Researchers al, who used a cylindrical-shaped, alternating stack of silver and sapphire to magnify and project a near-field image into the far-field [23]. They showed a pair of 35nm wide lines, spaced 150nm apart, could be resolved under an optical microscope using their lens.

Metamaterial lenses are still at a very early stage of research. Practical demonstrations are incredibly complicated to produce, and are of little use away from example samples.
that demonstrate the theory. Whilst the field is still developing, much of the modern research in metamaterials lies in the field of cloaking, hiding an object from visible and radar wavelengths[24]. Whilst some predict that lenses will be the more useful of the two applications, cloaking is a popular line of research, as it captures the public imagination.

We now move on to the current largest field of super-resolution imaging, used exclusively in biology, Fluorescence microscopy.

1.2.1.2 Fluorescence Microscopy

The simplest form of fluorescence microscopy involves attaching a fluorescent molecule to an object of interest. By stimulating the fluorescent molecule with a light source, such as a laser, the molecule can be forced to emit. The emitting molecule can be seen as a point source, very precisely defining its location[25]. The latest developments in fluorescent microscopy involve complex illumination schemes to maximise the precision of imaging the fluorophores. The two best known are STED and STORM.

STED stands for \textit{STimulated Emission Depletion}, and works by depleting the fluorophores in a region surrounding the target area, allowing greater resolution at the target. The technique uses two lasers, an excitation, and a de-excitation beam. It was first described by Hell \\& Wichmann in 1994[3]. The excitation beam forms a spot over the target area, whilst the de-excitation beam forms a ring, or doughnut shape surrounding the area. The purpose of the de-excitation beam is to force the molecules into their ground state. The area where the spot and doughnut overlap is unable to emit, so that only the sub-wavelength sized region within the central hole of the depletion ring is excited[26]. The technique has been shown to work at video rates in live cells[27] and has been extended to scan in three-dimensions[28].

The process for STORM is significantly different. \textit{STochastic Optical Reconstruction Microscopy} (STORM) involves a serial process of photo-activating a groups of sparsely positioned fluorophores[29]. By illuminating just at the energy required for fluorescence in the molecule, a statistically small number of fluorophores activate. As long as they are spatially separated by the resolution limit ($\lambda/2$), they can be localised to a single position computationally. The final step involves photo-bleaching the activated molecules so they can no longer fluoresce, and recording their positions. The process is then repeated a number of times, building up a picture of the positions of the fluorophores. This technique has created images of complex systems down to 19±3nm, and can also be used to form three-dimensional images[30]. The drawback to this method is it is significantly slower than STED, and so live-viewing is not possible.
Both of these techniques have been shown to resolve features down to below 20 nm, and are now accepted techniques in biological research. The downside of both these techniques is the need to dye the samples, and the relatively high-intensity light used, which leads to sample damage. Precise labelling can be a difficult process, especially for minute structures. The molecules themselves are also somewhat invasive, and have been proven to interfere with the biological process being observed[4]. The ideal super-resolution technique for biological purposes would have the high speed and resolution of STED, allow three-dimensional viewing, and not require labelling of any kind.

1.2.1.3 Near-field Techniques

The next area of super-resolution optics falls under the category of near-field techniques. As we touched on earlier, the near field is a region around one wavelength from the point of scattering. The fine detail that forms the sub-resolution features is made of decaying, evanescent waves, that don’t propagate far enough to reach the microscope objective. To overcome this, Scanning Near-field Optical Microscopy (SNOM) was developed.

In SNOM, a fine glass tip is coated in a metal, with just a few nanometre window exposed at the very end (typically < 50nm)[31]. The needle is held near the surface of the sample and scanned back and forth, in a similar manner to atomic force microscopy (AFM). Light scattered from the fine-structure of the surface is then captured within the glass needle and totally-internally reflected to the sensor. This way, the evanescent waves can be captured, and an image of the fine surface built from the boustrophedonic raster of the scanned tip. As a scanning and optical technique, it can be combined with other, similar techniques, such as AFM and Raman spectroscopy[32]. The greatest drawback of SNOM is the long acquisition times, which are improving along with atomic force microscopy (AFM).

1.2.1.4 Microsphere Optical Nanoscopy

The most recent development in near field techniques is Microsphere Optical Nanoscopy, where a micro-sphere of silica or similar material is dropped directly onto a sample, and the sub-diffraction structures viewed through the optics of the sphere via a microscope. The technique has demonstrated 50 nm resolution under white light[5], and 25 nm resolution under confocal scanning[6]. Within the field, the key areas of research relate to understanding the mechanism behind the super-resolution imaging, along with overcoming the method’s limitations.
Chapter 1. Introduction

Theory papers have investigated the mechanism behind micro-optic super-resolution\cite{33}\cite{34}, often coming to the conclusion that not enough is understood about the system to deduce it yet\cite{35}. The difficulty in positioning the nanospheres has been tackled by a few papers\cite{36}\cite{37}\cite{38}\cite{39}. Others have investigated the use of a liquid medium to semi-immerse\cite{40}\cite{41}\cite{42}\cite{34} or fully-immerse\cite{43}\cite{44}\cite{45} spheres to improve contrast and magnification. Some have begun using micro-spheres within other optical\cite{6} or spectroscopic\cite{46} techniques, enhancing their spatial resolution. Micro-spheres have also been used to view biological samples, including directly viewing viruses\cite{47}\cite{48}.

As the manufacture of micro-lenses is a key aspect of this thesis, within the papers we will be looking for how being non-spherical affects the imaging properties. This is discussed in several papers we will highlight in this review \cite{49}\cite{50}\cite{34}.

The first demonstration of micro-optic super-resolution was as recently as 2009, by J Lee et. al, involving dropping small lens-like structures onto a textured surface \cite{51}. The group showed that by placing grown CHQ (calix[4]hydroquinone) micro-lens structures on sets of milled lines with pitch smaller than the diffraction limit, that the lines were magnified and could clearly be resolved. The Rayleigh criterion for white light is 321 nm (for $\lambda = 500$, NA 0.95), and in the paper they resolved 220 nm lines, at a magnification of around 1.6x.

The micro-lenses were grown via a self-assembly process, forming spherical plano-convex lenses. Their process for manufacture allowed them to disperse lenses over a number of different surfaces, to be used as micro-viewing tools.

In conventional & industrial micro-lens manufacture, lenses are formed via the reflow technique. Patterned rectangular blocks of photoresist are heated up just slightly above their glass transition temperature, allowing them to flow into a droplet shape, and act as a lens\cite{52}. By controlling the size of the original photoresist block, as well as the reflow temperature and time, the profile of the lenses can be controlled\cite{53}, however without additional intervention, only spherical lenses can be produced\cite{54}. Such a process is used in industry now to produce large arrays of micro-lenses on CCD and CMOS sensors to improve the light gathering into pixels\cite{55}.

The reflow technique was used by Verma & Sharma in 2010, who self-assembled and reflowed polystyrene droplets onto the surface of a glass coverslip. They then inverted the structure and suspended it above a textured surface, allowing them to view 500 nm strips\cite{56}. Whilst not subwavelength, the micro-lenses markedly improved the optics of the microscope, which was not able to discern the structure without the lenses.
In 2011 came the breakthrough work that ignited the current field. Wang et. al. demonstrated a similar phenomenon to J Lee’s group, although using silica (SiO$_2$) microspheres instead of lenses, for sub-wavelength viewing[5]. They looked at the magnifying properties of 1-10µm silica spheres, of refractive index $n \sim 1.5$. Figure 1.2 shows an SEM and optical image of a sphere demonstrating an 8x magnification, whilst resolving 50 nm holes, with a 50nm spacing. This is the current record for white-light optical viewing in the field.

![Figure 1.2](image)

Figure 1.2: Figure from Wang et. al. showing an anodic aluminium oxide layer with 50 nm holes and hole-spacing, imaged in an electron microscope (indicated SEM) and via a white light microscope (indicated ON), through a microsphere (position highlighted by circle). Within the micro-sphere in the optical image the 50 nm holes can be resolved, and are magnified to 400 nm, demonstrating an 8x magnification. Scale bar, 5µm . Images reproduced with permission from Wang et. al.[5]

This work launched a series of similar research, the use of micro-spheres within the near field to view diffraction-limited objects. The work undertaken in this thesis fits within this field. By producing micro-lenses on a similar scale to the micro-spheres used, we have looked at whether high quality lenses could be used as an alternative to micro-spheres.

The first theory to predict micro-spheres may work as sub-wavelength optics began in the paper by Chen & Taflove in 2004[57]. They described nano-jet emission from silica micro-spheres when modelled by the finite-difference time-domain method. Nano-jets are long (few micron), non-diffracting beams of light which have a sub-wavelength full-width at half maxima, indicating a very small spot size. Whilst they did not discus the possibility for direct super-resolved imaging, many since have indicated that the nano-sized spot of the jets may play a crucial role in micro-sphere imaging[5]. Micro-lens structures have also been shown to form nano-sized spots, suggesting the mechanism for image formation is the same[58].

Current theory predicts the lens must convert evanescent waves to propagating waves, allowing the fine-structure to be imaged in the far-field. S Lee et. al. in their 2013 paper
developed the beginning of a theory of how the evanescent waves are saved\[59\], however the exact details of the conversion function are somewhat glossed over.

When viewing samples through micro-spheres or lenses, all of the experimental papers agree that the objective microscope must be focused below the focal plane of the sample surface\[5\][49]. This indicates the image formed is a Virtual image\[51\]. In Chapter 2.2.3 we discuss the properties of a plano-convex lens, highlighting how a virtual, magnified image is formed when the object is between the lens and its focal point. Maximum magnification is reached when the object is closest to the focal point.

A number of papers have noted the change in apparent magnification caused by moving the focal plane of the objective microscope with respect to the lenses\[33\]. Theory papers have explored this phenomenon, finding that for a Gaussian incident wave, moving the micro-sphere away from the central focal spot increases its focal length\[60\], which can account for the change in magnification.

As both micro-spheres\[5\] and micro-lenses\[51\] have been shown to magnify sub-diffraction limited objects, research has been conducted into how the profile of the sphere affects the super-resolution effect. Vlad et. al. carefully reflowed polystyrene (PS) microspheres onto structured surfaces, changing the surface contact angle and reflowing the spheres to form hemispheres, then lenses\[49\].

Figure 1.3, reproduced from Vlad et. al\[49\], shows four 2.4µm diameter PS spheres in a) SEM, b) modelled data and c) the optical image through the sphere. In their images, they show that a surface contact angle of less than 90° (shown in column 2) where the lens is still largely spherical, gives the greatest magnification (3.2x), however the resolution (contrast) is poor. At a contact angle of exactly 90°, where a hemisphere sits upon a post (column 3), the greatest contrast is seen, along with a 2.2x magnification. Beyond 90° (column 4) the structure is similar to a conventional lens, with an increased field of view, but lower magnification (1.4x). The modelled data shown in row b) indicates that when transforming from a sphere to a lens the focal point is pushed into the surface which, as we indicated earlier, reduces the magnification of the image. Further modelling agrees that modification of the sphere shape & profile creates a longer focal length, without a significant increase to the width of the focal spot\[34\].

The optics of micro-spheres and micro-lenses formed by reflow or growth are all that of a spherical profile lens. In macro-scopic lenses, imperfections in the spherical profile lead to a net increase in the size of the focal spot produced by a lens. This is known as spherical aberration and is caused by the edge of the lens focusing to a slightly different point than the centre\[61\]. Guo et. al. demonstrated via Mie-scattering simulations that
Figure 1.3: Figure of Reflowed Polystyrene micro-spheres, from Vlad et. al. Figure shows the deformation and optical properties of polystyrene microspheres as they are reflowed. Moving across the columns we see different spherical profiles, achieved via reflowing. Moving down the rows shows SEM images, computer models of the focal properties, and optical microscopy images relevant to that particular spherical profile. The figure demonstrates that pushing the focal point into the sample surface reduces optical magnification, as well as indicating non-spherical shapes, including a plano-convex lens shape can perform super-resolution imaging. Scale bars are 1µm.

Reproduced from Vlad et. al.[49]

The spherical aberration of a micro-sphere is small, due to the negative spherical aberration caused by diffraction counteracting the positive spherical error of the sphere[62]. The lenses we manufacture later in this thesis are parabolic in profile, which should completely negate spherical aberration from the profile.

Semi-immersing micro-spheres have been shown to increase the image contrast and alter the magnification, by changing the sphere profile to that of a lens, as well as increasing the refractive index at the sample surface[40]. In 2013 Ye et. al. experimented with the immersed lens/surface distance, by covering a Blu-ray disc in a thin layer of SU-8 photoresist and sitting the lens-like structure on top. By increasing the thickness of the SU-8, they demonstrated a non-linear decrease in magnification, but a linear increase in field-of-view (FOV) as the thickness increased from 0-14µm.

The group published again in 2014, describing both experiment and theory which showed that the meniscus that forms around the sphere displaces the focal point to further within
the surface, modifying the magnification\[50\]. Interestingly, by using SU-8 to form the meniscus and then removing the sphere, they showed that a form of concave lens made from the meniscus alone could image small structures. Figure 1.4 combines three figures from their paper. a) shows the layout of a semi-immersed sphere on an SU-8 layer, b) shows their models, demonstrating that the addition of a surrounding meniscus acts like a lens, with or without the sphere. c) demonstrates the optics of a sphere without a meniscus, one with the meniscus, and finally the meniscus alone.

The difficulty with semi-immersion is that, for the conventional liquids used (water, ethanol), due to the small volumes of liquid, evaporation causes the lens profile to change during imaging. It has also been noted that if viewing a biological sample with this technique the sample would dry out and die very quickly. The lenses we characterise in Chapter 4 don’t have such a drawback. During the chapter, we demonstrate imaging samples in aqua. The thin film on which our lenses sit acts as a cover slip, giving the additional benefit of reducing evaporation from the sample.

Whilst semi-immersion has been shown to increase contrast and magnification using regular glass or polymer spheres of \(n \approx 1.5\), others have tried total immersion imaging using higher-index spheres. This increases the refractive index of the transmission medium all the way to the microscope objective. Darafsheh et. al. first demonstrated that a higher refractive index sphere material, such as barium titanate (1.9 < \(n\) < 2.1), can form super-resolution images when fully immersed in water or oil\[43\].

Theoretical simulations predicted that the difference in refractive index between sphere and surroundings controls the focal spot properties, with an effective sphere index of 1.5 – 1.75 being optimal\[62\]. If the effective index of the sphere is greater than 2, the focal point is inside the sphere and no image can be formed\[59\].

Further experimentation using water, glycerol(40\%) & oil has demonstrated that increasing the refractive index of the medium compared with the sphere, reduces the magnification and contrast of the image formed \[44\], as expected from the modification of the focal length.

To position the micro-spheres with respect to the sample, two techniques are commonly used: large numbers of spheres are dispersed onto a surface and allowed to self assemble, or micro-positioning systems are used. The first method has little control over the actual position, and so significant numbers of spheres are used to attain a good image\[5\]. The second method has been used in several papers, but is tricky to perform in-situ under the microscope\[43\].

The earliest attempt at in-situ locomotion was demonstrated by a Shu-Ying et. al.\[36\] in which they fuse a single sphere to a glass micro-pipette, allowing manipulation of
Figure 1.4: Experimental configuration for understanding the meniscus in partly immersed lenses, from Yao et al. Figure combines three figures, including the experimental imaging setup, showing a micro-sphere sitting upon an SU-8 layer, surrounded by a meniscus. Computational simulations demonstrate how the meniscus pushes the focal point of the sphere further into the surface, making it act like a lens. The three optical images show images taken through a sphere with no meniscus (a), with a meniscus (b), and a meniscus with no micro-sphere (c). The pattern can only be resolved in the presence of the meniscus. Scale bar 2 \( \mu \text{m} \). Reproduced from Yao et al. [50]

the sphere. As this work was printed only in Chinese, it went largely overlooked. A similar work was independently performed by Krivitsky et al. [38]. They also attached the sphere to a micropipette, and then manipulated the pipette by a translation stage, shown in Figure 1.5 a). Other papers have tried connecting micro-optics to SNOM cantilevers for easy manipulation [39].

A significantly more complex method of locomotion was introduced by Banas et al. [41].
In their paper they manufactured a three-point holding device, in which the sphere sat centrally between a triangle of support rods, as shown in Figure 1.5 b). At the end of each rod is a knob, designed to be manipulated by an optical trap. By manipulating the three knobs via optical tweezing in tandem, the sphere can be moved in x, y and z.

Figure 1.5: Figure a) Setup used by Krivitsky for manipulation of micro-sphere. Scale bar in inset 10µm. b) schematic for Banas locomotion structure, showing a microsphere support structure manipulated by optical trapping

Whilst the original paper by Wang et. al. still has the record in the field for the smallest resolved distance by conventional white-light microscopy[5], a later paper published by the same group observed 25 nm resolution by performing confocal laser scanning microscopy through a micro-sphere[6]. The $\lambda/16$ gap between gold nanodots was clearly resolved, demonstrating resolution in the region of SNOM or STED microscopies.

Micro-spheres have already started to be used in conjunction with other optical techniques, to improve their resolution. Upputuri et. al. demonstrated coherent anti-Stokes Raman spectroscopy through a micro-sphere. In the paper they mapped an image of the polycarbonate ridges in a Blu-ray disc by selecting for the related peaks. This demonstrated Raman imaging of sub-diffraction limited objects, which normally requires a mapping technique, such as tip-enhanced Raman spectroscopy.

The final two papers to highlight are those of Yang [48] & Li [47], who have both used micro-spheres to image biological samples. Yang et. al. applied the full immersion technique discussed earlier to samples of fluorescently-stained centrioles, mitochondria & chromosomes along with mouse liver cells. They place high index (n=1.92) barium titanate micro-spheres onto samples, and then immerse them in water or oil. Through the micro-spheres they resolved 100 nm features within the cells, whilst demonstrating a 5.4x magnification gain.

Li et. al. used micro-spheres to perform label-free imaging of 75 nm adenoviruses. As we discussed in our fluorescence imaging section, fluorescent labelling of biological samples is the most common way of achieving sub-diffraction limited imaging. The adenovirus was
imaged via a white-light microscope using fully immersed micro-spheres, demonstrating a 2.4x magnification. The ability to view viruses under white light demonstrates the power of the technique. The possibility of live viewing the processes of viruses attacking cells would be a huge step forward to biological research.

1.2.2 Review Conclusion

In this review we have looked at work involved in super-resolution imaging by various means. We discussed a metamaterial approach, fluorescence microscopy, SNOM & micro-sphere/lens optical nanoscopy. Comparing these techniques we can talk about the similarities and differences. Metamaterial lenses, SNOM & MONs all require optics within the near-field, whilst fluorescence microscopy is a far-field only technique. However, the technique requires the presence of fluorescent dyes, which alter the natural processes within biological samples.

Metamaterials hold great promise, with a theoretically predicted ability to project all of the near-field evanescent waves that are usually lost, into the far field. In our review we highlighted the well developed theory, but the drawback to the technique is the difficulty in manufacturing a real lens. Silver hyper-lenses show significant progress, but the technique is far from lab-ready.

Fluorescence techniques, involving labelling biological samples with dyes, are the current norm in biological microscopy. In STED microscopy the molecules surrounding the target are carefully de-excited, leading to sub-diffraction limit spot sizes. As mentioned, the inventors won the Nobel prize in chemistry in 2014 for their work. The technique is becoming more prevalent, with off-the-shelf STED systems now available. STED, along with the alternative technique STORM, have both shown 20 nm resolution, and hence are incredibly useful tools. However, the use of fluorescent molecules fundamentally alters the biological process viewed.

Due to SNOM’s small interaction size, giving very high (nanometre) resolution, along with it’s ability to integrate with other techniques, it makes for an excellent tool for the lab. However, the long acquisition times preclude imaging live processes with the technique.

In micro optical nanoscopy, we discussed how glass & polymer micro-spheres & lenses are used to view diffraction-limited structures. We highlighted how the technique has demonstrated 8x optical gain under white light, and resolved 25 nm structures, when combined with a confocal microscope.
The technique has been compared to Solid Immersion Lenses, which are few millimetre plano-convex lenses designed sit upon a sample and improve the resolution. These work by increasing the refractive index at the sample surface, in a similar way to liquid immersion techniques, which reduces the size of the diffraction limit for the wavelength. These are still subject to the diffraction limit, but high-index SILs can see much finer structure than conventional optics alone.

Comparing this to MONs spheres and lenses, current theory has yet to define exactly why they can see so far below the diffraction limit (down to $\lambda/16$ under confocal), or what the minimum resolution will be. It is agreed that an immersion-type saving of the evanescent waves is part of the process, but it is unknown how the evanescent waves are converted into propagating waves.

In forming a sub-wavelength virtual image, the two key factors are needed: a small spot size, and a short focal length, just below the surface of the object. This maximises magnification, whilst allowing the highest quality images to form. Comparing spheres to lenses, spheres form sub-wavelength spots with extremely short focal lengths, exactly what is needed for image formation. Lenses have been demonstrated to also produce a sub-wavelength spot, however their focal length is significantly longer (can be an order of magnitude longer than a sphere). This leads them, on average to produce a much lower magnification, around 2x compared with the 3-5x seen in spheres.

Such lenses have been manufactured by a number of methods, including: self assembly, polymer reflow & semi-immersing spheres to create short lived lens structures. In general, a plano-convex lens structure appears to have increased contrast over a sphere, as demonstrated by Vlad et. al. among others. The higher contrast likely comes from the evanescent (decaying) wave from the surface travelling a shorter distance in-air, before entering the lens. This is a significant advantage to micro-lenses, and the only key development they require is a method of improving the magnification. We discuss in Chapter 4 how our relatively shallow lenses are often limited in their magnification by the focusing properties, and how our method may be extended to produce deeper, parabolic lenses.

Currently, MONs has only just begun to find uses in other optical techniques. We noted how it has been used in confocal microscopy, improving the resolution two-fold over white light. It has been used for super-resolution Raman spectroscopy, which hints that other optical spectroscopies could also benefit from near-field micro-optics. Its use in biology has already shown great promise, resolving 75 nm adenoviruses. Combining MONs into other biological techniques may provide further improvements to what biologists can view. Later in this thesis, in Chapter 4.7 we examine two biological samples, including a fixed, stained sample of yeast, and a living coccus chain in water.
During this journal review we have highlighted the strengths and weaknesses of micro-optical nanoscopy, highlighting how it can magnify detail usually lost to microscopy into the far field. We have described its limitations, including the positioning of the optics with respect to the sample & the control over the profile of the optical elements themselves. During this thesis we describe a method to manufacture high-quality micro-lenses for use in super-resolution imaging, looking to overcoming these drawbacks.
Chapter 2

Theory of Optics & Ion Beams

Summary
This chapter sets up the theory and tools used throughout the rest of this thesis. We start with an introduction to microscopy, including a discussion of the resolution limit, needed when we characterise our micro-lenses in Chapter 4. Then we move the discussion into Bessel beams, non-diffracting beams of light, relevant to chapters 5 & 6, in which we look at a curved profile micro-optic beam former. Then we introduce MEEP, an open-source computational modelling software for simulating the propagation of light, which we use in chapters 4, 5 & 6 to examine how light travels through our optical structures. Finally we discuss Focused Ion-Beam (FIB) microscopy, including the control variables and incident ion mechanics, which will be crucial to our discussion in Chapter 3, where we detail our manufacturing method alongside an ion-beam damage removal technique.

Acknowledgements: We would like to thank Dr. Marian Florescu for his help in the initial configuration of MEEP, including the correct coding for the boundary conditions.
2.1 Introduction

During the course of this thesis we describe a number of micro-optical devices, along with the techniques to manufacture them. In Chapter 4 we characterise PMMA micro-lenses, suspended upon on a thin film, which we use to view beyond the diffraction limit, to image the tracks on a Blu-ray disc not normally visible under white light illumination. Along with the tracks, we also view milled nano-structures, lacy-carbon grids and two biological samples. During the course of this discussion, we study the magnification and contrast of the lenses, to identify if a feature has been resolved. We begin this chapter by describing the conventional, geometric optics of the types of lenses we use, including plano-convex and ball-lenses. Whilst the assumptions that govern geometric optics fail for small lenses, due to diffraction from the lens aperture, the over-arching principles are the same. We then describe how resolving small features through a microscope is limited by the wavelength of the illuminating light, known as the Diffraction limit. Moving away from traditional lenses, we then discuss Bessel beams. In chapters 5 & 6 we describe a curved nano-jet forming structure that produces light jets of similar profile to a Bessel beam. To finish our section on micro-optics we detail the properties that make up a Bessel beam, such as that it is non-diffracting, self-healing and created by interference. We note how axicon lenses work, which are used to form an approximation of a Bessel beam. We also note the shape of the propagating and tangential profile of real Bessel-beams, giving us a comparison point later with our nano-jets, as well as detailing some of the mathematics of the profile of the Bessel beam.

In the next section we detail an optical simulation software package, known as MEEP; a finite-difference time-domain (FDTD) simulator that we use to predict the path of light through our structures. We use MEEP in chapters 4 & 5 to simulate our lenses and nano-jet forming structures. Chapter 5 specifically, is entirely based on MEEP simulations of jet formers. In this chapter, we describe how MEEP works, including the control file, used to describe the geometry, material, source and output. The details of the modelling process are helpful in understanding the limitations of the models in later chapters.

In Chapter 3 we detail the manufacturing process for our micro-optics, including ion-beam milling, chemical etching, and replication processes needed to create lenses. To finish this chapter, we move away from the field of optics, and describe the underpinning theory of focused ion-beam microscopy. It is important to fully grasp how the control variables defined when milling affect the final pattern. We begin by describing how the ion-beam itself is formed, allowing us to highlight the important control variables. In Chapter 3 this will let us jump straight into the discussion of the manufacture technique.
itself. We then move to the incident ion mechanics. Here we describe the damage intro-
duced to the sample by ion-beam milling, along with conventional methods of healing
this damage. For us, this damage has caused polymer replicas to adhere to the milled
silicon moulds, ruining the replica when delaminating. This underlines the discussion
we have in Chapter 3, about a wet-chemical etching method of removing ion-induced
damage.

Now, we begin with the theory of optics.

2.2 Theory of Lenses & Microscopy

The earliest traceable optical microscopes were invented in the 17th century, possibly
made by Hans Lippershey or Zacharias Janssen [10]. These early microscopes were
made from a pair of shaped glass lenses that magnified the small structure of samples.
The invention of the microscope allowed humans to see cells and bacteria for the first
time, unlocking a whole new area of biological science. Modern microscopes are made
of a series of compound lenses. The simplest microscopes can be made with only two
lenses, but additional lenses, apertures, filters and polarisers can be added to improve
the image quality, and allow different aspects of the samples to be imaged.

As we indicated in our literature review, in Chapter 1, there is a known limit to the
resolution of an optical microscope, related to the wavelength of the light imaged. Whilst
the Diffraction Limit is a well understood theory, there are several suggested limits used
in relation to microscopy. Abbe was the first to suggest the resolving limit of a simple
aperture in 1873, before Rayleigh improved the idea to describe the limit of a single
lens, and then a pair of lenses. Some researchers claimed that they could resolve objects
spaced closer than this resolution limit with a microscope, and so Sparrow[63] defined
his resolution theory, setting the absolute minimum that was conventionally resolvable.

Whilst the lens qualities we discuss here are useful to our later discussion, it is worth
noting the assumptions many of the equations follow. As they apply to macroscopic
lenses they ignore small scale diffractive effects from the lens aperture. In our later
micro-lens discussion, in Chapter 4, the lenses are close to the wavelength in size (1-
10\(\mu\)m diameter, for wavelengths of 300-700 nm), allowing diffraction to play a role. In
our literature review, Chapter 1.2.1.4, we noted how the path of light travelling through
a microsphere caused curvature in the wavefront of the light as it collected to a focal
spot, as a result of diffraction from the microsphere aperture[49]. We see a similar effect
in our modelling demonstrated in Chapter 4.
Lenses work by redirecting light transmitting through them to a single, convergent point. Whilst the Greek philosophers were the first to attempt to deduce how the angle of light changed as it moved from one medium to another, Ibn Sahl was the first to correctly describe the phenomenon, in 984 [13]. In his work, titled *On Burning Mirrors and Lenses*, he described mathematically how to form convergent lenses and parabolic dishes. It is now well understood that the basis of how lenses work is the refraction of light, as described by Snell’s law, Equation 2.1:

\[ n_1 \sin(\theta_1) = n_2 \sin(\theta_2) \]  

This describes how the angle of light with respect to the surface normal, \( \theta \), changes when it moves from a medium of refractive index \( n_1 \), into an index of \( n_2 \). When \( n_2 \) is the higher index, the angle \( \theta_2 \) is smaller than \( \theta_1 \), whilst a larger value of \( n_1 \) leads to the opposite situation. By modifying the profile of the interface between the media, it is possible to redirect all light passing through it to converge on a single point. This, in essence, is what a lens does.

It is worth noting that, when \( n_1 \) is the larger of the two indices, there are angles which cannot pass through the interface, due to the light being refracted to an angle greater than 90°. The incident angle that refracts to exactly 90° is known as the critical-angle, \( \theta_c \), and can be calculated from Equation 2.2:

\[ \theta_c = \sin\left(\frac{n_2}{n_1}\right) \]  

For many plastics or glass, where \( n \approx 1.5 \), this puts the critical angle, in air, at 42°. Light attempting to pass at higher angles than this is reflected. This property is used in fibre-optic cables, where due to the small radius all light incident on the side walls is beyond the critical angle, and so is totally internally-reflected.

Whilst this describes what a lens is, we still need to define the properties of a lens to discuss its use in later chapters.

### 2.2.1 Lens Properties

Figure 2.1 a) shows the properties of a simple bi-convex lens. This includes the focal length, \( f \), the lens-object distance, \( p \), and the lens-image distance, \( q \). The distance at which the image is formed is related to the focal length by the following formula:

\[ \frac{1}{f} = \frac{1}{p} + \frac{1}{q} \]  

(2.3)
This means that, as the object moves closer to the focal point, the imaging plane moves further from the lens. Alongside this, the height of the image, $h_i$, increases with respect to the height of the object, $h_o$, giving an overall magnifying effect. The magnification of the lens, $M$, is calculated as the ratio of the two:

$$M = \frac{h_i}{h_o} \tag{2.4}$$

When we discuss magnification through our lenses in Chapter 4, we relate the known size of an object (often measured in an electron microscope), to the size of that object as seen through one of our lenses under an optical microscope setup.

Figure 2.1: Properties of a bi-convex lens, including, the lens thickness, $T$, the focal points, $F$, the distance from object to lens, $p$, and lens to image, $q$. We also show the object height, $h_o$, and the image height, $h_i$, from which the magnification can be measured. As the object is at twice the focal length, $2F$, the image height is the same as the object height, giving no magnification gain.

Along with the magnification, another important tool when discussing both lenses and reflective dishes is the f-number or $f\#$. This number gives an indication of the optical element’s gathering power, and allows lenses and dishes of different size and focal length to be compared. A smaller $f\#$ indicates a higher gathering angle, which leads to more light being collected. It is normally calculated from the focal length, $f$, with the diameter of the component, $D$:

$$f\# = \frac{f}{D} \tag{2.5}$$

Moving on, we need to define the properties of several important optical elements we use throughout this thesis. These include: a reflective parabolic dish, a plano-convex lens, of both spherical and parabolic profile, a plano-concave lens, and a ball lens (or sphere).
2.2.2 Parabolic reflector focal properties

The focusing effect of a parabolic dish is well understood, and made use of in many disciplines including for signal collection in radio communications, and light and image magnification, in astronomy. The definition of a parabola is a line for which all normals converge at a single, focal point. To calculate the focal length, \( f_d \), of a parabolic reflector, we use the the diameter, \( D \), and depth, \( c \), of the dish.

\[
f_d = \frac{D^2}{16c}
\]

(2.6)

We use this equation heavily when calculating the properties of a dish to mill, as explained in Chapter 3. When making a mould for a lens, the dish focal properties are a useful estimation of the final lens properties. However, as a purely parabolic profile is rarely used when making lenses, we have had to derive the equation for the focal length of a parabolic lens ourselves, seen in 2.2.4. Before we go through the derivation, we quickly introduce the lens equation, and describe a plano-spherical lens, a much simpler geometry.

2.2.3 The Plano-Convex lens

To describe the focusing properties of any lens, the simplest place to start is the lens-makers equation[61]. This describes the lenses focusing power, \( P \), which is the inverse of the focal length, \( f \), with respect to the radius of curvature of both sides of the lens. In a standard lens, \( R_1 \), the radius of curvature nearest the light source, and \( R_2 \) is the radius of curvature for the interface furthest from the light source, as indicated in Figure 2.2. The other quantities needed to calculate the lens power are the refractive index of the lens, \( n \), and the lens thickness, \( T \):

\[
P = \frac{1}{f} = (n - 1) \left[ \frac{1}{R_1} - \frac{1}{R_2} + \frac{(n - 1)T}{nR_1R_2} \right]
\]

(2.7)

The standard adopted by the equation is that, when \( R_1 \) is positive, the first curvature is convex, and when negative, it is concave. When \( R_2 \) is positive, the second surface is concave, and when negative the surface is convex. The equation makes the assumption the lens is in air, and as \( n_a \approx 1 \), it is therefore not present. It is important to highlight the approximation made by this equation. When deriving the formula, they use the small angle approximation \( \sin(\theta) = \theta, \cos(\theta) = 1 \), which does not hold true for micro-lenses[64]. This, along with the lack of diffraction within the equation, are what stop geometric optics from working on small-scale lenses.
For a Plano-Convex lens, $R_1$ is considered infinite, whilst $R_2$ is finite and negative. This removes the factor of $1/R_1$ from the formula, simplifying down to Equation 2.8 for the lenses focal length:

$$f = \frac{R_2}{(n-1)}$$  \hspace{1cm} (2.8)

For a simple polymer spherical-profile lens, such as those reflowed by Vlad et. al. in our super-resolution literature review[49], 1.2.1.4, this would put a $3\mu m$ diameter hemispherical lenses focal length at around $3\mu m$ from the optical centre, $1.5\mu m$ from the lens surface.

![Figure 2.2: Diagram of the Lensmakers Equation Properties. For a light source at focal point $f$, the radius of curvature of the nearer lens side is labelled $R_1$, indicated in green, whilst the radius of curvature of the far lens side is labelled $R_2$, indicated in pink. Due to convention, for a biconvex lens, such as this, $R_1$ is positive, whilst $R_2$ is negative.](image)

When discussing a plano-convex lens, the distance between the object and optical centre of the lens in relation to the focal length is very important. Figure 2.3 shows the setup for a sample behind a plano-concave lens, for three different cases.

In case a), when the distance from the object to the lenses optical centre is twice the focal length, $p = 2f$, the height of the object and image are the same, $h_o = h_i$. The image itself is a real image, meaning viewing it directly it would appear inverted.

In case b) where the object has moved closer to the focal point, such that $2f > p > f$ the image height increases relative to the object height providing magnification, $h_o < h_i$, whilst the image is still real.

At the focal point, the light rays passing through the lens never meet, making the lens-image distance infinite. When the object is closer than the focal length such as in case c), $p < f$, the rays form a virtual image behind the lens. This is still magnified, $(h_o < h_i)$ but decreases in magnification moving closer to the lens.
The concept of a virtual image from a plano-concave lens, when the object is closer than the focal length, is very important with regard to our micro-lenses, as they are designed to work in the near-field (less than 1 wavelength from the sample). In Chapter 4 we see exactly what was described in our literature review, 1.2.1.4. Placing a micro-optical element on top of a sample and focusing the microscope below the sample surface, we see a virtual, magnified image, containing detail beyond the diffraction limit.

**Figure 2.3:** Demonstrates the magnification gain through a plano-convex lens with the object at three positions, relative to the focal point, F. In the top image, the object is at 2F and there is no magnification gain. In the middle, a *half-sized* object is between 2F and F, and is magnified up to twice its size. In the bottom image, the object is closer than the focal length F, and so a *Virtual image plane* is found on the same side of the lens.

Having described the properties of a standard plano-concave lens, we next need to discuss the properties of our parabolic lenses. Our lenses are made parabolic as a method of removing *spherical* aberration, whilst simplifying manufacture (described in Chapter 3.2.1). However, as most aspheric lenses are a modified spherical lens in profile, a mathematical treatment for purely parabolic lenses is rare. In this next section we will derive, from the lensmakers formula, the focal length of a plano-parabolic lens.
2.2.4 Plano-Convex Parabolic lens

Whilst the focusing properties of a parabolic dish are well understood, parabolic lenses have had very little treatment with respect to understanding their focal properties. Our aim here is to derive the focal length of a plano-parabolic lens in respect to the focal length of an identical parabolic reflector, \(f_d\).

Having already defined the focal length of a dish relative to its diameter and depth, in Equation 2.6, we next need the plano-convex lensmakers equation, Equation 2.8. This is defined in terms of the radius of curvature, \(R_2\), of the surface, necessitating we convert from radius of curvature into another coordinate system in which we can define a parabola. The following equation gives the radius of curvature for a cartesian equation[65]:

\[
R = \frac{(1 + (\frac{dy}{dx})^2)^{\frac{3}{2}}}{|\frac{d^2y}{dx^2}|} \quad (2.9)
\]

In this equation, the x-axis is radially outwards from the optical centre of the parabolic profile. In the example above, Figure 2.3 this would put the axis vertical, whilst the y-axis would be horizontal, along the light propagation axis.

This means that to find the radius of curvature, we need the cartesian equation for a parabolic curve, which is as follows:

\[
y = \frac{x^2}{4f_d} \quad (2.10)
\]

where \(f_d\) is the focal length of the parabolic dish, discussed in Section 2.2.2

Taking the first and second derivatives of Equation 2.10 gives us equations 2.11 & 2.12:

\[
\frac{dy}{dx} = \frac{x}{2f_d} \quad (2.11) \quad \frac{d^2y}{dx^2} = \frac{1}{2f_d} \quad (2.12)
\]

Placing these back into Equation 2.9 we get the radius of curvature of a parabolic lens:

\[
R = \frac{(1 + (\frac{x}{2f_d})^2)^{\frac{3}{2}}}{|\frac{1}{2f_d}|} = |2f_d|(1 + (\frac{x}{2f_d})^2)^{\frac{3}{2}} \quad (2.13)
\]

Which, when expanded and simplified leaves us with:

\[
R = |2f_d|\sqrt{1 + \frac{3x^2}{4f_d^2} + \frac{3x^4}{16f_d^4} + \frac{x^6}{64f_d^6}} \quad (2.14)
\]

When we put Equation 2.14 into Equation 2.8 we get the lens focus in terms of the dish focus:

\[
f = \frac{|2f_d|}{(n - 1)}\sqrt{1 + \frac{3x^2}{4f_d^2} + \frac{3x^4}{16f_d^4} + \frac{x^6}{64f_d^6}} \quad (2.15)
\]
In real terms, this means that the edges of a plano-parabolic lens focus to a different length than the centre of the lens, spreading spot and increasing the depth of field of the lens. However, for a small lens we can approximate that \( f_d \gg x \), giving us Equation 2.16, an estimate of the focal length.

\[
f = \frac{|2f_d|}{(n - 1)}
\] (2.16)

This final equation states that, for a standard glass or polymer lens, with a refractive index of around \( n = 1.5 \), the focal length of the lens is around four times that of an equivalent reflective dish. For our micro-lenses, for which we often aim for an \( f\# \) of the reflective mould of 0.25, this would put the focal length at a similar length to the lens diameter, \( f\# = 1 \). In Chapter 4 we have the simulated light focusing of lenses of varying diameter and \( f\# \), to test whether the assumptions of this derivation hold true.

### 2.2.5 Plano-Concave Parabolic lens focal properties

In chapters 5 & 6 we discuss concave Bessel beam formers. Whilst we discuss the optics of a Bessel beam further on, in Section 2.2.10, it is worth quickly discussing the focusing properties of a plano-concave lens, which in some respects is similar to what we use.

When discussing a plano-concave lens, the only difference from a plano-convex lens of identical radius of curved surface of the lens is that of the sign. \( R_1 \) is still considered infinite, but now \( R_2 \) is positive, which has the effect of reversing the sign in Equation 2.16:

\[
f = -\frac{|2f_d|}{(n - 1)}
\] (2.17)

As \( f_d \) is always positive, this gives a negative value of the focal length, placing the focal point before the ray of light meets the curvature. This indicates that the light source reaches the focal point before reaching the curved surface, meaning that the lens is divergent. This is useful for calculating the lenses \( f\# \) which, being negative, shows the ability of the lens to diverge the light.

### 2.2.6 Ball Lenses

Super-resolution imaging through micro-optic spheres is reliant on the optical focusing ability of a sphere. As our lenses are in direct comparison with such spheres, we need to understand those focusing properties.

Returning to the lensmakers equation, eq. 2.7, we see that, for a perfectly spherical lens, the radius of curvature of both sides of the lens are the radius of the sphere, \( r \),
whilst the lens thickness, $T$, is twice the radius, $2r$. With the sign convention, it means $r = R_1 = -R_2$. Placing this into Equation 2.7 and simplifying gives us:

$$f = \frac{nr}{2(n - 1)}$$

(2.18)

For glass, where $n \approx 1.5$, this puts the focal length approximately half the radius away from the edge of the sphere. For a refractive index of 2, the focal length is the same as the radius, placing the focal point on the sphere surface.

Having described how lenses work under geometric optics, including how they magnify, the next step is to move on to the topic of resolution. We touched on the resolution limit earlier, how it describes the smallest object visible by a given wavelength, but now we will add in some mathematical detail.

### 2.2.7 Resolution limit

In optical microscopy, it has been known for over 100 years that there is a limit to the size of objects that can be seen with light, related to the wavelength of illuminating light. This limit is known as the **diffraction limit**. As we touched on earlier in this chapter, the first limit, penned by Abbe, applied to circular apertures, and was extended by Rayleigh to cover lenses. Later, Sparrow noted a lower limit, which took light to the edges of what was possible. The description of the three limits hinges on the idea of an **Airy disk**, which described the ideal focused spot of a lens with a circular aperture.

When light passes through a circular aperture, including the aperture of a lens, it diffracts. This diffracted light interferes with itself to create a constructive and destructive interference. When projected onto a 2D screen, the pattern created is known as an Airy disk. At the lenses’ focal length, the maximum intensity is found within the central spot. Figure 2.4 shows a lens with its Airy pattern below it, in two dimensions. The green waves show out-of phase waves creating a minima, whilst the red waves are in phase, forming a maxima. Indicated in blue is $\theta$, the half-angle of the lens.

The size of the Airy disk is usually defined as being the distance between the two minima seen either side of the central peak, which is often used to help define the resolution limit of a lens. This central maxima is usually referred to as the **zeroth-order** diffraction spot, whilst the high intensity rings surrounding it are referred to as the first, second, third and $n^{th}$ order, moving outwards from the centre. A cross-section through the Airy pattern is similar in form to a **zeroth order Bessel function of the First Kind**, with a drop in peak intensity as you move outwards radially from the central peak.
Figure 2.4: A lens illuminated by a single source forming an Airy disk. The red waves show the path of two constructively interfering light rays, forming a maxima, whilst the green path shows destructive interference of two rays. The Airy pattern itself has a Bessel function-like profile, with a central peak, describing the lenses focal point, surrounded by decreasingly intense peaks.

Abbe Limit & numerical aperture

In Chapter 1 we touched on the work of Ernst Abbe, who first described the minimum resolution of an aperture, due to the diffraction of light. Abbe’s resolution criterion uses the concept of the numerical aperture (NA) a term that describes the light-gathering ability of a lens. It links the refractive index of the lens to the gathering half-angle of light by the lens, θ, (as shown in Figure 2.4.a). It is described by Equation 2.19:

\[
NA = n \sin(\theta) \tag{2.19}
\]

This means that for a given-size lens, a shorter focal length gives a larger collection angle, and a higher numerical aperture. \( n \) is the refractive index of the medium in which the lens sits, usually air (\( n = 1.000277 \approx 1 \)), or an optical fluid, such as silicone-oil (\( n \leq 1.4 \)) or water (\( n = 1.333 \)). As \( \sin(\theta) \) can never exceed 1, \( n \) dominates as the limiting factor when maximising the numerical aperture (it will become apparent as to
why you want to do this soon). In real terms, lenses have numerical apertures up to 0.95, for use in air, or 1.4 for immersion lenses.

Abbe’s final equation for the diffraction limit of a lens is a function of the numerical aperture of the lens, and the wavelength of the illuminating light. In this case \( d_A \) is the minimum radius of the spot created by a wave converging under these conditions:[1]:

\[
d_A = \frac{\lambda}{2NA}
\]

In air, this puts the minimum resolvable distance between two features (\( d \)) at around half the illuminating wavelength (for \( NA \approx 1 \)). For an object viewed under green light (500 nm wavelength) in air (maximum \( NA \) of 0.95), this estimates the minimum resolvable feature size as 263 nm.

\[\text{Figure 2.5: Images show the Airy profile overlap of the a) Abbe Limit, b) Rayleigh criterion and c) Sparrow Limit. The black and blue lines are the individual Airy patterns from two, close, sources. The red line shows the combination of the two. In ability to determine if two sources are resolved is based on the dip between the peaks in the red. The Rayleigh criterion has the largest dip, whilst the Sparrow limit has none at all.}\]

**Rayleigh Criterion**

Abbe’s limit describes the minimum spot-size achievable by an aperture, but in microscopy, it is more accurate to describe the limit to which two objects can be spatially resolved. Rayleigh described the more commonly used resolution limit of a single lens as[2]:

\[
d_R = \frac{0.61\lambda}{NA}
\]

This comes from the concept of two closely positioned point sources of light, each of which can separately be thought of as having its own Airy pattern. Figure 2.5 shows a pair of Airy patterns overlapping at the Rayleigh limit.
When two point sources are positioned under a lens, such that the zeroth-order maxima of one source’s Airy disk is at the same position as the first minima of the other source, the Rayleigh criterion is reached. At this point the dip in intensity between the two peaks is usually between 20-30% of the total intensity.

The Rayleigh criterion sets the minimum resolved distance at very slightly higher than the Abbe criterion, at around 321 nm for green (500 nm) light in air.

When considering the Airy pattern of a point source in three dimensions, the total pattern is known as a point spread function (PSF), and gives the x, y and z (optical axis) size of the focal spot. The Rayleigh criterion can be thought of as reached when the distance between the PSF of two sources reaches zero.

### Sparrow Limit

The Sparrow resolution limit, whilst most commonly used in telescopes, is still applicable to microscopes, especially in techniques using fluorescent molecules. In astrophysics, astronomers look at stars, which optically can be considered as point sources, due to their great distance away. Whilst the Rayleigh criterion can be used to describe the resolution limit of telescopes, as well as for microscopes, many astronomers claimed that they could still resolve two stars at distances below this limit. The Sparrow limit is defined as the point at which there is no visible dip in intensity between two point sources [63], as shown in Figure 2.5 c). Equation 2.22 describes the limit at which this is reached:

\[
\frac{d_S}{\lambda} = \frac{0.47}{N_A}
\]

This gives a value just slightly smaller resolvable distance than the Abbe limit. Green light (500 nm) in air has a minimum resolution of 247 nm.

Whilst this limit is not as widely used in microscopy as Rayleigh’s limit, it is applicable to fluorescence microscopy, as the fluorescent molecules act like point sources.

For all three of these resolution limits, the smallest resolvable distance under green light is between 247-321 nm. Later, in Chapter 4 we will be using Blu-ray discs as an imaging standard, as the pitch of the ridges on a Blu-ray disc are of the order of 320 nm, right at Rayleigh’s resolution limit, whilst the individual fine features are between 100-200 nm.

### 2.2.8 Defining Contrast

When looking at whether an object is resolved, it is important to discuss magnification, and contrast, \( V \), which indicates the ability to pick out an object from the background
illumination. Looking back to Rayleigh’s and Abbe’s resolution criteria, the size of the dip in intensity between two Airy patterns is critical to whether the features are distinct enough to be resolved. We define the contrast in terms of the maximum intensity $I_{\text{max}}$ and the minimum intensity $I_{\text{min}}$. In the Airy disc example, Figure 2.5, these correspond to the peaks (maxima), and the dip between Airy disks (minima). The diffraction limit merely states that, when this dip is below a certain value, the contrast between two object is not enough to resolve them.

To calculate $V$ we use Equation 2.23:

$$V = \frac{I_{\text{max}} - I_{\text{min}}}{I_{\text{max}} + I_{\text{min}}}$$  \hspace{1cm} (2.23)

In Chapter 4 we discuss images taken through our plano-parabolic lenses, and how the lenses affect the contrast and magnification of the image using this equation, and Equation 2.4.

Having defined the limit of resolution, along with the properties of the lenses that we will be discussing throughout this thesis, it is interesting to discuss how to overcome these limits. All three limits defined are based on certain assumptions. These include conventional optical geometry, the uniformity of the excited light, and that only linear (zeroeth order) terms of the light’s electric field are looked at.

Overcoming the limit can be achieved by several known methods, each discussed in our literature review, Chapter 1.2.1, including: Metamaterial lenses which, via negative refraction, gather all components of the wavepacket; Structuring light to illuminate fluorophores, in techniques such as STED and STORM; And finally, increasing the gathering ability of the microscope by placing it in the near-field, used in techniques such as SNOM.

Our near-field micro-lenses, and the micro-spheres they are based on, have the advantage of a very short focal length, which may help to gather higher-angled light into the half-angle of the microscope lens.

### 2.2.9 The Near-Field

The near-field is a region of space approximately 1 wavelength from an electromagnetic source. In the case of a light scattering from a sample, it would be the point of scatter. When the size of the feature causing the light to scatter is below the diffraction limit, the scattered light is *evanescent*, meaning it decays as it propagates. These evanescent waves are only present within the near field because the rate of decay is great enough that their intensity drops to zero before entering the far field ($> 1\lambda$).
The mathematics to explain this comes from Fourier optics. In Fourier optics we define a wavevector of the light, \( k \). Note that this is not the same as the imaginary component of the refractive index, also sometimes referred to as \( k \). The wavevector is an inverse length measurement, and is related to the wavelength, \( \lambda \) by the following equation\(^{[66]}\):

\[
k = \frac{2n\pi}{\lambda} \tag{2.24}
\]

\( n \) is the refractive index of the medium. As \( k \) is a vector, it can be broken down into its three components, \( k_x, k_y, \) & \( k_z \), which relate to it by \( k^2 = k_x^2 + k_y^2 + k_z^2 \).

To understand what it does we define a simple wave form, based on the \( x, y \) & \( z \) coordinates, the wavevector, \( k \), angular frequency, \( \omega \), time, \( t \), and the phase, \( \phi \):

\[
E(x, y, z, t) = E_0 e^{i(k_xx+k_yy+k_zz−\omega t+\phi)} \tag{2.25}
\]

This fully described the electric component of the light, \( E \).

When light scatters from a very small object, the \( x \) and \( y \) wavevectors grow very large, hence for the total wavevector \( k \) to remain unchanged, the value of \( k_z \), the wavevector in the direction of propagation, must become very small. If \( \sqrt{k_x^2 + k_y^2} > k \), then \( k_z \) becomes imaginary. Substituting an imaginary propagation wavevector into Equation 2.25 we get:

\[
E(x, y, z, t) = E_0 e^{i(k_xx+k_yy−\omega t+\phi)} e^{-(k_zz)} \tag{2.26}
\]

The first exponential is mediated by the factor of \( i \) and forms a wave, the second exponential is real and negative, and hence decays. As stated earlier, the distance over which the wave decays is less than 1 wavelength, so to save these waves intervention (such as a micro-lens) must sit within the near field.

Due to the presence of \( n \), in Equation 2.24, the refractive index of the medium plays a role in how far an evanescent wave can propagate. When in a higher refractive index medium than air, the total wavevector is increased, and hence larger \( x \) and \( y \) wavevectors can propagate without the \( z \) component turning imaginary. This principle is used in large scale solid immersion lenses (SIL), where a plano-convex lens of a few millimetres in size, and of a higher refractive index than the medium (1.5-1.8) is placed upon a sample, lowering the diffraction limit within the lens. Whilst comparable with the technique of MON, it only reduces the resolution limit to \( \lambda/n^{[58]} \), unlike MON, where the resolution limit has yet to be defined.

Next we discuss axicon lenses and Bessel beams, in preparation for chapters 5 & 6.
2.2.10 Axicon lenses, Bessel Functions and Bessel Beams

A Bessel beam is a non-diffracting beam of light surrounded by rings of decreasing intensity. It follows the profile of a rotationally symmetric Bessel function. As it is non-divergent over the length of the beam, laser Bessel beams are used in many places for precise positioning of light, such as injecting impermeable substances into cells[8].

In biological imaging, light sheets are scanned through samples. A light sheet is a thin (≈ λ) beam of light that is extended along one axis. In cross section, such sheets have a Bessel profile. In Chapter 6 we demonstrate micro-optic light sheet formers that can curve the light sheet, as well as directing it away from the normal to the lens. This sort of control may be useful in both biological imaging, and light tweezing type applications.

Another suggested use for a Bessel beam, is the theoretical tractor-beam, a forward propagating beam which puts a net force on the target object towards the illuminating source[67]

An ideal Bessel beam has four, very distinctive properties, which we will discuss in order: it is formed by interference, it is non-divergent, it is self healing, and the intensity within each ring is the same as in the central beam. The final property noted here requires that a true Bessel beam have infinite energy, due to having infinite peaks, so experimental beams are actually Quasi-Bessel beams, an approximation containing finite-energy.

Forming a Bessel beam requires the correct optics. The axicon lenses used to make them are triangular wedges (forming a light sheet), or a rotationally symmetric conical lenses, shown in cross section in Figure 2.6. The Axicon needs to be illuminated by a wave-front with a Gaussian profile. The rays drawn in 2.6 a) show the path of light through an axicon lens. The light overlaps and interferes in front of the tip of the lens, forming the beam (shown in red). The higher-order components are formed by the interfering light around the central beam. Due to being formed by interfering light, the width of the beam can remain thin over its full length, with an ideal Bessel beam displaying zero divergence.

The beam formed here is a Gauss-Bessel beam, similar to an ideal Bessel beam but with finite energy and length. The length of the beam, \( L_b \), is dependant upon the diameter of the axicon lens, as well as the angle that the light leaves the axicon, which depends upon the axicon profile angle, \( \gamma \) as well as its refractive index, due to Snell’s law (Equation 2.1). The equation for the beam length comes from the diameter of the illuminating gaussian beam, \( w_0 \)[68]:

\[
L_b = \frac{w_0}{(n - 1)\gamma}
\]  \hspace{1cm} (2.27)
Figure 2.6: The figure shows an axicon lens forming a beam, with the x and y profile of the beam. a) shows how a Gaussian wave-front of light incident on an Axicon lens forms a beam of length $L_b$. The regions surrounding the central beam where the light overlaps form the higher-order intensity peaks. b) is the y-intensity profile across a Bessel beam, showing the standarding Bessel function, whilst c) is the x-profile, showing a typical shape of a Gauss-Bessel beam, or Quasi-Bessel beam.

Therefore, in a glass axicon, for the length of the Bessel beam to be greater than the diameter of the incident beam, a very shallow axicon angle of 2° or lower is needed.

Because it is formed via interfering light coming in from the off-axis of the beam, if an object partially obstructs the path of the beam it will self heal. This means the beam will reform after the object.

The third property we noted, that the intensity within each ring matches that of the central ring, is what causes the drop off in intensity profile. As the surrounding rings have an increasingly larger area than the central beam, the peak intensity must be reduced.
In our previous section on optics, we touched on the idea of a Bessel function when describing an Airy disk. We saw the pattern of a central peak, surrounded by peaks of decreasing intensity. Mathematically, the profile of a Bessel beam is a Bessel function, a solution to Bessel’s differential equation[69], shown in Equation 2.28:

\[
x^2 \frac{d^2 y}{dx^2} + x \frac{dy}{dx} + (x^2 - \alpha^2)y = 0
\]  
(2.28)

In this equation \(\alpha\) can be any number. The general solution to this equation, when \(\alpha\) is an integer, comes in the following form:

\[
y(\alpha) = AJ_\alpha(x) + BY_\alpha(x)
\]  
(2.29)

Where A & B are constants. The two functions of \(x\), \(J_\alpha(x)\) & \(Y_\alpha(x)\) are known as Bessel functions of the First and Second Kind respectively.

The difference between the first and second kind is that, when \(x\) is zero \(J_\alpha(x)\) is finite, whilst \(Y_\alpha(x)\) is infinite. Bessel beams follow a Bessel function of the First Kind:

\[
J_\alpha(x) = \sum_{m=0}^{\infty} \frac{(-1)^m}{m!\Gamma(m + \alpha + 1)} \left(\frac{x}{2}\right)^{2m+\alpha}
\]  
(2.30)

In this \(\Gamma\) is the gamma function, a factorial function extended for complex numbers. \(\alpha\), is a real number, that can be negative, which denotes the order of the function. A Bessel beam is usually of zeroth order, with a central peak, seen in Figure 2.6 b). Higher-order functions have two peaks, surrounding the centre, which then progress into lower intensity peaks, examples of which are in Chapter 6.

2.2.11 Optics Conclusion

In this first section, we have covered the basics of geometric optical theory, including Snell’s law of refraction, and the basic properties of several varieties of lenses, including: bi-convex, plano-convex, plano-concave and ball-lenses.

We defined the magnification of a lens in Equation 2.4, the ratio between object size and image. The magnification along with the contrast, shown in Equation 2.23, are key variables we use to describe the effectiveness of our micro-lenses, in Chapter 4.

We described how a plano-convex lens forms an image, and how, when the object is closer than the focal point, a virtual image is formed below the lens. We noted how in our literature review, researchers viewed the super-resolution patterns only when the objective microscope was focused below the sample surface, suggesting they were seeing a virtual image. In Chapter 4 we see a similar result with our lenses.
We then derived the equation for the focal length of a plano-parabola lens, as the equation was not commonly in use. We found the focal length of the lens \( f \), in terms of the focal length of the parabolic dish that defined it, \( f_d \), seen in Equation 2.16. It indicated that for a refractive index close to conventional glass \( (n \approx 1.5) \), the lens focal length is 4 times longer than that of the dish. In Chapter 4 we look at simulated data (run using MEEP, discussed in the next section) to see whether this derived equation is accurate.

Next we defined three resolution limits of light, all of which dependent on the wavelength of illumination. The limits are defined based on the distance between the Airy-disks of two point sources. Whilst the Rayleigh criterion sets the highest limit, at around 321 nm spacing for 500 nm light, it is possible to resolve down to 263 nm for the Abbe limit, and 247 nm for the Sparrow limit, for the same illuminating wavelength. When discussing whether we have achieved super-resolution imaging, in Chapter 4 we will look at whether we have imaged below any or all of these three limits, baring in mind that the Sparrow limit is not often used for conventional microscopy, and is more relevant when discussing fluorescent, point-sources.

The final area we covered was that of Axicon-lenses. These are used to form Bessel beams, long beams of light with a Bessel function for a profile. In chapters 5 & 6 we look at simulated and experimental data for a curved beam former. The key quality of a Bessel beam is that it is formed by interference from the opposing sides of the lens. This allows it to be non-divergent over long distances, as well as self-healing, reforming after an obstruction. In profile, they follow a Bessel function, seen in Figure 2.6, where a central peak is surrounded by peaks of radially diminishing intensity. We described a little of the mathematics, noting that the function followed is that of Bessel function of the First Kind, which is finite at \( x=0 \), whilst the Second Kind is infinite, and so not physically achievable with optics.

With the theory of optics in place, including an understanding of how lenses work, and their key properties, as well as an understanding of Bessel optics, we now move into computational optics. In the next section, we discuss \textit{MEEP}, an electromagnetic simulation software, used for testing optical elements.
Chapter 2. Theory & Modelling

2.3 Computational Modelling

2.3.1 Introduction

When modelling optics, the two most common techniques used are ray-tracing and the finite-difference time-domain (FDTD) method. Ray tracing is a technique in which light is modelled as travelling in thin, straight lines, usually only changing direction at an interface (such as a change in medium). This technique is commonly used to design macroscopic optics, such as telescopes [70] and can give a very quick tool to understand how several lenses or mirrors will interact with one another. However, for near-wavelength scale optical systems, diffraction and interference strongly affect the behavior of the light. Ray-optics do not take into account diffraction type effects, and so can not be used to fully model very small lens, or mirror systems, or even thin-layers.

FDTD is a common technique for optical modelling in which Maxwell’s equations are solved point-by-point over a computational mesh, increasing the time-step after each full iteration to evolve the system. This way the system slowly progresses in time, and can give very accurate results. The downsides of FDTD is that, compared with the geometric optic approach, it is both time and computationally heavy.

When it comes to running FDTD, several software options are available, including; Comsol, Silvaco, Rsoft and MEEP. Comsol and Silvaco are both products of similarly-named software companies, designed as full-physics package simulators. Both are able to model optical systems as well as thermal, electrical, and other properties. Rsoft is a specialist electromagnetics modelling software, used in many industries from optical-communications to microscope design. MEEP is an open source FDTD solver written by Massachusetts Institute of Technology (MIT)[71].

Unlike the other three options, each of which are expensive, there was no cost for MEEP, which is distributed under an open-source license. Whilst Silvaco was available to us, due to the university owning several licenses, using the software was more complicated then MEEP, meaning several months solid work would be needed before any results could be obtained. This lead us to choose MEEP as our main modelling tool.

2.3.2 Introduction to MEEP

MEEP is an open source software to model the propagation of electromagnetic waves. To create a model, the user writes a control file, defining the properties of the geometry, source and boundary conditions. This control file can be written in either C++ or Scheme, an open-source scripting language. The control file is then run by the MEEP
program, which outputs the data specified as an \texttt{.h5} file, a format known as a *Hierarchical Data File* (HDF5). These files are particularly good for storing large sets of n-dimensional data and images.

With the \texttt{.h5} file in hand, the data can either be output as a set of images using the \texttt{h5topng} command in Linux, or it can be read directly into Matlab, a mathematical environment tool, and processed within. For this project several Matlab scripts have been written to extract useful data from the large datasets created by MEEP. Whilst we will not go into detail about them in the main text of this thesis, they have been detailed in appendix A.3.2.

Before detailing how the model was built, it is useful to discuss some of the nuances of MEEP. Due to the scale invariance of Maxwell’s equations, MEEP was written to be similarly scale invariant. A pseudo-scale factor $a$ is given by the user (in our case $a=1\mu m$), defining the apparent size of 1 unit of the computational cell. The beginning of any script written in MEEP starts by defining the size of the computational cell, based on the scale factor needed. In our case, we use a 26 by 26 unit computational cell, corresponding to a square with 26$\mu m$ sides. The edge 1$\mu m$ is saved specifically for defining the boundary conditions, giving a 24$\mu m$ square to build our geometry within.

As ‘$a$’ defines the scale, it also sets the relation between the size of the computational cell and the wavelength, $\lambda$, & frequency, $\nu$, of the illumination. Constants, such as the speed of light, $c$, and the permeability and permittivity of free space, $\mu_0$ & $\epsilon_0$, are normalised to 1. As much of the output from MEEP is in the form of a ratio, these values naturally cancel anyway, making preparation of the unit cell much easier. Due to these changes, the commonly known formula for converting between frequency and wavelength, is modified to:

$$\nu = \frac{c}{n\lambda} = \frac{a}{n\lambda}$$ \hfill (2.31)

Where $n$ is the refractive index of the material. This means a wavelength of 500nm (green light), equal to a MEEP wavelength of 0.5 units, gives a frequency of 2.0 units. As all MEEP sources are specified in frequency, and not wavelength, understanding this conversion is important. From setting $c$ equal to 1, it arises that our value of $a$ controls not only the spatial length of the cell, but also the temporal length of the model, as $\frac{a}{c} = a$ giving it a time component. The number of timesteps for which the program needs to be told to run, $T_i$, is controlled by the frequency and the intended number of timesteps for which the program is to run, $T_t$, and relate to one another by Equation 2.32.

$$T_i = \frac{T_t}{\nu}$$ \hfill (2.32)

As ‘$\nu$’ is ultimately controlled by ‘$a$’, this shows how it affects the time in the program. Now that we understand how the spatio-temporal size of the cell is defined, we can begin
to understand the resolution. Whilst the computational cell defines the full size of the computational domain in units of ‘a’, when modelling, each unit length is subdivided by the resolution, to finally make up the mesh of points that MEEP calculates over. This means that as the resolution increases, the precision of the model increases, but the output size for a single timestep increases by the square of the resolution (for a two-dimensional model) or the cube of the resolution (for a three-dimensional model, which we have chosen not to use). This increases the computational time by the same square, or cubed factor.

Similarly to how a can be considered a metric of both space and time, the resolution also controls the coarseness of the timesteps, further increasing computational time for the model for a higher resolution. From testing, a resolution of at least \( \lambda/8 \) is needed for an accurate model, with \( \lambda/12 \) being an optimum balance between resolution and computational time.

Later on we begin to introduce metals to our models, allowing us to create slits and look at how thin-metal layers on surfaces affect the light. Whilst MEEP will accurately produce data with the real index of refraction alone, and no absorption information about the material, modelling of metals requires processing of the polarisability of the materials, alongside the refractive index.

As a full discussion of how MEEP handles metals is outside of the scope of this thesis, we will just highlight the important points. MEEP bases its modelling of metals from the Lorentz-Drude model, using the following equation for the materials permittivity[72]:

\[
\epsilon(\omega) = 1 - \frac{\nu_1 \omega_p^2}{\omega^2 + i \Gamma_1 \omega} + \sum_{j=2}^{n} \frac{\nu_j \omega_p^2}{\omega^2 - \omega_j^2 - \omega^2 - i \Gamma_j \omega} \tag{2.33}
\]

Simplifying this equation down to three values, \( \omega_j, \Gamma_j \& \sigma_j \), gives MEEP enough to describe how a specific metal reacts to light. Usually, several sets of these values are needed to fully describe the metal (j =2..n), such as in the case of the silver model we use, in which 6 sets of the three values are used. In a similar way to how increasing the resolution increases computation time, the higher that \( j \) is taken to, the longer the model takes to run.

In the models, silver has been the primary metal chosen. The values of \( \omega, \Gamma \& \sigma \) for silver had been previously calculated by Krishna-Juluri, and so his values were used[73].

### 2.3.3 Building the model

As mentioned earlier, the model itself is built in 5 stages. First, the size of the computational cell is specified, along with the resolution. Next, the geometry of the model
is defined. Whilst simple geometries, such as a circle or a box, have pre-programmed commands to define them, complex geometries, such as a parabolic curve, needs to be input as mathematical formulae. These must be input at the earliest stage of defining the geometry.

After the geometry is defined, the source has to be specified, as mentioned earlier, in units of \( a \). The source can either be continuous, switching on and staying on for the duration of the run time, or Gaussian, ramping up and then down over a time period. For a Gaussian source, the central frequency, \( F_{\text{cen}} \), and frequency width, \( F_{\text{wdth}} \), have to be defined, along with positional information on the source. The central frequency can easily be converted into a wavelength by Equation 2.31, giving the central wavelength. The frequency width, it is worth noting here, defines the width of the Gaussian wave-packet of frequencies. Therefore converting from frequency into wavelength gives the range of wavelengths surrounding the central wavelength that will be within the wave-packet.

Next, the boundary-conditions of the computational cell have to be set. Three possible conditions exist, Bloch-periodic boundaries, a perfectly matched layer (PML) designed to absorb 100% of incident light, or a set of metallic reflecting walls. As reflections interfere with the main data in our model, we decided a surrounding PML layer was the best way to minimize interference. Unfortunately, the PML layers at the top and bottom of the cell do not absorb as well as intended, due in part to the direction of the light being at 90° to the ideal absorption direction for the layer. Whilst some reflection is experienced within our model, the combination of choosing an appropriately short run-time, and enlarging the size of the computational-cell limit the effect of reflection on the central structure.

Finally the model has to be told what to output. MEEP can output many different types of data, including the electric (E or D), or magnetic (B or H) components of the field, or the Poynting vector (S). It can also output the 'Total Power', giving the sum of the energy density of the electric and magnetic fields. We tend to use the total power, unless otherwise stated, as it gives the fullest picture of how the light reacts when passing through our optics.

Within the .h5 datafile is a 3D data set, including the x, y and time axis. We extract this data, and then sum each point over the whole time-set, creating a single, 2D intensity map of how the light progressed through the structure. Figure 2.7 shows a pair of examples, a parabolic and a spherical profile dish, each reflecting light to form a focal spot. The incident light came from the right as a flat wavefront. Comparing the two, their focal points appear almost identical. But measurements show that, whilst the width is less than 1% wider for the spherical focal point, the length is 5% greater. If a
Figure 2.7: A comparison of a) a parabolic and b) a spherical profile dish. The diameter and depth of the dishes was 6µm and 1µm respectively, giving an f# of 0.375. The focussing properties appear almost identical, but the spherical focal point is almost 5% longer.

longer focal-length dish were used, we would likely see the length of the spherical focal point increase relative to the parabolic, due to spherical aberration.

The data itself is normalised to the intensity of the incident light. Giving an example, the peak intensity of the focal spots in Figure 2.7 is just under 14 times the incident waves intensity. As the run-time dictates the number of timesteps summed over, for a true normalisation the data should be divided by the runtime. However, for all of the sets of models we compare, the same run-time was used within the set, making this step unnecessary.

With an understanding of how MEEP operates, it is interesting to briefly describe an example of an early simulation, run to test that the software was giving realistic output. All of the MEEP control files that were written as part of the work detailed here, and in Chapter 6 have been added to Appendix A.3 at the end of this thesis.

2.3.4 Anti-reflection

In an early test of MEEP, to judge whether the software was suitable for our purposes, we modelled structural anti-reflection. Anti-reflection is a means of reducing the reflection from a surface when light moves between two media of different refractive indices. It can be achieved by two methods, by placing a thin ($\frac{1}{4}\lambda$) layer of a material with a refractive index between that of the two media. This is commonly used in spectacles, and can be seen as a green/purple sheen. The second method is known as moth-eye anti-reflection, in which small, sub-wavelength structures are built upon the surface of a medium, creating a refraction index gradient at the interface, increasing transmission
into the surface\cite{74}. It is named after the micro-structure found upon a moth’s eye, which gives the moth better night vision.

To model this, we simplified the structure to a sinusoidal wave. Simulations were run for several sin-curved surfaces, along with a flat plane. They were illuminated by a Gaussian wavepacket of $500 \pm 250$ nm wavelength. Figure 2.8 a) shows a set of images showing the structure. The grey region is a refractive index of $n = 2.5$, whilst white is that of air. The wavefront (shown in blue) is travelling from right to left through the structure.

The function used to create the structure was that of a simple $A \sin(Bx)$ wave. The depth of the structure, which is controlled by $A$ was kept at a constant 650nm, whilst the distance between the peaks, controlled by $B$, was varied between 0-36. The resolution was set to 40 pixels per micron ($\lambda/12.5$).

In Figure 2.8 a) are images of a single time-step, as the light interacts with the surface of the medium. The darkest blue shows intense concentration of the light, which is present where the wave-front suddenly slows upon entering the medium. For the higher values of $B$, the wave is broken up, seeing a more gradual refractive index change between air and medium.

The graph in 2.8 b) reflects this analysis, showing that where no structure is present, just 2% more light is transmitted than reflected. As the value of $B$ increases, and the structures become thinner, the ratio increases, peaking at $B = 12$, with a 13% increase in transmission from a flat surface, reaching 64% total transmission.

![Figure 2.8: Demonstration of Anti-reflection using structured sin-waves. The function $A \sin(Bx)$ was used, with $A$ held constant and $B$ increased, forming closer and closer peaks. a) shows the set of increasingly packed sin-wave interfaces created within the model. b) shows the transmission/reflectance ratio for the varying values of $B$](image)
Our data appears to agree with anti-reflection theory, that smaller, close packed structures, form better anti-reflectors than larger, wider structures. The peak in the transmission at \( B = 12 \) suggests that, for the given depth, we found the sweet-spot, in terms of structural diameter. The peak-to-peak depth of the structure is \( \sim 750 \text{nm} \), close to the arbitrarily chosen depth (650nm). As we see from our data, anti-reflective structures enable more light to enter a high-index medium. Structural anti-reflection has been investigated for use in solar-cells\cite{75}, to increase light-gather and improve current generation.

### 2.3.5 MEEP, Conclusion

In this section we have introduced MEEP, an electromagnetic simulation software. We use MEEP in later chapters (4, 5 & 6) to test our lenses & optical structures. We went through the design flow of a MEEP control file, describing each section required in turn. These included: creation of the computational cell, definition of geometry, creation of light sources, the setting of boundary conditions and defining the output parameters.

When setting the geometry, the simulation defines regions by their refractive indices. Whilst \textit{MEEP} can simulate both the real (\( n \)) and imaginary (\( k \)) components of the refractive index, geometry can be defined as refracting only, and not absorbing (\( n \) only). We tend to avoid using absorption in our models, as we are most interested in the focusing properties caused by geometry.

We also highlighted the use of the scaling variable ‘\( a \)’, which sets the length of 1 unit cell within the model. We noted how, due to setting constants such as the speed of light to one, the scaling variable is used to convert between frequency and wavelength.

The critical value in modelling is the resolution. In MEEP we define resolution as the number of pixels per unit length (‘\( a \)’). In 2D or 3D simulations, the resolution is squared or cubed to build the volume, making an increase in resolution very computationally heavy. As the resolution also controls the temporal-resolution of the model, setting the period of a single time-step, it makes computational time increase by a factor of a cube, or to the fourth power, for 2 and 3-dimensional models. We noted that ideally, the resolution should be the size of the smallest feature divided by 10, or the wavelength over 10, whichever is smaller.

We also described how metals are defined in MEEP, using an approximation of the Drude-Lorentz model. In Chapter 6 we use thin (\( \sim 100 \text{nm} \)) silver films to block light in our curved jet-forming structures.
Whilst MEEP can output a wide variety of information, including the $E$ & $B$ fields and the Poynting vector, we choose to output the total-power, which combines the total field density of the $E$ & $B$ fields. Our final note on the output is how we use Matlab scripts to extract the data, compressing it to form time-integral images. We showed two reflective dishes in Figure 2.7, with parabolic and spherical curvature. Both dishes had the same focal length, giving them an $f\#$ of 0.375. Comparing the two, the intensity and spot size were almost identical, with the only difference being the spherical spot was 5% longer, and less than a 1% wider.

The last thing we described was a test of structural anti-reflection, run by modifying a flat surface of a higher refractive index ($n=2.5$) with a $\sin$ wave. By increasing the close-pitch of the $\sin$ peaks, we saw an increase in transmission, demonstrating moth-eye anti-reflection.

Having discussed the optical properties of conventional lenses, to compare with our micro-lenses in chapter 4, and then described a method of computer simulating unusual geometries, the next section of this chapter is on focused ion-beam milling. We use this technique to produce unusual geometries for Bessel-beam formers, along with high-quality micro-lens moulds. To aid the description of our manufacturing technique in Chapter 3 we will now discuss the use and drawbacks of ion-milling.
2.4 Focused Ion-Beam Microscopy

2.4.1 An introduction to Focused Ion Beam lithography

Focused ion-beam microscopy is a technique used in research and industry for its ability to quickly and accurately mill, ion-implant or deposit material upon a surface. It has several advantages over photo-lithographic techniques, including the fact it requires no mask, and hence very little sample preparation. The patterns themselves are very quickly and easily modifiable allowing the technique to be used for fast prototyping of small structures\[76\].

The technique is able to work on both the micro- and nano-scale, with accuracy down to a few tens of nanometres. When combined with an electron microscope it can be a powerful tool for imaging and then altering small structures. It has been used this way in the semiconductor industry for many years, to find and fix faults in micro and nano size electronic structures. The biggest flaw it suffers from is scalability. The maximum area a standard focused ion-beam system can mill is around a hundred microns square, much smaller than conventional lithography systems, and milling such an area to any appreciable depth (\(\sim 1\mu m\)), may take days. It is also much slower than other techniques when milling large scale or deep patterns.

New, Ion-Plasma FIB systems, are able to mill on a much larger scale, up to 150x faster than a conventional gallium-ion (Ga) source\[77\]. This is a big step to overcoming the drawback of slow, serial milling in FIB systems, but still does not compete with wafer-scale optical lithography.

The greatest asset of the FIB is the ability to quickly adapt patterns, remove or deposit complex geometries, and create metal contacts. For a lab-scale technique, the FIB is an excellent method of producing small prototypes. In Chapter 3 we use replication techniques to turn a single, milled geometry into multiple sets of copies, turning production into a scalable process.

The roots of ion-beam microscopy are linked with the electron microscope. Richard Feynman famously suggested the FIB microscope in his talk *There’s plenty of room at the bottom*. When discussing how to write the Encyclopedia Britannica on the head of a pin, he suggested ‘\textit{A source of ions, sent through the [electron]microscope lenses in reverse, could be focused to a very small spot}’, indicating this could be used to write\[78\].

The method works by accelerating a beam of ions, often gallium, towards the target substrate, so that upon collision the ion forces its way into the surface, implanting itself,
whilst surface atoms are sputtered away. By using a large number of ions, considerable volumes can be milled.

In the surface interaction that takes place, there are three important mechanisms to consider: surface sputtering, ion implantation, and damage to the substrate caused by the implanting ion. In the case of a crystalline substrate, such as crystalline silicon (c-Si), as the ion travels into the surface it causes damage to the crystal structure, leaving vacancies and misplaced atoms. When large numbers of ions are used, this can totally amorphise the surface.

This is the second of the FIB’s flaws, the damage induced in a sample by viewing or milling it. The implanted ions and damage can cause changes to the optical and electronic properties of the substrate. Researchers in ion-beam microscopy have developed many methods to reduce the effect that the beam has on the surface, including alternate ion-sources and heat-treatments, which we discuss in Section 2.4.8. Our interest in this area is related to the replication work we carry out in Chapter 3.3.5, where the surface amorphisation increases adhesion between the milled mould and the polymer replica. We introduce a method of removing the amorphisation and implanted gallium, using a hydrofluoric acid surface etch. This work was published in the journal *Micron*, in 2014[9].

In this section, we will discuss the FIB in detail, to prepare for the discussion of our manufacturing technique in Chapter 3. We begin by detailing the microscope itself, describing how the ion-beam is formed. This highlights the key variables used to control the FIB, which we then discuss in detail. Then we move on to the incident-ion mechanics, touching again on how damage is formed. Our discussion then moves to the main modes of use of a FIB, including imaging, implantation, milling and deposition. Finally, we finish the chapter by discussing current damage-healing techniques, along with a variation in ion-sources, something investigated heavily over the last few years and only recently emerging as an off-the-shelf product.

### 2.4.2 The FIB microscope

The focused ion-beam microscope consists of an ion column, chamber and surrounding pumps. In a dual-beam system, such as the one used for this thesis (FEI,nova-nanolab 200) there is a vertical electron column with an the ion-column is held at a 52° angle from the electron column. The additional column is used for imaging, and in some systems spectroscopy of milled samples. The gas injection systems (GIS), used for depositions, are also not shown. Several different species can be deposited, including tungsten, and platinum. Both are widely used for protecting samples, as well as creating electrical
pathways. Figure 2.9 shows a simplified schematic view of an ion-column, excluding the vacuum systems.

The mechanics of the ion-column are dedicated to focusing a tight, cohesive beam of ions that is rastered across the surface of a sample. Understanding the formation of this beam is key to understanding how to control the microscope.

### 2.4.3 Background and Operation

High-current focused ion beams, able to implant and mill, were first made possible by the creation of the liquid metal ion sources (LMIS) [79]. LMISs are based on Taylor cones which describe how liquids under electric fields evaporate [80]. Figure 2.9 shows a simplified schematic of an ion column. In FIB systems, a large voltage is applied to a sharp heated tungsten tip, coated in liquid metal, often gallium, causing it to pull into a cone. Due to the conical shape the electromagnetic field is magnified at the tip of the cone, leading the metal to evaporate in the field and form ions [81][82].

These ions are then accelerated by the electrostatic plates, up to the set voltage of the machine (usually 30kV). A Condensing lens (first lens) compresses the ions to form a tight beam, before a strip of apertures of varying size (beam selection aperture) selects the current of the beam[83]. A blanking systems allow the beam to be turned on and off in nano-second pulses, to protect the sample from accidental exposure when the beam is moving. An octupole magnet is used to shape the final beam-spot, to control beam stigmation, deflection and alignment[84]. The final lens controls the focal length of the beam, aligning the focus to the sample height.

To form a pattern, the focal-spot of the beam is moved from point to point, being blanked during movement, and allowed to rest in position for a short time. This rest time is the dwell time, which, for a given current, sets the depth to which an area is milled.

The voltage, current, and spot-size (or spot area) are three of the key control variables used when defining a pattern, alongside the dwell time, ion dose and the pitch of movement. Each of these must be defined and to do so we must understand how they relate to one another.

### 2.4.4 FIB control variables: Dose

The beam-current (I) is used as a measure for the number of ions arriving within the beam-spot every second, and is measured in pico-amps (pA) or nano-amps (nA). From the current and the beam-spot area ($A_s$), the current density can be calculated. The
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Figure 2.9: Simplified Schematic of a focused ion-beam column (excluding vacuum systems). The (red) path of the beam shows how the beam is narrowed from a high current, down to the small (pA-nA) milling current used.

dwell-time ($t_d$) is the time that the beam-spot spends in any one position, usually measured in micro-seconds ($\mu$s).

When designing our patterns for manufacturing parabola in Chapter 3, we often discuss the ion dose. The dose is used to calculate the depth to which a pattern will mill. It is measured in charge per unit (length/area/volume), from which we usually use the area dose. The area-dose ($D_A$) is a measure of the charge due to ions in the beam, arriving at an area of the substrate, measured in $\frac{nC}{cm^2}$. It is defined in Equation 2.34 as:

$$D_A = \frac{q}{A_S}$$  \hspace{1cm} (2.34)
Where $q$ is the charge of the ions passing through the spot-area, $A_s$. If we recall the relationship between current and charge, $I = \frac{q}{t}$, we can redefine the dose in terms of the current, spot-area, and dwell time, shown in 2.35:

$$D_A = \frac{I.t_d}{A_s}$$  \hspace{1cm} (2.35)

When considered alongside the sputter-rate of the substrate ($r_s$), which is the number of atoms each ion removes from the surface on average, the dose controls the depth to which an area is milled. In general, the relationship between depth and dose for a single crystal material is linear, where $\text{depth} = a.dose$. In Chapter 3.2.1 we discuss how we empirically measure the ratio between depth and dose, finding $a$, as a calibration step.

For our discussion later in this report it becomes relevant to define a related quantity, known as the ion-fluence ($f_I$). This is similar to the dose, but is measured directly in the number of ions passing through the spot area at any time, instead of charge. The ion-fluence is more commonly used by ion-implantation researchers, but will be relevant to Chapter 3.3.3 when we use ion-implantation modelling software to investigate FIB-induced damage. Calculating the ion-fluence can be done by dividing the dose by the fundamental electron charge, $e$, giving the following formula:

$$f_I = \frac{D_A}{e} = \frac{I.t_d}{e.A_s}$$  \hspace{1cm} (2.36)

### 2.4.5 Spot & pitch.

To form an image or to mill a pattern, the beam is rastered over the surface of the sample by moving the beam-spot from position to position. In imaging mode, the secondary electrons (or ions) emitted are measured, in a similar method to an electron microscope. When milling, the beam is allowed to dwell in each position for longer, sputtering more surface atoms.

If we consider the simplest case of the beam-spot being a circular area in which all the ions fall, we can look at beam movement as the distance between the centre of one spot position, to the centre of the next. This is known as the pitch. Looking within Figure 2.10 a), at the section labelled d), taken from a paper by Han. et. al.[85], three spots of equal diameters are shown with a variation in the pitch. As can be seen, changing the pitch changes the way the spots overlap. Due to this, altering the pitch has a non-linear effect on the amount of ions each area is subject to.

To add to the complexity of the situation, the profile of the beam must also be taken into account. Whilst we discussed the beam-spot as a simple circular area in which the
dose is uniform throughout, in reality the profile of the beam is a Gaussian with the peak at the spot centre. The spot diameter is taken as the full-width at half-maxima of the Gaussian beam profile\[86\], seen in 2.10 a), part b). This causes the spot edges to experience a smaller number of ions then the centre. Based on this definition, the spot diameter can be as small as 5-10 nanometres, increasing in size for higher currents. As the current pulled from the LMIS tip is continuous, when the current is selected by the aperture stip, the beam spot size is also set by this aperture size. This makes the spot area dependant on the selected current.

Whilst ions that fall within the FWHM of the beam profile are considered inside of the spot, a noticeable percentage of ions fall outside of this area. These ions make up the beam-tails, which have been shown to be far longer then the diameter of the beam-spot. As the ion-dose in the region of the beam tails is orders of magnitude lower then the beam-spot itself\[87\] the area exposed is only Ga implanted, but not sputtered. In Chapter 3.3.5 we demonstrate that they can have an effect 50 µm from the the beam centre.

Applying this understanding of the beam profile to the pitch, we can see in Figure 2.10 how the substrate is left uneven, with several protrusions, due to the Gaussian beam-spots not summing to a flat line. By increasing the overlap and reducing the pitch, the surface does become flatter; however, this increases the number of beam-spot positions needed to cover a given area, increasing the calculation and milling time for the pattern. Usually a 50% overlap is used (shown as 0.5 in Figure 2.10 d)), where the beam-spot radius is the same as the pitch.

Now that we understand the control variables of the FIB; current, dwell time, dose and pitch, we can begin looking at the physical mechanisms that cause sputtering, implantation and surface damage to understand how the FIB removes and deposits material.

### 2.4.6 Incident Ion Mechanics

By discussing the mechanism by which the FIB removes material we get a better understanding of the surface of the substrate after milling. When an ion hits the surface of a substrate, three mechanisms come into play: surface sputtering, implantation and damage.

The incident ion displaces surface atoms, entering the material and implanting itself within the material’s structure. The surface atoms, if given sufficient energy to break bonds, will be removed from the surface, sputtering away. As the ion travels into the sample, it releases energy as it slows itself, breaking bonds and releasing electrons from
atoms, creating damage sites\cite{88}. In dielectrics this causes surface charging, which can deflect the beam, causing uneven milling.

If enough energy is given to a surface atom to break its bonds, the remaining energy goes into moving the atom away from the surface, known as sputtering\cite{89}. These sputtered atoms often re-deposit elsewhere on the material, forming a very light amorphous layer. When looking at the sputtering of material in such a technique, the substrate material and the mass of the incoming ion play a big role in how many atoms are sputtered per incident ion. This ultimately controls the mill rate and therefore the depth/dose ratio. Ion-implantation simulations, using SUSPRE, a program further described in Chapter 3, show that the sputter ratio ($r_s$) of silicon to gallium ions is around 2.6 atm/ion. Using heavier ions to mill, such as gold, can increase this sputter rate and improve milling times. We touch on this in Section 2.4.8.

Due to different crystal facings of a given material sputtering at a different rate, the

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{beam_pitch_profile}
\caption{Beam pitch and profile, from Han et. al.\cite{85}. Figure shows the Gaussian shape of the ion-beam profile, and demonstrates how pitch affects the roughness of the milled surface.}
\end{figure}
ideal material to mill is single-crystalline, or amorphous. Polycrystalline materials (such as sputtered metal films) mill at variable rates and so, unless the milling area is smaller than a single crystal grain, the depth of milling is not continuous over the area.

When the ion enters the surface, it rebounds off of the atoms within the substrate, taking a random walk whilst knocking atoms out of position. For this to happen, the ion must break bonds between the substrate atoms, which in a crystalline structure creates vacancies and dangling bonds[88][89]. Ion implant modelling estimates that, for a gallium ion in silicon, $\sim 700$ vacancies are created per ion. The difference between the sputter ratio and induced damage, per ion, is enormous, and shows how even for very small depths milled, the surface of the sample can be completely amorphised.

The ion itself comes to rest in either a vacant area of the crystal lattice, or in an interstitial region. With the large number of ions implanted into the sample, the chemical, electrical and optical properties of the sample can be changed[90][91].

This leaves the substrate with an implanted ion within the material, a hole around the ion entry point and a damaged region down to the ion.

This understanding of the ion-implantation mechanism is crucial to our discussion of removing ion-beam induced damage in Chapter 3. By amorphising the surface of a silicon sample, and then exposing the surface to air, their is a chemical difference between the ion-implanted silicon (both amorphous and oxygenated), and the surrounding crystalline silicon. By hydrofluoric acid etching, the amorphised region can be removed, taking with it the implanted gallium. We calculate what percentage of the gallium is removed by the etch, as a function of the dose used during milling.

### 2.4.7 Modes of Use

Based on what we now understand about ion-beam microscopy, we can begin discussing the four main modes of use. These are implantation, milling, deposition and imaging. Ion-implantation has already been discussed in some detail. If the dose is kept low enough, surface sputtering is minimal, and so ions are implanted into the substrate. This alters the properties of the material, and can lead to p-type doping in silicon[92] or act as an etch stop for ZnO to HF etching[93]. In this low dose mode, it is common to see small amounts of surface swelling (1-4 nm steps), caused by the slight amorphisation of the surface.

Milling is where a high dose is used to sputter material. With the precise control over the spot position the FIB offers surfaces that can be sculpted into a wide array of shapes[94][95].
Deposition involves introducing a gas that contains the substance for deposition into the FIB chamber, near the beam. A gas injection system (GIS) is used, made of a needle that can be inserted close to the sample and introduce the necessary gas. When the beam passes through the gas, the ion reacts with the gas molecule giving it momentum in the direction of the beam, whilst additionally decomposing it to remove additional material that was used to make it gaseous. The molecule then adsorbs onto the surface, building up to form a layer within the area defined by the beam\[96][97].

Focused ion-beam microscopy is a useful tool for imaging on the small scale. Images can be formed by either secondary electrons, or secondary ions, both released by the energy deposited into the material by the incident ions\[83].

One perk of using a FIB microscope to image secondary electrons is the effect of ion-channelling. When an ion meets a crystalline structure at the correct incident angle it may travel down a crystal lattice between the atoms. The angle of entry is important to how far into the crystal the ion can travel, making the facing of the crystal structure important for an ion to be able to channelled. The further into a crystal the ion travels, the fewer secondary electrons are released that can escape the surface. Due to this, a visible contrast difference can be seen between different crystal facings, so that FIB microscopy can be used for viewing the grain structure of metals\[98].

A good example of ion-channelling is demonstrated by Kotan et al, when looking at the grain structure of iron-nickel alloys\[99]. Figure 2.11 shows images of Fe-Ni alloys made by a ball-milling process. The changes in crystal-grain orientation are clearly visible by the change in contrast throughout both images shown.

In any mode of FIB operation, including imaging, the beam still causes a small amount of damage and implantation to the surface of the substrate. As the majority of FIB machines use gallium as the ion-source, this leaves the surface both chemically and physically altered. Research has been conducted to look at the effect of this damage, and how it can be removed or simply avoided.

### 2.4.8 Ion sources and Damage Healing

As mentioned before, gallium is the most common choice as the ion-source in a FIB due to its low melting point\[100]. When a surface is subjected to an ion beam, it leaves an amount of gallium within the surface, along with possible broken bonds, dislocations and vacancies in the surface, depending on its original crystallinity. The effect of this gallium and surface damage is to change the optical and electrical properties of the material.
Figure 2.11: Focused Ion-Beam image of Iron-Nickel (Fe-8Ni) Alloy grain structure, from [99] Contrast differences come from a change in grain orientation, leading to a change in channelling acceptance angle.

Optically, implanted gallium has been shown to lower the transmission of light through FIB milled quartz, for wavelengths below infra-red[90]. Whilst electrically, implanted gallium has been shown to act as a p-type dopant[101][102].

Whilst these properties have shown to be useful when interested in doping, or otherwise changing the properties of the base material, quite often these changes are unwanted. Methods of reducing these gallium induced changes have been investigated, an annealing process being the preferred method, due to healing ion-beam induced damage as well as affecting the gallium concentration. Even at low temperatures, annealing has been shown to cause gallium to diffuse to the surface of silicon samples. There it agglomerates into beads, which then form into a thin gallium layer [103]. Similar gallium layers, formed by annealing FIB-milled ceramic oxides, have been shown to be very difficult to remove. Being resistant to nitric and hydrochloric acids, the gallium required further anneals in vacuum and pure oxygen then a plasma etch to fully remove[104].
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For a low ion dose \(5 \times 10^{13} \text{ ions/cm}^2\), high voltage (150kV) implant, Sato and his group found, via secondary ion mass spectroscopy, that after annealing at 850°C, 70% of the gallium was still left within the substrate\[105\]. As this was performed using a collimated ion-beam, not a focused ion-beam, we know the control voltage was significantly higher than that used by a FIB. This means an increased ion-penetration depth. Whilst the voltage is 5x higher then in a conventional FIB, leading to higher penetration and deeper implanted ions (105nm deep, compared with 25nm for 30kV), the data should still be relevant, being mindful that for a shallower implantation profile, more of the gallium has a chance to diffuse to the surface.

Reducing the surface gallium content is of particular interest in photonics. One study has shown that a liquid-based annealing technique improves the transmission of FIB milled photonic structures\[106\]. Whilst annealing both re-crystalises silicon layers and forces some gallium to the surface, it does not entirely remove the gallium from the material. Another method that does remove gallium from the equation is to change the ion-source in the FIB to another species.

2.4.9 Alternative ion-sources

Alternative ion sources are available, with liquid alloy metal sources (LAIS) being the current trend in research. Many different combinations of materials have been looked at, including: gold-silicon, gold-germanium, cobalt-neodymium, erbium-nickel, cobalt-dysprosium and many others\[82]\[107]\[108]\.

One advantage of these sources is that a single ion species can be chosen to form the beam spot by using a mass filter after ion-extraction from the tip\[107]\.

Interest in these sources exists for the implantation and sputtering regimes, low and high doses respectively. Figure 2.12 shows a graph from a study looking at both implantation and milling regimes, performed by Bischoff\[107\]. It shows the depth against dose for silicon-carbide (6H:SiC) milled using ion-beams of various elements. It shows how at low doses \(1 \times 10^{15}\) and below, all of the ion species implant leading to a small degree of surface swelling. When the dose is increased, all of the ions, bar silicon, lead to sputtering of the surface. The higher atomic mass elements show a tendency to sputter
at a lower dose, whilst milling much deeper than comparable doses of lighter atoms. This shows the increase in sputtering caused by a higher atomic mass ion source. The lightest element used, silicon, did not sputter surface atoms even when a high dose was used.

![Figure 2.12: Sputter and swelling depth as a function of Dose for various ion-species in silicon carbide; from Bischoff et. al.[107]. The diagram shows how increasing the ion-dose increases the depth of the milled region. The depth milled for a specific dose increases with the atomic mass of the ion-species used. No sputtering occurs for any ion species at doses below 5x10^15, demonstrating how very low dosages only implant ions.](image)

The most recent development in alternative ion-beam species is the plasma-FIB. In these FIB microscopes an alternative source, a plasma of ionised non-reactive atoms, such as argon, is ignited and controlled to form a beam using electromagnetic lenses[109]. This alternative source can deliver much higher currents than conventional LMIT sources, (185nA or greater, compared to 20nA). The key is that the high current from plasma sources can be formed into a smaller beam-spot than gallium ions, where a 200nA source would have a minimum spot diameter of 5µm , a plasma source would be 10x smaller, at 0.5µm , making accurate milling an achievable goal[77]. Plasma sources have now passed the development stage, and are now being sold commercially, with milling rates estimated at 20-50x that of a conventional gallium-FIB.

To finish with an unusual case, some research has been undertaken into cold-atom sources for FIB microscopes. In this setup, magnetically-trapped, laser-cooled neutral atoms are ionised by a precision laser pulse[110]. These ions are then controlled into the FIBs optics, forming a beam. The advantage of such a technique is the ability to use a selection of different neutral atoms, including the conventional cold-atom species such
as caesium and lithium\[111\]. The second advantage is the low ion straggle, reducing the size of beam-tails. However, the technique is keyed towards high-resolution viewing, and implantation, as the maximum current the system can currently deliver is $\sim 10\text{pA}$, close to the lowest current a gallium FIB would provide.

2.4.10 FIB Conclusion

In this section we discussed focused ion-beam microscopy, and its various methods of use. We described how an ion-beam is formed within the column, highlighting the variables that control the formation of the beam.

In Chapter 3, an understanding of these variables is crucial to the discussion of our ion-beam manufacture technique. Summarising these variables: The current controls the spot-area and the number of ions arriving in that area, which directly controls the total milling time. The voltage at which the ions are accelerated, we will see in Chapter 3, controls the depth to which the ions implant. When rastering across the surface, the beam-spot is moved in steps of the pitch, stopping at each point for the dwell-time. The combination of current and dwell-time controls the ion dose, which is a measure of the number of charges (ions) an area is exposed to, during milling. The dose is a very important variable, as it controls the depth to which an area is milled, and will be heavily discussed in Chapter 3.

We next discussed the incident ion-mechanism, highlighting the three key components: sputtering, implantation and damage formation. In FIB lithography, sputtering material is the goal, to remove controlled volumes. Implantation and amorphisation damage are side effects that researchers look to remove. We highlighted a few removal techniques, including high-temperature annealling. The annealing process has been shown to heal amorphisation damage in silicon, and force interstitial gallium to the surface. However, a large amount of the gallium is left within the surface, up to 70\% at high kV, whilst the gallium that does seep out is hard to remove with chemical etches. Comparing this to our result from direct acid-etching of the amorphised silicon, seen in Chapter 3, we estimate to be able to remove 99\% of implanted gallium in high-dose milling, by removing the amorphous silicon entirely.

As we have now defined the theoretical underpinnings behind FIB, in the next chapter we build upon this understanding, by describing our manufacture techniques. These include both FIB, and replication and reflow techniques.
Chapter 3

Mould Fabrication & Lens Replication

Summary
In this chapter we discuss our method for making silicon lens moulds, hydrofluoric acid treatment of the moulds, and further replication of the silicon in PDMS rubber to make a final mould onto which lenses can be spun. The first part of this work, detailing mould production & HF acid etching, was previously published in the paper "The fabrication of aspherical micro-lenses using focused ion-beam techniques", Micron, 2014 [9]. Some of the work detailed here, and in the paper is part of a patent (WO2010112827 A2), upon which the author of this thesis is not a named inventor. The mould replication and lens manufacture work is part of a second patent application, number (GB1418180.4), upon which the author is named.

3.1 Introduction

Having discussed in Chapter 1.2.1 where our work fits in to the area of super-resolution and micro-optics in general, we now move on to describe our method for producing micro-optical elements. We begin this chapter by detailing how we produce smoothly curved, micron-scale 3D features in silicon, of parabolic and spherical profiles. We then detail our method for hydrofluoric acid etching (necessary for overcoming adhesion issues), and replication from the mould, to produce PMMA micro-lenses suspended upon a thin-film. At the end of our method section we discuss modes of use for our micro-lenses, including
specially built apparatus to allow us to hold and move our lenses with respect to a small sample.

Having detailed our method we move on to our results, and examine the quality of the dishes milled. As part of this we continue our discussion on ion-beam damage from Chapter 2.4, by investigating how effective the method of using hydrofluoric acid etching is at removing the surface damage caused by the ion-beam & at restoring surface properties. This treatment was necessary to help reduce the adhesion between the Si mould and any polymer we pour onto it, as part of later replication steps. Finally we look at the quality of our replication, looking at the profile and roughness of our fully formed lenses.

We start by detailing how we make a parabolic dish.

### 3.2 Method

In this section we detail the focused ion-beam techniques used for manufacturing the original master moulds, followed by the polymer replication steps needed to create the final thin-film lenses we use in Chapter 4. We then cover our methods for characterisation, including atomic force microscopy, used later in this chapter.

#### 3.2.1 Theory of ion-beam milling a paraboloid

In focused ion-beam milling, the depth milled in any region is controlled by the dose of ions to which the area is exposed. Due to a broadly linear relationship between depth and dose in silicon, any region exposed to a controlled dose will sputter to a known depth\[112\]. This gives an advantage in that the depth for multiple patterns milled in the same place will be a summation of the individual pattern depths.

The dose is controlled by the dwell time, beam current and number of passes the beam makes over an area. Increasing the dwell time in a single-pass pattern results in a non-linear increase in depth for long dwell times\[113\], caused by the increasing difficulty of removing material from deeper structures. By increasing the number of passes made, dividing a single long-dwell into several shorter dwell-times, linearity between the depth and the dosage can be restored. By better redistributing the redeposited material, a higher number of passes also leads to a smoothing of the surface.

The technique we use involves an optimum combination between the dose and number of passes to lead to 'ideal' milling, overcoming these drawbacks.
We make use of the linear depth-dose relationship by milling a series of concentric entities, starting from the largest to the smallest, whilst varying the dose, such that the sum of the entity depths creates the desired profile. During our discussion the dose and depth can be considered inter-changeable.

As the entities in the pattern can be of any shape, from a circle to an n-sided polygon, this method allows for lens moulds of any footprint to be milled. Whilst we make dishes (rotationally symmetric shapes) as standard, troughs (symmetric along a single axis) are possible, by extending one of the sides of a polygon.

In Figure 3.1 we show how varying the milled depth of each entity in the pattern, we can achieve an approximately parabolic profile. The entity depth linearly decreases with entity diameter, such that when the overwritten depths are summed a parabolic profile is formed. In theory, this leaves a step edge, creating areas where the surface is either above or below the parabolic shape required. However, due to an edge effect whilst milling, the sharp peak of the corners will be eroded more quickly than the surrounding area. Some of the sputtered material, caused by milling, will redeposit and fill in the corners which dip below the parabolic function. Whilst we mill starting with the largest diameter entity, it makes the mathematics more straight forward to define the smallest entity as 1 and count upwards with diameter.

We have found only a small number of entities are necessary in the pattern to create a smooth profile, from 10-30 circles for 1-10µm dishes. For a spherical profile, the change between the deepest and the shallowest depth milled is quite dramatic. Using a larger number of entities in such a situation can be helpful, as it reduces the depth of each individual entity, forming a smoother profile.

As the dose is calculated by a combination of the ion-beam current (which is intrinsically linked to the beam spot size) and the dwell of the spot, the choice of beam current controls the speed of milling for a given depth. Most of the dishes that we have manufactured have been milled at 50-300pA, at an accelerating voltage of 30kV, optimizing the beam spot size for smoothness of the dish, whilst keeping milling times to a minimum.

3.2.2 Parabolic Profile Design

In Chapter 2.2.2 we defined the properties of a parabolic reflector. Its optical properties are determined by its diameter and depth, with the focal length of a parabolic dish, $F_d$, given by Equation 3.1:

$$f_d = \frac{D^2}{16c}$$ (3.1)
Figure 3.1: Diagram of Parabolic milling along with image of a Dose calibration trough. a) shows a diagrammatic cross-section of how linearly increasing the depth milled, for a set of concentric circles can sum to a parabola. Whilst this simple model predicts this method should create a step edge, over or under milling areas, preferential edge erosion during milling removes edges, as indicated, whilst redeposited material refill overfill areas to create a smooth parabola. b) shows a cross section taken through a 2µm silicon trough, made during calibration of the depth to dose ratio ‘a’. The trough had a platinum protection layer deposited on top for cross-sectioning. The parabolic curvature of the trough can be seen at the Pt/Si interface. Image c) is of a set of as-milled dishes, decreasing in depth from left to right, and decreasing in size from top to bottom.

In Chapter 2.2.4 we derived the equation for the focal length of a plano-convex lens, \( F_1 \), in terms of the focal length of the optically reflecting dish it is moulded from. We will restate it here in terms of the diameter, \( D \), and depth, \( c \), of the dish. The focal length of a lens of refractive index \( n \), is given by Equation 3.2 [114]:

\[
f = \frac{D^2}{8c} \cdot \frac{1}{n(\lambda) - 1}
\]  

(3.2)
From this we know exactly how controlling the depth of the dish will control both the dishes focal length, as well as that of a lens moulded from it. Longer focal length dishes and lenses are made from shallower dishes, requiring less material to be removed and therefore reducing milling times for a longer focus.

The dose required for each entity in the pattern is calculated from the total dose, $D_T$, required to mill the full depth of the dish, using the following formula:

$$D_T = \frac{c}{a}$$  \hspace{1cm} (3.3)

Here $'a'$ represents an empirically found ratio of the depth to dose. It is found during a calibration procedure by milling a pattern with a given value of $'a'$, and then cross sectioning the pattern and measuring the depth visually with the SEM column, pictured in Figure 3.1 b). $'a'$ is usually found to be $1.80\pm0.18 \text{ nm}/(\mu\text{C/cm}^2)$ for Si.

The next step is to sub-divide the total dose into a parabolic series of $n$ steps, where $N$ is the number of entities in the pattern. For any entity $i$, numbered from smallest to largest diameter, the depth milled can be calculated by equation 3.4:

$$D_i = \frac{2i - 1}{n^2}D_T$$  \hspace{1cm} (3.4)

Due to the nature of a parabola, you can calculate the smallest and largest entity depths and linearly interpolate between them to find all other depths. As the NPGS software is able to automatically interpolate between two given dosages, we need only calculate the smallest and largest dosages, using equations 3.5 & 3.6:

The lowest dose, applied to the central entity is calculated using :

$$D_1 = \frac{1}{N^2}D_T$$  \hspace{1cm} (3.5)

Whilst the highest dose, applied to the largest entity, comes from :

$$D_n = \frac{2N - 1}{N^2}D_T$$  \hspace{1cm} (3.6)

Whilst this gives us the dosages for a parabolic profile, it is possible to mill spherical, elliptical, hyperbolic or other profiles by using the relevant equation for the doses. We next cover calculating the dosage for a spherical profile dish.

### 3.2.3 Spherical Profile Design

Designing a pattern for a spherical profile lens is not as simple as for a parabolic lens. For a parabola, the dose from entity to entity increases linearly, which is not the case
for a circle. Unlike a parabola, for a hemispherical mould profile each entity must have its dosage input in by hand, as currently there is no method of doing this automatically. Along with that fact, for spherical profiles we must use more entities (usually up to 50) to get the best profile; this makes it preferable to manufacture parabolic profiles, where a spherical profile is not specifically required.

As before, the total dose, $T_D$, is calculated first, using Equation 3.3. The focusing properties of a sphere are well understood, and depend on the radius of the sphere, $R$, such that $F_s = R/\sqrt{3}$. Therefore, to create a spherical lens we must take a slice of depth $c$ (our mill depth) through a circle of radius $R$ to get a given lens diameter of $D$, and focal length $F_s$. By calculating the lens radius from the focal length, depth and diameter of the intended lens, this allows the focal length, which is dependent on the radius alone, to be varied for a fixed lens diameter, merely by changing the lens depth.

To attain the correct sphere radius required from the dishes diameter and depth, we use equation 3.7:

$$R = \frac{D^2}{8c} + \frac{c}{2}$$  

(3.7)

The distance from focus above the flat side of the lens we label $A$, and simply comes from the radius and lens depth:

$$A = \frac{R}{\sqrt{3}} - c$$  

(3.8)

As before, a set of concentric entities are used. We still define $i$ as the entity number, but now start from the largest to smallest, again to simplify the mathematics. The step size, step, is the size difference between any two entities, given by $D/N$. To calculate the depth any entity, $i$, comes from a geometrical calculation of the total depth up to that pattern, minus the sum of every previously calculated depth. As before, the calculated depth is converted into dose by multiplying by the total dose. For any entity in the pattern the equation is as follows:

$$D_i = \left[ \sqrt{\frac{R^2}{3} - \left( \frac{D}{2} - i \text{step} \right)^2} - \frac{R}{2\sqrt{3}} - A - \sum_{j=1}^{i-1} c_j \right] D_T$$  

(3.9)

Having used this to calculate the dosage of each entity, a runfile is set up in NPGS and the spherical dish is ready to mill.

### 3.2.4 Other Patterning Options and milling

Whilst we have mentioned a variety of possible footprints for the lenses in our discussion, in Figure 3.2 we show the designCAD files for a) our standard circular footprint, b) a hexagonal footprint as an array, c) a standard trough, in which a square array has
been stretched in one direction, and d) a more complex two-part close-packed array of octagons. For each of these designs the same dose-calculations apply, making it very simple to create complex 3D geometries.

![DesignCAD drawings](image)

**Figure 3.2:** DesignCAD drawings of a) a 5\(\mu m\) set of concentric circles, used for a standard dish, b) an array of seven 3\(\mu m\) hexagonal patterns, c) a 5\(\mu m\) diameter by 10\(\mu m\) long trough, made by stretching a set of square entities in one-direction, and d) close packed array of 3\(\mu m\) octagons with 1\(\mu m\) squares.

Before the pattern is milled, it is good practice to use a Faraday cup to measure the beam-current of the ion beam, to help account for any variance that can occur to the calibration step, calculating \(A\) as used in Equation 3.3. Over time, the beam-current tends to increase, due to the aperture strip used to select beam current getting worn away, allowing more current through.

With the CAD and runfile prepared, a standard silicon wafer (p+ Boron doped [001] lattice) is placed in the FIB. Usually the wafer is triple-cleaned in acetone, methanol and IPA, followed by a 5 minute oxygen-plasma clean. However, for brand new wafer, out-of-the-box, this is skipped.
A standard procedure of aligning the electron to the ion column is followed, and a few basic 5\(\mu\)m diameter, 1\(\mu\)m deep troughs are milled and cross-sectioned to get a value for ‘\(a\)’ (as defined in Equation 3.3). Any necessary final-modification to the runfile is made and the pattern is milled. After milling, samples are measured by AFM, to get the pre-etch roughness and profile, before hydrofluoric (HF) acid etching.

### 3.2.5 Hydrofluoric Acid Etching

The initial Si mould is made using a focused ion-beam milling technique. Which whilst highly accurate, leaves a thin (~60nm) layer of amorphous silicon (a-Si) implanted with gallium ions, Ga (seen later in figures 3.11 & 3.12). This causes adhesion problems when attempting to release drop-cast, or spun-on polymers from the mould.

To overcome this, we use a hydrofluoric acid wash to remove the implanted section almost entirely, which we explore further in Section 3.3.3. Before etching, samples are washed in acetone, and de-ionised water, before drying with nitrogen. This removes the silver electrodag used to attach the sample to its SEM stub during milling. Next, samples are placed in a bath of 48\% concentration hydrofluoric acid (HF) for 30\(\pm\)1 minutes, at room temperature. When removed, they are washed in de-ionised water and dried with nitrogen.

To get an idea of any change in depth or profile, atomic force microscopy (AFM) profiles were taken before and after HF etching, seen in Figure 3.6. A Veeco Dimension 3100 AFM system was used with a tap-300G tip.

### 3.2.6 Low Dose

To understand how etching changes the morphology of the surface, we need to begin by looking at the smallest possible dose, and how dose affects acid etching.

To look at the minimum amorphisation required to successfully etch, which ultimately helps us understand how much Ga is left within the surface after etching, we manufactured Siemens stars at a very low dose (Figure 3.7). By AFM-measuring the depth of HF etched stars milled at known doses, before and after etching, information about the maximum depth the acid etches was found. Small doses were chosen, 10-90 \(\mu\)C/cm\(^2\) with one additional pattern at 900 \(\mu\)C/cm\(^2\), to limit the effect beam tails play on the depth measurements (described in Chapter 2.4.5). We call these doses ‘small’ when compared to the doses used in the milling of dishes, where a depth of 1\(\mu\)m requires a total dose of 20,000 \(\mu\)C/cm\(^2\) to mill.
To compare with these measurements, theoretical computer simulations were run using SUSPRE (Surrey University Sputter Profile Resolution from Energy deposition) [115], simulating the amorphisation and gallium ion concentration profiles going into the sample surface. The ion and target chosen were Ga and Si respectively, with an ion energy of 30keV, and fluences reflecting the dosages used, 10-20,000 \( \mu C/cm^2 \). The system uses a bucket fill method for calculating the number of ions implanted, throwing away ions that pass the ion solubility limit, rather than relocating them. Whilst the ion solubility of Ga in Si is low, less than 0.002 Ga ions/Si atom [116], we use 2 ions/atom to compensate for the effect of ‘bucket filling’. This turns out to be more physical than it first appears, as the system does not take into account interstitial Ga, only substitutional Ga, meaning more ions can sit within the lattice than SUSPRE would normally allow, which is similar to the effect of increasing the solubility limit.

When an ion is incident on the silicon surface, three events take place, which we described in detail in Chapter 2.4.6, but will re-state here: 1) the ion releases energy into the surface, causing the top most Si atoms to release from the surface and sputter; 2) the ion usually then enters the surface and follows a ‘random walk’, displacing Si atoms and creating vacancies. Finally, 3) when the Ga-ion has expended all of its kinetic energy it is left to either replace a Si within the lattice, or sit in a fracture between the lattice positions, and acting as an implanted ion.

It is the cumulative effect of the second event over many ions that amorphises the surface, leaving a thin layer of a-Si . A higher number of ions (beam-current) or a higher ion energy (beam-voltage) increases the degree of amorphisation and leaves more Ga-ions sitting within this amorphous layer. SUSPRE models this, showing the ion-implantation profile (approximately Gaussian) and the degree of amorphisation with depth. In Section 3.3.3 we look at models of Ion implantation developed from this concept of ion-implantation.

### 3.2.7 Preparation of cross-sections

To examine the ion-beam damage, and to directly view the extent of the damage before and after acid etching, cross sections of milled troughs were examined in a scanning transmission electron microscope (STEM). Parabolic troughs were milled: 3\( \mu m \) diameter, 30\( \mu m \) in length and 300nm deep. A trough pattern was used to avoid missing the centre when cross-sectioning a rotationally-symmetric structure.

Thin cross sections were cut using the same FIB as was used for dish preparation, an FEI nano-Nova dual-beam FIB microscope system with a Zyvex manipulation system, via the conventional means [117]. This was performed on an as-milled and an as-etched
sample. High-angle Ga ion-polishing was used to thin the sample to below 100nm to allow for high resolution imaging. By using this method, the amount of Ga implanted into the cross section is limited, due to the reduced chance of implantation at grazing incidence[118]. Whilst implantation does occur during sample thinning, the amount of Ga implanted should be uniform across the foil. Therefore, when looking at quantitative data, such as energy dispersive x-ray spectroscopy (EDX) our arguments are made with reference to this background that is present throughout the foil.

The STEM (Hitachi 2300A HD STEM with a Schottky Field Emission Gun, 200keV) used to view the cross-sections had an attached EDX detector (EDAX Genesis), which we used to map the chemical elements and their spatial distribution within the sample. We examine the data gathered in the STEM in Section 3.3.4.

3.2.8 Lens manufacture

With the silicon master-mould made, the next step is to make lenses from this mould, which we shall refer to as the ‘silicon master mould’ or Si-mould. We have made lenses using several polymers, including polystyrene (PS), polydimethylsiloxane (PDMS) and polymethylmethacrylate (PMMA). The simplest methods for making lenses is to pour the plastic onto the mould in liquid form and let it set, before peeling, or cracking it off of the mould surface.

3.2.9 Preparing the PDMS

We have used PDMS in many different ways, to produce lenses and lens-moulds, due to its ability to very accurately replicate small features. Here we will describe three methods for PDMS preparation we have used, including hot and cold setting, as well as spinning of PDMS.

3.2.9.1 PDMS hot and cold setting

The PDMS we use is Silgard 184, from Dow-Corning. It comes in two parts, base and hardener. We begin by mixing a 10:1 ratio of base to hardener (by weight) and stirring thoroughly. The vessel of PDMS is then placed into a vacuum chamber at room temperature for 15-40 minutes and allowed to out-gas, drawing out air bubbles from the mix. The out-gassing time is dependent largely on quantity of PDMS and the quality of the vacuum. It is worth noting here that PDMS has a working time of 2 hours before thickening beyond the point it pours readily.
When as many of the bubbles as possible have been removed, the PDMS is then poured directly onto the surface of the mould. We pour next to our features, and not directly onto them, to reduce the possibility of trapping air-bubbles as we pour (evidenced in Appendix A.1.1).

To cold set the PDMS the sample is placed in a bell jar, which is subsequently evacuated. Air is allowed to leak in over the 24 hour setting period, which slowly oxidises the top of the PDMS, helping to form a harder skin.

Hot setting involves baking the PDMS in a vacuum oven for 45 minutes to an hour at 80-100°C at a pressure of 200mbar. Air is then vented into the chamber and the baking is allowed to continue for another 45 minutes to an hour. This part of the process, as with the cold setting, helps a thicker oxidated layer form on the PDMS, hardening it. Then the sample is removed and allowed to cool for several hours before peeling.

3.2.9.2 PDMS spincoating

PDMS is prepared in a similar method to that used hot and cold curing. The sample is placed in a spin coater (Speciality coating systems, Spin-coat G3P-8). The prepared PDMS mixture is drop cast directly onto the substrate, with no out-gassing stage. The bubbles that form normally when mixing are forced out centrifugally during spinning. The sample is spun at 500 rpm for 30 s, at a ramp rate of 100 rpm/s. Next the sample can be hot or cold set, as before, with either 24 hours in a bell jar at room temperature, or 2 hours in a vacuum oven at 100°C. The PDMS sample thickness at this spin speed is around 500 µm.

3.2.10 Polystyrene and PMMA preparation

We mix our own PS & PMMA as it gives us control over the weight:volume ratio of the solution, as well as guaranteeing the age & viscosity of the mix. As the solution ages, solvent tends to be lost, leaving a more viscous solution than when first mixed. This can affect the thickness of the spun layers.

Polystyrene beads are mixed with toluene at a 1:10 ratio, by weight to volume. Similarly, PMMA beads are mixed at a 1:10 with anisole by weight:volume. In both cases, the mixtures are left on a digital orbital shaking table for 24 hours, to allow the beads to thoroughly disperse into solution. We choose to use toluene and anisole, so that when evaporated off all that remains is the pure plastic, in a hardened form.
Another method of making PMMA is to use methylmethacrylate (MMA) and add a polymerising agent. We have had little success with this method due to the oxidiser failing to trigger the reaction, probably due to a poor batch of MMA.

### 3.2.11 Silicon Master Mould preparation

The silicon master mould by this point has been milled and HF etched, removing much of the damage from ion-beam milling. Whilst pouring and peeling directly onto the silicon has been shown to be successful, sputtering a thin (20-50nm) metal layer on top of the silicon drastically reduced adhesion. From experience, the combination of HF etching and metal-coating increases the likelihood of successful removal from the mould over either method alone.

Gold, with a titanium adhesion layer, Nickel, Palladium and Chrome have all been found to work well to reduce adhesion. Nickel is often the worst to release from, we believe due to its tendency to catalyse the PDMS, binding it more strongly to the polymer. Whilst this metal layer can affect the shape of the dish, as the layer is so thin this only matters in the shallowest dishes, and can be accounted for by slightly increasing the depth of the largest entity in the CAD pattern by the intended metal depth.

### 3.2.12 PDMS mould making & PMMA lenses

We have made lenses out of a range of materials, including PS, PDMS and PMMA. Direct replication from a Si-mould is a very successful method. In Section 3.3.6 we discuss the quality of a few such lenses.

Using milled silicon to make moulds has several advantages. Single crystal silicon mills very uniformly, lending control over the process. It is also a common enough material to both labs and industry that there is much experience working with it, plus it is a relatively cheap material (when compared with other, single crystalline materials). However, as a mould it has one fatal drawback. It is highly brittle, and can crack under stress, including the stress of release.

PDMS peeling causes very little stress if performed correctly, and so the silicon mould is excellent for making PDMS lenses, however, due to sitting on a very thick film (∼5mm), PDMS lenses are not easily able to sit in the near-field of a substrate and so are not suitable for our use.

To overcome the issues relating to silicon as a mould, we have instead devised a way to replicate the mould in PDMS, replacing the master-mould entirely. Final lenses are
then produced in PMMA. Figure 3.3 shows a diagrammatic overview of each step in the method, from milling the mould, etching & metal coating, replicating in PS, replicating the PS with PDMS, and finally spin-coating and releasing PMMA lenses.

As we have discussed steps 1, 2 and 3, where we manufacture the mould (Section 3.2.1), chemical etch it (Section 3.2.5) and metallise it (Section 3.2.11) we will move straight on to the manufacture of the PS replica.

A metal ring is placed on the Si-mould, and lightly weighted down. Polystyrene solution is poured within the ring. The toluene is allowed to evaporate off over 3-7 days, leaving a thin (few mm) layer of polystyrene on the silicon within the ring. By gently running a scalpel around the outside of the ring, and using a small lever the polystyrene can be removed by getting an edge up, and cracking it off the surface, where it tends to smoothly de-laminate in a single go, without damaging the silicon.

Whilst cracking the polystyrene off had been successful several times when we first tried it on as-etched wafers, we began to find the polystyrene was adhering very strongly to the wafer around the milled-region, and the silicon mould would sometimes crack when de-laminating. This lead us to introduce the metal coating step, as previously discussed.

![Diagram of lens manufacture from start to finish, along with photographs and SEM images of the product at each stage.](image-url)
In Section 3.3.6 we look at how well these 'lenses' conform to a parabola, as well as their roughness.

The next step in the replication process is to make a final mould out of PDMS. A similar method to before is used, where a metal ring is placed upon the polystyrene mould and weighted down. PDMS is prepared as discussed in Section 3.2.9 and then poured into the metal ring, to form a thick layer (∼1cm). It is then placed into a vacuum chamber, and left for 24 hours. When fully hardened, the PDMS is carefully peeled from the PS. This is made tricky by the thickness of the PDMS not allowing it to bend like the thinner layers of PDMS used as lenses. The PDMS is then released from the ring, and is ready to be used as a mould. It is worth noting that the PS lenses are undamaged by the process, and so several PDMS moulds can be made from a single set. Currently we have made up to 4 PDMS moulds from a single PS lens set, without seeing any noticeable deterioration of quality of the lenses.

With the PDMS mould finished, the final step is to create a set of lenses on a thin enough layer to get the sample and lens feature in close proximity. The PDMS mould is placed into a spin-coater (see Section 3.2.9.2 for details), and a ∼1ml drop of PMMA solution is placed in the centre of the mould. It is then spun at 6000 rpm for 16 seconds, with a ramp-rate of 660 rpm/s. This gives a film thickness of 750nm, as described in Appendix A.1.2. The thickness of the layer can be varied between 500-800nm with spin speed alone. For thicker or thinner layers, the viscosity of the PMMA would require changing, by changing the weight:volume ratio of the mix.

The ramp-rate and spin time are set so that the PMMA forms a complete layer, but is still tacky. This allows a gently pressed PMMA ring to stick to the thin-films surface which, after scoring the film around the ring, facilitates release and future handling of the film.

An interesting note at this point is how the film pulls away from the PDMS mould. The film pulls outwards, ballooning slightly, before (very quickly) tightening up like a drum skin. The film is still slightly malleable for approximately a minute after release before eventually setting hard. When hard set, the film is surprisingly tough for such a thin layer, able to stand up to a small amount of abuse before breaking.

In Section 3.3.6 we discuss the quality of our replicated moulds and lenses. Next we look at methods of using our lenses with respect to samples.
3.2.13 Microscope Rig & Modes of use

Whilst developing the process to manufacture the lenses, we designed several rigs to hold both the lenses and sample, whilst allowing free movement of the sample with respect to the lens. The first of these rigs was made of brass, and can be seen in Figure 3.4 a). A stack of Attocube piezo positioners gives full x,y,z control of the sample below the lenses, which sit in the top plate of the setup. Figure 3.5 a) shows a schematic view of a sample, held underneath a set of lenses in the setup. The piezos allow fine precision in the z-direction, and scanning in the x and y. By holding the lens-holder and sample-holder in a single setup, the lenses can be held nominally flat with respect to the sample, helping when trying to position the sample into the close-proximity of the near-field.

![Figure 3.4: Photographs of the manipulation rigs used for lens & sample manipulation. The images show the a) first and b) second piezo rigs used in this project.](image)

Whilst giving excellent positioning ability, the biggest drawback with this setup is in its size. Being well over the 35 mm maximum distance that standard microscopes have between stage and lens, it does not fit under many microscopes without removing the stage. This makes fine positioning of the lenses with respect to the microscope almost impossible.

The second rig needed to be simpler, more compact, and importantly, fit under a standard microscope on top of its regular stage. This lead to a complete re-design. The key to making the setup small enough was to move the positioners out from under the sample. The sample then sits on a dropped platform, controlled by, but next to the positioning system, as can be seen in Figure 3.4 b). The Z positioners for both the lenses and sample are micrometres, giving around 1 µm in precision. The x,y positioners
are Omicron MS5 slide-piezo positioners, giving down to a 50nm step. This rig fits very well under a microscope, and gives the ability to very precisely move the sample underneath the lenses. Its biggest drawback is in the lack of piezo z-control on the sample, making carefully moving through the lenses focus impossible. Also, due to the long arm from micrometer to sample, moving in z gives some movement in x or y.

![Diagram showing the modes of use for a thin-film of micro-lenses.](image)

**Figure 3.5:** Diagrams showing the modes of use for a thin-film of micro-lenses. a) a schematic view of the first rig made, along with a lens film used as a cover slip for b) biological microscopes and c) an upright microscope

Future microscope rig designs could easily include the missing piezo z-movement whilst fitting under a standard microscope, by using brand new, smaller piezo stacks (the smallest available at the time of writing can be 25 mm for a full xyz movement). As modern piezos can include interferometric feedback controls, the absolute position between micro-lenses and sample could be measured, allowing proper mapping the sample with respect to the lenses focal point.

### 3.2.13.1 Lenses as coverslips

Whilst our piezo setups allow us to scan a sample with respect to the lenses, they are large, somewhat clunky, and require piezo controllers to allow their full use. The simplest possible solution for imaging with our lenses is to simply drop a sample onto the underside of the lenses and use the lenses as both a magnifier and a coverslip. Figure 3.5 shows the two possible configurations for using such a method of viewing. c) is used for
conventional, top-down microscopes, such as those commonly used in materials science. In this mode a very small sample can be held on through the electrostatic or capillary force. b) inverts the lenses, making it ideal for use on an inverted microscope, such as those commonly found in most biology departments. This is a workable solution for larger area samples (such as cells coating an area) where, due to the high number of samples and lenses, a few are likely to be centred correctly for viewing. However, it does lack the ability to position the sample, and guarantee a good image.

Having thoroughly described our method for producing lenses, we now move on to characterising them, along with a discussion of the effectiveness of HF etching to remove implanted gallium.

### 3.3 Results and Discussion

In this section we will begin our discussion about the quality of the optics we make, including the moulds and the lenses. We start with work we did looking at the quality of the original Si mould, leading quickly to how HF etching can improve its quality, and a strong discussion of how effective the etch is at removing implanted, surface Ga. We then move on to look at PDMS lenses, and then our final lens moulding process including, PS lenses, PDMS moulds and finally the PMMA lenses, looking at their shape and roughness specifically.

#### 3.3.1 AFM Profile

To look at the quality of the milled features, we begin by examining how closely a set of dishes follow a parabolic profile, before and after etching, along with the surface roughness. Figure 3.6 shows a parabola fitted to the profile of an as-milled and as-etched dish (5um diameter, 10um focal length). The fitted parabola fits well, with the exception of the bottom of the dish. The $\chi^2$ (chi-squared) is a measure of “goodness” of fit, with values closer to 0 indicating a higher quality fit. The $\chi^2$ value of the as-milled and as-etched dish is 7.96 nm and 9.03 nm respectively. This tells us the fitting is not as ideal post etch, due to the increased roughness at the dish bottom. Before etching, the increased deviation at the apex of the parabola is due to redeposition of material (see Section 2.4). The etched sample appears to have been cleanly etched everywhere except for at the bottom of the dish. Before etching, the increased deviation at the apex of the parabola is due to redeposition of material in the dish centre than usual (eg. dishes where another dish is milled in close proximity, or the dish is very deep). It is caused by the extra Ga-rich material shielding
the dish bottom from incident ions, lowering their implant depth. This means the c-Si region doesn’t get fully amorphised, leading to non-uniform removal by the HF acid.

The roughness of the dish can be investigated by closely fitting the dish, using a high order-polynomial (to account for skewness in the AFM measurement, introduced by the triangular AFM tip being non-symmetrical). The fitted profile is then subtracted from the original measurement, then the RMS-roughness, $\delta$, calculated from leftover noise.

We have calculated the mean RMS-roughness over several dishes to be 4.0 nm, before etching in HF, with the roughness changing very little after etching, where it reaches 4.1 nm.

Using the RMS-roughness, the total integrated scattering (TIS) of the features can be calculated using Equation 3.10, which gives a percentage of the light scattered at a given wavelength due to the roughness [114]:

$$TIS \approx \left( \frac{4\pi \sigma}{\lambda} \right)^2$$

(3.10)

As we are interested in minimising light scatter, we can re-arrange Equation 3.10 to find the smallest illuminating wavelength usable whilst keeping to below 1% light scatter. This gives us a minimum wavelength of 51 nm, well below that of visible light. Lenses accurately moulded from these dishes should, therefore, be of high enough quality to work for the whole visible spectrum, starting from extreme-UV up to infra-red, depending on the refractive and absorptive properties of the lens material.

### 3.3.2 Profile variation due to etching

In many of our deeper dishes, we see a larger variation from our intended profile than in our shallower ones. It is well documented [119][120] how the ion-beam incidence angle affects the sputter rate of material from a surface. When milling, we begin with an ion beam normal to the Si surface, however the topography quickly becomes curved. When milling a curved surface there is a continuous change in incidence angle, allowing certain angles that have a higher sputter rate to more quickly erode. This causes a non-uniform milling rate across the dish, leading to a change in the surface’s final shape. Due to high angles sputtering more readily, we tend to see this as an increase in the width to depth ratio, slightly flattening the profile of our dishes.

After etching, the shape of the dish can alter slightly again, due to the material removed by the acid. Figure 3.6 shows an AFM profile of a 5 $\mu$m diameter dish, a) before, and b) after etching. Whilst the dish remains parabolic after etching, the depth and diameter...
of the dish are seen to change. The depth of the dish increases from 85.4 nm to 112.7 nm after etching, comparing the dish edge to centre. As the dish edge profile after etching changes from flat to skewed towards the dish, as indicated by the red arrows, it suggests the dish edge has been etched along with the dish. The dish diameter decreases from 5.7 μm to 5.6 μm, caused by the non-uniform removal of material along the profile of the dish. Comparing the profiles of before (red dashed line) and after (blue dotted line)
in Figure 3.6 b) seems to show that the flattening effect caused by ion-beam incident angle is at least partially reversed by HF etching, with the etched dish showing a smaller width to depth ratio. The sample had an alignment marker scratched into the silicon to keep the orientation of the dish the same during every measurement. This removed the possibility that the effect seen was caused by the dish being imperfectly circularly symmetrical.

These changes to the depth and diameter of the dish subtly change the focal length of the dish. Other dishes, similarly measured, showed an increase of up to 30 nm in depth (for 1 µm deep samples) and 500 nm increase in diameter (for 10 µm diameter samples). For high precision work, this variation in depth caused by material removal will need to be accounted for, so that the final profile conforms to the required dish. In Section 3.3.3 we look at the depth of Si that 48%-concentrated HF removes in 30 minutes, as a function of the milling dose, helping estimate the change in profile expected after etching.

The cause of the surface profile skew in Figure 3.6 is the ion beam-tails. Whilst the ion-beam is described as a finite spot, its true shape is closer to a Gaussian distribution [83]. The tails of the beam have a sufficient number of ions to amorphise the silicon, allowing it to etch, as shown later by STEM imaging in Figure 3.11. Ion-range modelling software (The Stopping and Range of Ions in Matter, SRIM[121]), used to predict the implant depth of ions in matter, puts the sputter rate of Si for a 30kV Ga ion at ≈ 2.6 Si per Ga, whilst the damage caused by each ion is in the 100’s of dislocations per ion. This demonstrates how the surface can be very quickly amorphised by relatively few ions, without sputtering to a noticeable degree.

Due to this, the surface just outside the dish has been etched to a similar degree to the dish itself, causing the measured dish depth to remain consistent, whilst etching the surface outside the pattern decreases with increasing distance from the dish edge. As this keeps the depth of the dish close to the original milled depth, this works in our favour, as changes to the dish depth change its focal length. Our observations in Section 3.3.5 put these beam tails going out to a radius of as much as 50 µm from the dish centre, showing how large an area is affected even when milling small structures.

### 3.3.3 Ion beam damage and modelling

Having seen that the very low doses created by the ion beam-tails are enough to allow the Si to etch, we chose to investigate how small a dose is required to allow preferential etching, and how much of the implantation damage is removed by etching, as a function of the ion-dose. Doing so gives insight into how the depth etched is linked to the dose. When using very small doses, the effect of the beam tails is negligible, and so a true
measurement of etch depth can be made by AFM. This allows us to expose low dose samples, HF acid etch and then measure the depth etched accurately.

To look at the effect of HF etching in this dose region, a series of Siemens stars where milled, at doses between 10-90 µC/cm² plus one at 900 µC/cm². At the lowest doses, below 70 µC/cm², the silicon surface sputters very little, causing a slight surface swelling due to implantation and amorphisation, seen in Figure 3.7 a) at 10 µC/cm². Only minor, inconsistent removal of material is seen at the highest dose used, 900 µC/cm², as shown in (Figure 3.7 b).

![AFM profiles of 10 µC/cm² and 900 µC/cm² Siemens stars before and after etching. Inset shows the topographical map of the star. The horizontal colour change in the before 10 µC/cm² and after 90 µC/cm² are caused by a scan error in the AFM.]

Before etching, the milled patterns are not visible using the AFM; after HF etching, the depth of the stars increased, shown as the bottom images and profiles, c) and d) in Figure 3.7. Even for the lowest dose, the depth of the pattern increased to 12 nm post-etch. This shows how little a dose is required for the ion-beam to amorphise the surface sufficiently to preferentially increase the rate of etching.

In Figure 3.8 a) we plot the experimentally measured depth etched as a function of dose (black squares). The etch depth shows a logarithmic relationship, with the curve rapidly increasing after 100 µC/cm². The blue-triangular line shows the estimated
surface amorphisation at the depth to which the surface etched, taken from SUSPRE plots (shown in Figure 3.9). The line is centred around 31% amorphous, to within ±5%. This gives us a useful clue as to how effectively the HF attacks the surface.

Figure 3.8: Graph a) plots the etch depth against dose (black squares), as well as the percent amorphisation of the silicon surface at that depth (blue triangles). b) shows the modelled depth at which 31% amorphous is reached (black squares), as well as the estimated remaining gallium left in the surface after etching (blue triangles).

From this data we needed to understand whether restoring the properties of the surface is possible. This is important for reducing adhesion to the mould when replicating, and to restore the optical properties of directly milled lenses. To do this, we need to investigate how much of the Ga is removed along with the a-Si during etching. The etch-depth has already been estimated at 31%, but it is important to understand how much of the gallium is removed with the amorphous material during the etch. First we touch on the SUSPRE models themselves.

The models, shown in Figure 3.9, plot the concentration of implanted ions (black solid line) and the percentage amorphisation (blue dashed line) as a function of depth, for
four doses. The four doses represent four points on the logarithmic scale between our lowest dose, $10 \mu C/cm^2$, and the dose required to mill to a depth of 1 um in Si, $20,000 \mu C/cm^2$ (as calculated from our own calibration, described in Section 3.2.1). In all four plots, the implanted ions form a Gaussian distribution, peaking at the same depth, 26 nm under the surface. This is because the depth of implantation is controlled by the accelerating voltage (ion-energy), which we keep constant, and not the beam current (see Chapter 2.4.6 for our discussion on ion-beam properties). The $20,000 \mu C/cm^2$ distribution is noticeably truncated, caused by the implant reaching the saturation limit of Ga ions in Si.

![Figure 3.9](image-url)

**Figure 3.9:** Figure shows the SUSPRE plots of Ga implanting into Si, at four different dosages: 10, 90, 900 and 20,000 $\mu C/cm^2$. The black line (Gaussian profile) shows the concentration of Ga in the surface as a function of depth, whilst the dotted blue line shows percentage amorphisation with depth. The sample surface is at zero. The red line highlights the 31% amorphous point, where our 30 minute etch stops. The voltage was a constant 30kV, controlling the implantation peak and maximum implantation depth.

Figures 3.8.b) & 3.9 together tell us a story of how the depth of the amorphisation front moves with respect to the Ga implantation profile, changing the amount of Ga left below the critical 31% etch-depth.

The black-square line in Figure 3.8.b) shows the theoretically modelled depth at which the silicon reaches 31% amorphous, for various ion-doses. It climbs steadily from $\approx 9$ nm to 45 nm jumping to 54 nm from which point the increase is shallower and more linear.
The blue-triangle line in Figure 3.8.b) shows a theoretical prediction of the amount of Ga left in the surface after a standard 30 minute etch. This was calculated by looking at the size of the Ga peak below the 31% amorphous depth for the given dose, and appears to show three strong trends.

To explain these we look to the images in Figure 3.9, where we see theoretically modelled data of the percentage amorphisation (blue-dashed line), and concentration of implanted Ga (black line) with respect to depth. Four, increasing ion-dosages are shown. We can see that an amorphisation front (a-front) moves into the surface as the dosage increases. The shape of the implanted ion concentration (i-peak), a Gaussian distribution, stays static, but increases in magnitude with dose.

Linking back to Figure 3.8.b) it appears that the first region of the blue-triangle line (0-500 µC/cm$^2$) shows the i-peak getting bigger faster than the a-front moves into it. The second region (500-2000), in which the amount of Ga falls, shows the front moving into the surface faster than the Ga peak size increase. The final region (3000+) is an exponentially increasing region, but with an order of magnitude decrease in implanted Ga.

Attempting to understand the sudden drop between 2000-3000 µC/cm$^2$, we examined the modelling software. It was found that there was a sudden change in numerical method at a dose of 2800 µC/cm$^2$. This leads to a jump in the movement of the amorphisation front, which can be seen when comparing figures a) and b) in Figure 3.10. Figure a) shows an ion dose of a 2784 µC/cm$^2$, whilst b) is of 2800, a difference of 16 µC/cm$^2$. The amorphisation front has taken a dramatic step deeper into the surface, whilst also changing the rate of curvature at the top and bottom of the drop off region. This apparent increase in amorphisation depth greatly increases the amount of material removed, and hence the overall Ga removed from the surface.

Whilst the changeover from the low to the high amorphisation regime is discontinuous, the method for the higher and lower implantation dosages are considered reliable by implantation experts[115][122][123]. Hence, it is expected that the data further from the changeover should be accurate. This suggests that our remaining Gallium value at a dose of 20,000 µC/cm$^2$ should still be noticeably lower than for a dose of 10 µC/cm$^2$.

As we have used a standard etch time of 30±1 minutes, the depth etched across all our samples is reasonable standard, getting down to 31%±5 amorphous material. By increasing the etch time, it may be possible to etch further into the material, thereby gaining a further advantage by removing more implanted Ga.

So far, we have shown that theory predicts HF acid etching will remove most of the Ga implanted by FIB milling, with higher doses showing more efficient removal then
Chapter 3. Mould & Lens Fabrication

Figure 3.10: SUSPRE data demonstrating the transition in calculation method between a low and high dose. Both figures show the SUSPRE plot of implanted ion concentration against depth (black line), with the amorphisation percentage against depth (blue line), for Ga ions at 30kV into Si. The Si surface is at 0. Figure a) shows an implantation dose of 2784 $\mu$C/cm$^2$, whilst b) is of a dose of 2800 $\mu$C/cm$^2$. The sudden jump in position of the amorphisation front (blue line) between the images shows the change in mathematical method.

lower doses, which places it as useful process in the manufacture of many possible FIB manufactured structures. The next step is to look at Ga removal experimentally. In the next section we use scanning transmission electron microscopy (STEM) to image cross sections of the damage caused when milling.

3.3.4 STEM

To image the underlying damage caused by ion-milling, and confirm that HF acid etching removes both the Ga along with the a-Si, restoring the original surface properties, two cross-sectional electron-beam transparent foils were prepared using the FIB. The first was cut from an as-milled trough, the second from an HF etched (as-etched) trough; both were imaged in a STEM.

The brightfield image (transmission electron image, labelled (TE)) shown in Figure 3.11 shows four distinct layers, labelled 1-4, of an as-milled trough. The top two layers (1 and 2) are platinum, deposited to provide contrast, and to protect the silicon top surface during cross sectioning. Below this layer are two silicon layers (3 and 4).

Imaging layers 3 and 4 using high resolution TEM, shown in Figure 3.11 a), and taking Fourier transforms of each layer shows the loss of crystallinity in the top Si layer. The Fourier transform of layer 4, labelled c), shows the distinct bright spots denoting crystallinity in the layer. Layer 3’s Fourier transform, labelled b), has a smeared band.
of brightness surrounding the centre. Whilst this suggests a lack of order in the material, indicating it is amorphous, the band is not rotationally symmetric in intensity, suggesting some vestiges of order. This may be because the angle of incidence of the milling beam is consistently in the same direction (90° from the sample). Whilst ions take a random walk when moving through the Si crystal, causing spread in the damage caused, they can channel down crystal planes giving an overall direction to the damage.

Between the two silicon layers lies a thin black line, suggesting the change between amorphous and crystalline silicon. The line is 4±2 nm thick, and represents the drop off from 100% to 0% amorphous. Relating this back to the SUSPRE models in Figure 3.9 we see that, for a realistic milling dose, 20 000 µC/cm², the change between 100 and 0% amorphous is dramatic, occurring over 4nm, which agrees well with the measurements of this line.

The extent of the amorphous region shown in the TE and ZC images in Figure 3.11 varies between 46-52 nm deep within the milled trough area (to the right of the red arrow), and 61-70 nm outside of the designated milling area (left of the red arrow). From SUSPRE we expect the depth to be exactly 60 nm, and no deeper, but neither area agrees with this well.

Within the trough, this can be explained by the high percentage of Ga within the surface after the first few passes of the ion beam. The implantation and amorphisation depths decrease, due to the incident ions having to pass through a heavy, Ga rich surface, causing the ions to expend a significant portion of their energy closer to the surface. This could account for the reduction in depth from theory. Our method of using multiple passes helps to overcome this by helping a steady state to be reached.

Outside of the trough, the deepest 70nm region is directly next to the trough, with the depth reducing linearly to 61nm at a distance of 250nm away from the edge. We know the surface is not directly milled, and is only damaged by the beam tails, as we concluded in Section 3.3.2. The much smaller dose of the beam tails drops off as you move away from the dish, leading to a decrease in the amorphisation. This causes the slanted profile of the Si/a-Si interface visible in Figure 3.11, which we also saw by AFM in Figure 3.6. As fewer ions are implanted in this region, with little redeposition and no sputtering, this allows the Ga to reach the full implantation depth given by the model. The additional depth, above that predicted by modelling, may have come from an edge effect, with ions scattering from within the trough through the shallower trough, and adding to the depth.

Looking to the high-angle annular darkfield image (Z-contrast, labelled ZC) shown in Figure 3.11, the brightness is proportional to the $Z^2$ (atomic weight squared), material
Figure 3.11: The figure shows the transmission electron (TE) and zed-contrast (ZC) STEM images for a cross section of milled trough. The different layers of the milled surface can be seen. 1 and 2 are ion- and electron-beam deposited platinum, whilst 3 is amorphous and 4 is crystalline silicon. Image c) shows a HRTEM image at the a-Si /c-Si meeting point. The full width of the image is 36 nm. The smaller images are the Fourier transforms of the boxes marked in red (top transform) and blue (bottom transform) rectangular regions shown in c). They demonstrate a lack of crystallinity above the black line, whilst a crystalline layer below the boundary.

density and thickness (approximately constant) of the sample. We can see the ion-beam and e-beam deposited platinum (layers 1 and 2 respectively) as two brighter layers on top of two much darker silicon layers. Within the amorphous layer (3) in the area where the trough was milled, we can see a brighter line running near the top of the amorphous region, below the platinum, which we have highlighted in red & indicated with a red arrow. As the line is high contrast, suggesting a denser region or higher atomic weight, this could be indicating the presence of Ga. The lack of such a line outside of the trough
indicates a much lower degree of Ga implantation.

To confirm the presence of Ga, an EDX map of the area around the trough edge was taken for the as-milled and acid-etched troughs, as is shown in Figure 3.12 a) and b) respectively. The images show the EDX Ga signal (blue dots) mapped onto the transmission electron image (TE). Looking at the a-Si region within the trough for the as-milled sample, a), we see an area of very high Ga concentration (highlighted by the red ellipse) when compared with the a-Si outside of the trough, or the c-Si region.

This means that much of the Ga still sits within the a-Si layer in the trough, altering the layer’s optical and chemical properties. Comparing this to an EDX map of a chemically etched sample (b), we see very little Ga within the left-over Si, and no distinct a-Si layer. This agrees nicely with our SUSPRE model, that suggested the bulk of the Ga is removed along with the damaged a-Si.

EDX linescans of both samples were taken, seen in Figure 3.12 c). The red-triangle and black-square lines show the intensity of the Ga L-edge through the as-milled sample, through the trough-centre and trough-edge respectively, whilst the green-circle line is through the centre of the as-etched trough.

For both as-milled sample profiles, the signal increases within the a-Si layer, following a profile similar to the Gaussian profiles shown by SUSPRE. The Signal then drops off within the c-Si layer. At the trough edge, the peak Ga profile is much nearer to the surface, dropping off to the background before meeting the c-Si interface. The difference in profile is caused by the change in angle of the surface between the trough centre and edge.

Compared to SUSPREs prediction (Figure 3.9), both as-milled profiles peak nearer to the surface than expected, showing some skew when compared with the modelled Gaussian distribution centred tens of nanometres below the surface. Material removal by sputtering may account for this, as the milled top surface moves into the implanted front; the peak implanted region becomes spread, as new ions implant deeper into the surface, creating an overlapping set of profiles.

In the as-etched sample the Ga signal drops off towards the Pt/c-Si interface, with a reduced noise within the c-Si. Compared with the distinct peaks seen in the as-milled samples, the Ga percentage appears to be just over the background found in the bulk of the c-Si, showing most of the Ga has been removed along with the a-Si. This means the surface properties of the material should be, if not entirely restored, much closer to that of the original surface.
Figure 3.12: EDX maps and linescans of a trough before and after etching. The top and middle EDX maps show the Ga-L edge of a trough a) before and b) after etching, demonstrating the removal of a high-concentration region of gallium (blue-dots). Image width is 400 nm. c) shows EDX linescans taken before etching through the centre of a trough (red), edge of a trough (black), and after etching (green).

3.3.5 Silicon discolouration and Lens replication

Having demonstrated how HF acid can remove ion induced damage from silicon, we now move on to look at the chemical and optical changes that etching has on the Si mould, before moving on to lenses made from such a mould. Figure 3.13 a)-d) shows reflectance optical images of a set of Si moulds, etched for increasing amounts of time. In all four images, a bloom is visible around the dishes, caused by the beam-tail effects; note how they extend up to 50 µm from the dishes themselves. The first mould a) was left without
etching, but a slight lighter shading can be seen around the dishes. As the etch time increases, to b) 7 minutes, c) 15 minutes and d) 30 minutes, the size of the bloom around the dishes grows, and is visible without optical magnification for all etched samples. In d) the etching has created a colourful pattern, moving through hues of red and blue as you move away from the dishes.

As the colour change radiates outward from the dishes, it follows that it is related to the skew of the surface we saw in sections Section 3.3.2 and Section 3.3.4, caused by etching. From our models in section Section 3.3.3 we have seen how a decreasing dose does not change the depth to which Ga is implanted, but does reduce the depth at which the minimum amorphisation required to etch occurs, hence a smaller etch depth. It follows that for smaller doses, the depth of the remaining implanted and amorphised region after etching is larger. The colour we see in Figure 3.13 d) is caused by the thickness of the remaining damaged layer (a partially amorphous layer), which would increase from 4-60 nm radiating outwards. The colour itself appears to come from an interference effect[124]. In a paper by Jae Young-Lee et. al.[125] they manufacture transparent, thin-film a-Si solar cells where the colour is dependent on the layer thickness, which varies between 6-31 nm. This appears to demonstrate exactly the sort of interference effect we see caused by the remaining layer of damage.

In a single test, where the wafer was allowed to etch in HF for a full hour the colouration disappeared, returning the colour to its original state. Whilst only anecdotal, this suggests longer etch times may allow full removal of the damaged region.

The graph in e) shows the profile of dishes from the un-etched sample (solid green line), and the longest etched sample (dashed red line), after replicating PDMS lenses. Whilst both dishes should have approximately identical profile, the green profile shows large amounts of debris in the dish. This indicates a large amount of PDMS has remained stuck to the dish. Whilst this did not happen to all lenses, it did occur multiple times, across the small number replicated. In the 30 minute etch sample, no such problems have been seen, with similar smoothness and profile before and after replication. This shows that etching does reduce the adhesion between PDMS and the milled silicon substrate, aiding the replication process.

### 3.3.6 Lens Quality

With the milled & etched dishes known to be of very low roughness (~4nm), and of a good fit to a parabolic profile, we must now look at whether lenses and lens-moulds replicated from the silicon are also of good quality. We start with PDMS lenses we looked at early on in the project, before moving on to the PS, PDMS mould and PMMA lenses.
Figure 3.13: A silicon mould shown after an etch time of a) 0 min, b) 7 min, c) 15 min, d) 30 min. The silicon gets darker as the etch progresses, until colours appear in the 30 minute etch. e) shows the AFM profile of a mould after peeling PDMS lenses with (red) and without (green) HF etching the mould. The green (no HF step) profile clearly shows significant PDMS remaining within the dish.

3.3.6.1 PDMS as lenses

Figure 3.14 shows a set of PDMS lenses made this way. Optically, we see the reflected image in a) showing the lenses and transmission image in b) demonstrating their focal points. c) shows the AFM profile of a PDMS lens, along with a parabolic fit of the lens. The parabolic fit was performed in Origin, using a function made using equation 2.10

The $\chi^2$ is a measure of the goodness of fit, which we first mentioned in Section 3.3.1, is 0.5 nm. It suggests an extremely very good fit. We expected most of the error in the fit to be coming from the roughness in the profile. This roughness appears not to be a real-feature, but a side-effect of using too firm an AFM cantilever.
Whilst PDMS creates a very high quality lens, its biggest drawback is the thickness of the layer poured with it. Whether poured and set, or spin-coated, the PDMS is difficult to release without tearing unless around half a millimetre thick or more. Whilst such lenses could be suspended with the parabolic curve facing downwards towards a sample, and viewed from above the PDMS layer, such lenses appear to work best with the curved side facing upwards, as discussed by [51]. Requiring such a thick PDMS film means the flat side of the lenses cannot be moved close enough to the sample surface for near-field viewing.

To solve this problem we chose to make a replicated mould from PDMS, and to spin thin (700 nm) layers of PMMA to form our final films of lenses, as described in Section 3.2.12. In the next section we characterise the lenses and moulds used to produce these PMMA lens films.

![Figure 3.14: PDMS micro-lenses. A) and B) show optical images of a set of lenses of varying diameter, and the focal points of the lenses respectively. C) shows an AFM profile taken across a single lens, with inset topological image. The red-dashed line shows a parabola fitted to the AFM profile, which shows the lens is parabolic.](image-url)
### Material Roughness Measurements

<table>
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<th>$\lambda$ at 1% TIS (nm)</th>
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</tr>
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<td>292</td>
<td>61.6</td>
</tr>
<tr>
<td>PDMS Dish</td>
<td>5.3</td>
<td>5901</td>
<td>66.6</td>
</tr>
<tr>
<td>PMMA Lens (Film)</td>
<td>11.7</td>
<td>192</td>
<td>147.0</td>
</tr>
</tbody>
</table>

**Table 3.1:** AFM roughness measurements of the different moulding steps. The Silicon dish has a very low roughness before and after etching. The process steps introduce minor roughness, but the final PMMA lenses on a film have less than 12 nm roughness, making them of good quality. The $\chi^2$ shows how closely the dish/lens follow a parabolic profile, but does not account for skewness. The 1% wavelength shows the wavelength at which the roughness causes greater than 1% light scatter, deeply into the UV and beyond the usable wavelength range for all the materials.

### 3.3.6.2 Lenses and Lens Moulds

In the final section of this chapter, we will look at the quality of our replication and moulding steps, along with the final PMMA lenses. Figure 3.15 shows the AFM profiles of a) A PS lens used as a pre-mould, b) the final PDMS mould and c) PMMA lenses on a thin film. The inset shows the 3D topographical map of each sample. A parabolic curve has been fitted to each dish, and the $\chi^2$ measured, as well as the roughness. Table 3.1 holds the roughness and $\chi^2$ values of fits for the silicon mould (pre- and post-etch), as discussed earlier, alongside the polystyrene, PDMS mould, and PMMA lenses shown in Figure 3.15.

The $\chi^2$ indicates two of the three follow a reasonable fit to a parabolic curve, with Polystyrene and PMMA having a value below 300 nm. The $\chi^2$ of the PDMS trough however reaches 5901 nm or 5.9 $\mu$m of inaccuracy in the fit. This number is surprisingly large but, looking at all three of the images of the fitted profiles, Figure 3.15, the 'error' in fitting appears in the same regions of all three, near the top of the dish and the right hand side. It appears that the fitting function we use idealises for the left-hand side of the dish, which damages the fit of the right hand side. The cause of this miss-fit is due to skew in the measured shape of the lens or dish. Whilst this could be a feature of the sample, it is possible that a non-planar sample, and asymmetric AFM tip could also introduce such skew.

Comparing these values to the dishes, seen earlier in Section 3.3.1, these fittings appear much worse. However, as the depth of the mould used in Figure 3.15 is significantly deeper, it is not unexpected that the profile would conform less to a parabola, partly due to a higher amount of redeposition (leading to a flatter bottom), and partly due to the mathematics of the $\chi^2$ measure. As the error between fitting and data is squared, the difference is exacerbated. In the case of the PDMS, a single measurement contributed 4.2 $\mu$m to the sum of its $\chi^2$. The other aspect to consider is skewness.
We can see from Table 3.1 that the roughness of the surface increases when etching, and replicating, up to 11.7 nm on the final lens film. It is not surprising that the surface gets slowly rougher as we move from step to step, but if we calculate the minimum wavelength allowed for a 1% scatter for the PMMA lenses, as we did for the mould in Section 3.3.1, we get a wavelength of 147 nm, which is still deep enough into the UV to allow conventional fluorescence-microscopy and photolithography to be performed through the lenses.

We will further discuss the optical properties of the PMMA lenses in the next chapter, where we begin to use them for imaging small structures.

### 3.4 Chapter Conclusion

In this chapter we have described a method of making parabolic and spherical lenses suspended upon a thin film. We began by detailing an accurate method of milling smooth,
curved surfaces of microns in size. Due to surface damage caused by milling, we describe a method for removing the implanted gallium and amorphised silicon using wet chemical etching in hydrofluoric acid. This single step process is quicker then the more conventional two-step method of annealing and acid etching to remove implanted gallium. We also describe our method for estimating how effective removing the amorphised silicon is at also removing the gallium, using AFM to measure changes in surface profiles before and after etching, coupled with ion-implant simulations.

We then describe a replication method of producing micro-lenses from moulds, made using this method. Using a two-step replication process we form a copy of the original silicon mould using PDMS, a rubber mould that can be used to produce thin films of PMMA lenses (or potentially other spinnable materials). This mould can be used many times, without a noticeable degradation of the lenses; we have shown one to last for well over 50 spins. This turns the very slow initial milling step into a single “tooling manufacture step”, that only needs to be run once. Several moulds can be created from a single milled mould, allowing the process to be scaled, and thin-film lenses to be mass-manufactured.

We then detailed our methods of use: how we plan to use the lenses to view real-samples, including two setups that allow micrometer and piezo movement of samples with respect to our lenses. We also speak of using our lenses as coverslips, dropping samples directly onto them, or using piezo’s under a microscope to position them, and then pushing the sample into hard contact. This method is good, as it is simple and as we will show in Chapter 4, very effective.

Having described our method, we then moved on to look at the results of milling, etching and replication. We showed that etching our silicon mould does not increase its roughness by much, 4.0 nm to 4.1 nm, but the shape of the mould is changed. From this, we calculated that the lowest wavelength we could use on the dishes whilst keeping scattering due to roughness to below 1% was 51nm, deep into the UV.

When milling, redeposition of material can cause a shallower dish than initially expected (lower depth/diameter ratio); however, etching corrects this by increasing the depth/diameter ratio, leaving us closer to the expected result.

Looking at low-dose implantation effects with etching, we saw that even at the lowest dose of $10 \mu C/cm^2$ the depth of the feature dramatically increased, creating a reasonably square indent, where the depth increases and the bottom flattens as the dose increases. Using SUSPRE modelling, we calculated that a 30 minute etch in 48% hydrofluoric acid would remove all of the damaged silicon above the point at which it reached $31\pm5\%$ amorphous, independent of the dosage. From this, we concluded that, by increasing the
dosage above $3000 \, \mu C/cm^2$, the etch depth has sufficiently increased so as to remove most of the gallium, leaving less implanted then at the lowest $10 \, \mu C/cm^2$ etched samples.

Using STEM we saw the change from amorphous to crystalline silicon, and with EDX spectroscopy confirmed that the bulk of the gallium sat within the amorphous silicon region, and was removed along with it during etching.

Looking to the replicated lenses and moulds, we saw PDMS lenses replicated directly from a silicon mould with very low roughness, $0.3$ nm, and a low $\chi^2$ of $0.5$ nm.

Finally we saw that, for the 3 step replication process we use, the roughness does increase from $4.0$ nm in the original mould, up to $11.7$ nm for the final PMMA lenses. Whilst the roughness does increase, it is not enough to cause problems with the lenses’ optical properties, having a $1\%$ total integrated scatter at a wavelength of $147$ nm.

This chapter demonstrates we can make high quality, bespoke micro-optics with a replication technique. In the next chapter we will be using these lenses to view samples, quantifying how effective they are at being lenses.
Chapter 4

Micro-Optic Imaging

Summary
Micro-sphere super-resolution imaging techniques are a recently discovered field, as discussed in our literature review Chapter 1.2.1.4. During the previous chapter, we described how to manufacture thin-films of micro-lenses on a similar scale to the spheres used in the super-resolution technique. In this chapter, we characterise our lenses, discussing whether they can be used to image beyond the diffraction limit. We begin looking at micro-spheres, testing whether we can replicate the results seen by others on Blu-ray discs. We then move on to look at an ion-milled chequerboard and a *lacy carbon grid* using lenses, discussing how moving the lens relative to the sample and microscope objective changes the magnification. We resolve a 200 nm, diffraction limited feature, before moving on to look at lenses on a Blu-ray disc, discussing the lens diameter and thickness, as well as the wavelength and polarisation of the light in relation to sub-wavelength imaging. We show some confocal 3D scans through our micro-spheres and lenses, before ending the chapter looking at biological samples.

4.1 Introduction

As technology shrinks in size, for example transistors reaching 14 nm [126], there is a growing need for simple methods for both viewing and directing light at the small-scale. Current industrial methods for viewing fine-structure involve electron-microscopes, which are large and expensive to purchase.
When viewing with an optical microscope, the resolution limitation is the *diffraction limit*, which we discussed in detail in Chapter 2.2.7. Rayleigh’s criterion defined that the resolution limit, $d_R$ was based on the *numerical aperture* (NA) of the lens. We will restate his equation here for ease of reference:

$$d_R = \frac{0.61 \lambda}{NA} = \frac{0.61 \lambda}{n \sin(\theta)} \quad (4.1)$$

This shows the wavelength and NA of the lens control the minimum resolvable feature size. The numerical aperture is defined by the index of refraction of the surrounding medium, $n$, and the gathering angle, $\theta$. In air, the value of $n \approx 1$, and with the maximum gathering angles approaching $72^\circ$, we get a maximum NA of 0.95. In white light, where the deepest blue component can have a wavelength of 400 nm, this sets the resolution limit at 257 nm, in air.

Imaging in water ($n = 1.33$) or silicon oil ($n = 1.336 - 1.582$) features can be resolved down to 154 nm, however full liquid immersion is required.

Super-resolution optical imaging techniques allow this limit to be circumvented, in both air and liquid immersion. We discussed in detail a set of micro-sphere based methods, in which a 1-10 $\mu$m SiO$_2$ sphere is placed on a textured surface. The spherical lens magnifies the fine-structure, making it visible under a standard optical microscope (discussed Section 1.2.1.4). Features as small as 50 nm (8x magnification) have been viewed under a conventional microscope using this method.[5]

In the previous chapter we developed a lens manufacturing technique that allows fast ($\sim$2 min) production of thin-films of polymer micro-lenses of a similar size to the micro-spheres. During this chapter, we look at whether these lenses are able to replicate the super-resolution magnifying ability of micro-spheres. Due to sitting on a thin film, it makes the lenses positionable relative to a sample, making them much more usable than a micro-sphere based nano-imaging method.

We will characterise our lenses, looking at small (50-300 nm) pitch samples, including the Blu-ray disc. The disc has a combined pitch of 320 nm, which is at the diffraction limit for green ($\lambda = 500$ nm) light. The individual ridges of the Blu-ray are 120 nm troughs, with 200 nm ridges, each well below the diffraction limit of white light.

As we highlighted in our introduction, super-resolution imaging is a huge area in biology, with the fluorescent technique *STED* taking the nobel prize in 2014. Label-free super-resolution is the current holy grail of biological imaging, so we end the chapter by looking at some basic biological samples, including bacteria and yeast, one unlabelled and one labelled.
In this next section we aim to thoroughly investigate the ability of microspheres and microlenses to magnify near and below the diffraction limit. We specifically show interest in how near-field micro-optics increase the contrast of an imaged substrate, and what that may mean to sub-diffraction limited imaging.

**4.2 Micro sphere Imaging**

We begin by looking at micro-spheres, replicating the effect seen in previous papers [5][49], after which we move to look at early images taken using our micro-lenses.

Images were taken of 3.2µm diameter glass spheres on the surface of a Blu-ray disc under a standard optical microscope (Zeiss Axiophot). Our earliest images are shown in Figure 4.1, which shows the a) optical and b) SEM image of a set of micro-spheres. In the optical image a pair of lines are clearly visible within each of the spheres, which appear to be an image of the Blu-Ray lines. Looking to the SEM image, we see that the lines travel in the same direction, where any difference is likely due to the slight change in imaging angle. Measuring the optical image, the centre-to-centre spacing of the two lines average 600±25 nm. Dividing this by the real spacing of the Blu-ray (320 nm, confirmed in the SEM), gives a measure of the micro-sphere’s magnification power, at 1.9±0.1x. As the trough is 120 nm in size and is clearly resolved, this shows at least λ/3 resolution.

![Figure 4.1](image)

**Figure 4.1:** Images of 3.2µm silica micro-spheres sitting upon a Blu-ray disc. a) shows an SEM image, confirming the presents of optically unresolvable lines. b) shows an optical image, clearly resolving the 120 nm trough between the ridges. Magnification is measured to be 1.9±0.1x.

To confirm the effect additional images of micro-spheres on Blu-ray were taken using an alternative optical microscope (Leica DM2500). The figure below, 4.2, shows images of
the Blu-ray ridges. The main image is of an area with several groups of scattered 3.2\(\mu\)m spheres on a Blu-ray disc. The ridges are just barely visible in some of the top-right spheres.

Inset 1 (top-left) shows an enhanced image of some of the spheres, where the contrast has been amplified to the maximum. The lines are clearer, but still lack contrast. This suggests the component of light scattering from the Blu-ray discs is very weak compared with the bulk-illumination. Inset 2 (middle-right) shows a further set of spheres that have been contrast enhanced, as well as put through a Fourier band-pass filter, selecting frequencies corresponding to the imaged Blu-ray. This has amplified the lines in the image, but adds complexity to image processing.

The difference in image quality between the microscopes is largely due to the state of the two microscopes, and serves to show how a well treated microscope (Zeiss) gives a much higher quality image than one that has not been well maintained (Leica).

![Figure 4.2: Optical image of microspheres on a Blu-ray disc, as taken by a poorly maintained microscope. Central image is overview, with no lines clearly visible. Top left inset has been contrast enhanced and faint Blu-ray ridges are visible within the spheres. Middle right inset has been further contrast enhanced, and red lines have been added to indicate the direction and position of the lines within the sphere.](image)

To find the super-magnified image, we began with the microscope focused above the top of the sphere. The microscope focus was then lowered through the sphere (by raising the sample in the z-direction). In both the case of the micro-spheres and the micro-lenses, which we will discuss later, the images are formed when the microscope is focused below...
the surface of the sample. This is caused by the light through the sphere/lens having a longer path distance through a slower medium (higher refractive index). This puts the virtual imaging plane of the sphere below the focal point of the surface.

The viewed sample had been prepared by dropping a given concentration of microspheres onto a carefully stripped Blu-ray sample. The concentration of spheres was varied by mixing the pure suspension, as bought from the supplier (BangLabs), with de-ionised water at 1:10, 1:100 and 1:1000 parts suspension to water. Micro-litre droplets were pipetted onto the disc. As anticipated, increasing the ratio of the water whilst reducing the droplet size was an effective method of reducing agglomeration of microspheres, allowing individual spheres to be found and viewed. This was done to allow us to rule out interference effects caused by multiple spheres.

Attempts were made to drop spheres onto ion-milled structures with a 50 nm pitch, however accurately dropping spheres on top of a small structure was found impossible, demonstrating the biggest limitation of the spheres.

We have seen the magnification effect described by Wang et. al.[5] and others in our literature review (1.2.1.4), and confirmed that the lower-end of the claimed magnification (∼2x) is achievable even with a poorly maintained microscope. With this boost in magnification above that of a regular microscope, it can readily reach the diffraction limit and view ∼200 nm objects. With a firm grasp of what is attainable with microspheres we will begin looking at our manufactured lenses using similar methods.

### 4.3 Magnification & Early Images

Whilst micro-spheres are an interesting method for sub-diffraction viewing, they have several draw-backs which we aim to overcome using micro-lenses. These include the inability to move or position the spheres with respect to a sample and the lack of control over the optics (lens diameter and profile are easily modified by our method). To show that our lenses are not only comparable, but superior, we need to demonstrate that our lenses can magnify sufficiently to beat the diffraction limit; that the lenses are movable with respect to the substrate; and that they are usable with both dry-material and wet-biological samples. We begin by looking at the lenses ability to magnify large samples.

Figure 4.3 shows a chequerboard pattern that has been ion-milled into a piece of gold-coated glass. The image was taken in the first of our two rigs (discussed in Chapter 3.2.13), allowing precise control of the x,y and z position of the sample. The pattern itself is made up of 5µm squares, each made up of 250 nm spaced lines that run either
horizontally or vertically through the square, causing the change in hue from light to dark.

Images 4.3.b, c, d and e) are of a square in the pattern being moved upwards, towards a 10µm lens (position indicated by the yellow circle). We can see that the position and size of the square changes as it moves, increasing in magnification from 1.4x to 1.7x (±0.1x) as we push through focus. Whilst accurate information on the exact height of the sample as it moved was not obtained (due to a lack of interferometric readers in the Piezo stack), the trend is an increasing magnification as the sample drew closer to the lens.

From our discussion of plano-convex lenses in Chapter 2.2.3, we know that the magnification increases as the sample moves towards the focal point. The super-resolution effect is only seen when viewing a virtual image, that is, when the sample is closer to the lens than the focal point. As the magnification increased as the sample was moved towards the lens, we can deduce we were not in the correct area for super-resolution imaging.

![Figure 4.3: Optical image of an ion-milled chequerboard. Each 5µm square is made of sub-50 nm lines separated by 250nm gaps, with the lines alternating in direction between the squares. Images b-e) show a 10µm lens sitting over a square. Moving from b) to e) the distance between the lens and sample is decreased, increasing the apparent magnification from 1.4x to 1.6x.](image-url)
Figure 4.4 shows a visual comparison of a magnified lacy carbon grid with an SEM image of the same grid. The grid was viewed under an 150x microscope objective (Zeiss, apochromat, NA 0.95). A lacy carbon grid is a thin film (∼100 nm) of carbon covered with holes, held by a copper grid. They are commonly used in transmission electron microscopy to hold small samples, such as micro- or nano-particles. To confirm the area imaged in the SEM was the same as the optical microscope, the area was registered with respect to the corner of the copper grid surrounding the lacy carbon (visible in Figure 4.4 & Figure 4.6), and a measurement was made from the corner of the square to the area of interest.

In Figure 4.4 a) we show a close up of a lens magnifying the holes in a film, next to b) an SEM image of the same region. The scale bar in the optical image is 10µm, the same size as the lens diameter. Subjective image measurements suggest a 1.2-2.5x optical gain, with the smallest resolved distance just over 200 nm. The optical image is visibly blue, caused by over compensation of colour balancing by the camera, due to the yellow tint of the light from the tungsten filament lamp. The visible holes have been numbered within the SEM image for reference, with the hole numbered 1 alone in the very centre of the lens. Similar holes to those visible are present at the edge of the image, but are only visible where an adjacent lens falls over them.

Whilst the subjective (by hand) measurements of the material between the holes places the maximum magnification at 2.5x, a more accurate measurement can be found by taking a profile from the centre of the pairs of holes, and measuring the full-width at half-maxima to get the distance, along with the contrast. Figure 4.4 c) shows a graph of the FWHM magnification and contrast taken from profiles between the numbered holes, plotted against the real measured distance between the holes. Rayleigh’s criterion is indicated for both green and yellow light, with the appropriately coloured lines.

The most striking feature is the magnification, which is almost double the previous estimate, at just under 5x. The lowest magnifications, below 3.0±0.1x, include only a single hole spacing of less than 0.3µm, whilst all longer distances are similarly low. Over all, the contrast is low, peaking at 0.25 (equating to the intensity of the trough being $\frac{2}{3}$ of the peak intensity), and dipping below 0.1. This was after artificially boosting the contrast of the image using software (ImageJ,[127]), suggest only a very small amount of light scattered from the small structure is collected.

Looking at the trend of both the magnification and contrast, below a hole-spacing of 0.3µm (near the Rayleigh criterion), they peak and trough at similar values. This indicates that the magnification is entirely dependant on the contrast available within the image. Above the Rayleigh criterion, > 0.3µm, the trend appears to change, with the magnification reducing with the dramatic increase in contrast. This strongly suggests
Figure 4.4: Lacy Carbon Grid imaged through a micro-lens. a) Optical image, b) SEM image, with numbered holes. c) shows the apparent magnification (distance in optical image divided by that measured in SEM), as well as the contrast for the various spacings between numbered hole pairs. Optical image scale-bar 10µm

In diffraction limited imaging, the contrast and magnification are controlled by the same factors.

Within the image, the smallest hole spacing was between holes 2 and 3, measured as
95 nm. The material between the two holes was not visible in the optical image, and the two had merged, even with careful analysis of a profile taken over the hole. This sets our lower viewing limit within this image at 199 nm, seen between holes 2 and 6. Referencing back to Chapter 2.2.7 where we defined the diffraction limit, 199 nm is below the minimum resolution of blue/green light (\(\lambda = 475\) nm), which is 305 nm for the Rayleigh limit, and still below the Sparrow limit of 205 nm. As a reminder from our previous discussion, the Sparrow limit is only conventionally applied to point sources, such as fluorescent molecules or distant stars. Resolving objects of such sizes through the eye-piece (as was achieved here) is not usually possible. This shows that diffraction limited imaging can be achieved with our film of parabolic lenses.

Our lenses appear comparable in ability to the reflowed polystyrene spheres discussed by Vlad et. al. [49] who, looking back to Chapter 1.2.1.4, magnified 180 nm gaps to between 1.4-2.2x with hemispherical reflowed spheres. Whilst our measurement of the FWHM magnification is significantly higher than theirs, the subjectively measured values, of 1.2-2.5x, is much more comparable with their findings. Our lenses however, can be repositioned and their geometry is bespoke and uniform.

Moving on to Figure 4.5 we show images taken moving through the focus of the microscope. The optical images show a zoomed out view of a section of lacy-carbon grid, as magnified through 10 \(\mu\)m lenses. The distance between the lens and LCG was kept fixed, held by the first of our two rigs discussed in Chapter 3 whilst the setup was moved relative to the microscope objective (150x, NA 0.95). The lens discussed in Figure 4.4 can be seen on the bottom row, second to the right.

Unfortunately, as the microscope stage used did not have a reliable micrometre stage we were unable to quantitatively measure the change the change in height, and can therefore only form a qualitative argument from these images.

The images show 5 steps in moving through focus, beginning with a), focusing above the carbon-film surface, at the level of the lenses. b)-d) move through the lenses, focus at the LCG surface, and then just below. The final image e) is noticeably below the LCG surface, which can be seen by how out of focus the periphery of the image is. The holes in the carbon film show a clear progression as we move from a)-e), first coming into focus, before expanding, whilst fading. The graph shows the apparent increase in magnification, measured between holes 1 and 2 (as labelled in Figure 4.4). The inner and outer denotes whether it was the distance measured between the closest points of the holes (inner), or the further edges of the holes (outer). There is a definite increasing trend in both as we move down through the sample, although there is some distortion in the images where the inner and outer values move apart then back together. Optically
speaking, moving the objective lens in relation to the micro-lens will change the focal length of the micro-lens, similar to what was seen in micro-spheres [60]. We know from our discussion of optics in Chapter 2.2.3 that the magnification of a virtual image, formed when a plano-convex lenses focal length is longer than the lens/object separation, decreases as the focal point moves further from the object (deeper into the surface in our case).

The increasing trend in magnification, as we move the microscope focus down through
the sample, suggests that the focal point of the micro-lens is being moved upwards, towards the surface. As there is a limit to how much the microscope can affect the micro-lens focal length, it suggests a short focal length micro-lens, in close proximity, is the ideal setup.

The next image, Figure 4.6, shows a lower magnification image of a LCG, taken under a 20x magnification lens (Zeiss, A-plan, NA0.4). Due to the lower numerical aperture, the resolution limit for the lens, under blue light($\lambda$ 400 nm), is 610 nm, above the size of the wavelength.

4.6 a) displays an overview of a full set of 3/5/7/10 $\mu$m lenses suspended over a LCG. Whilst the holes in the carbon themselves are generally too small to see, where lenses are present the holes in the carbon film are magnified, becoming more clearly visible and in-focus. 4.6 b) and c) show an expanded view of some of the 7$\mu$m lenses along with an SEM image of the region viewed. Measuring one of the holes visible in both images, we see that a 950 nm feature has been magnified to 2$\mu$m, an increase of 2.2±0.1x. We can calculate the wavelength for which 950 nm is the smallest resolvable feature, which for the lens is 623 nm, or deep red. This indicated that a greater magnification gain was achieved by the lenses under a lower power microscope.

![Figure 4.6 showing images of a lacy carbon grid taken at low magnification (20x lens). A 950 nm feature can be seen to have magnified to 2$\mu$m, showing a 2.2±0.1x magnification](image)
To verify the observed effect, further measurements were made on another microscope, using a variety of lens powers. Using a Keyance microscope (VHX-5000 series, NA~0.82, Rayleigh criterion at $\lambda$=400 nm is 298 nm), images were taken of imperfections seen on a copper grid using the equivalent of 20, 30, 40 and 50x microscope objective lenses. Sets of images where the microscope objective magnification were taken through three micro-lenses of varying lens thickness.

Figure 4.7 graphs the magnification increase caused by the micro-lens against the absolute magnification of the observing microscope. Each of the three micro-lenses used were 10$\mu$m in diameter, but varied in depth with the deepest at 2.5$\mu$m, the mid value 1.25$\mu$m and the shallowest at 1$\mu$m deep. In each case, the trend is for the micro-lens magnification to decrease as the microscope magnification increases. The thickest of the three lenses, with the shortest focal length (10$\mu$m according to Equation 2.17), shows a significant increase in magnification over the thinner two.

From this observation we have the basis for two premises: Firstly, that thicker micro-lenses give a higher magnifying power, and secondly, that increasing the objective power actually reduces the gain of using a micro-lens. As we touched on with Figure 4.5, the maximum magnification is achieved when the focal point of the lens is just below the sample surface. This means that thicker lenses, with shorter focal lengths, give a greater magnification that a shallow lens with a long focus. It is likely that the change in gathering angle between a low and high powered lens has a similar effect on the micro-lenses focal-length to moving through the focus. A low-powered (smaller gathering angle) lens moves the focal point nearer the surface, and hence gets a greater magnification.

Due to thicker lenses appearing to give better magnifying properties, we would suggest any further work carried out with micro-lenses to use significantly deeper lenses than we have here. In Section 4.6, we look at the focusing properties of the lenses by confocal microscopy alongside computer simulation, to get an idea of how deep a lens needs to be to minimise its focal length.

It is worth noting that this particular work, in which we viewed a single object with multiple lenses, was only made possible by the fine piezo positioning available when using rig #1, which allowed us to move our lenses relative to a flat sample. Had we attempted to perform a similar experiment with a number of micro-spheres of varying diameter, micro-manipulation of multiple spheres would have made this work unfeasible.

So far in this chapter we have looked at micro-sphere magnification, replicating the results of other journal papers. We then demonstrated that thin-films of micro-lenses can magnify on the small scale, even bypassing the diffraction limit. In the next section
Figure 4.7: Graph of change in micro-lens magnification with change in microscope magnification. Three micro-lenses of increasing thickness were imaged at varying microscope objective power. The graph demonstrates that increasing the microscope magnification decreases the magnification seen within the micro-lens. The inset images show the pair of dots imaged without (top) and with (bottom) a micro-lens. The lines in the graph correspond to microlenses of thickness: 2.5µm (black squares), 1.25µm (red circles) & 1.0µm (blue triangles).

we return to the Blu-ray standard with microlenses to examine the variation in lens diameter, looking at how this affects magnification and contrast.

4.4 Super-lens Images

We now move to look at a sample of microlenses in direct contact with the ridges on a Blu-ray disc. A small square of Blu-ray disc was placed into a holder, then a set of lenses were aligned under a microscope and slowly lowered onto the sample.

Figure 4.8 shows four images showing the Blu-ray tracks as seen through sets of a) 3µm, b) 5µm, c) 7µm & d) 10µm lenses. In each image the tracks, seen as sets of dark lines, are only visible within the area of the lens.

As there is a 700 nm PMMA layer covering the entire surface (upon which the lenses are suspended) we can rule out the super-resolution effect coming purely from a thin-layer interference effect. Hence it must come from the focusing caused by the shape of the lenses. This lets us know that, whilst the ideal shape is still unknown, shape and curvature of the lens are important.
Looking between the various lenses in Figure 4.8, it can be seen that the number of lines visible within the lenses does not increase linearly with the diameter. The larger diameter lenses show more lines per-micron than the smaller lenses. This suggests that whilst the field of view of the largest diameter is bigger, the magnification of the smaller lenses is greater. According to our observations so far, this also suggests a higher contrast from the smaller diameter lenses.

For each lens diameter, lenses of various thickness were available, from 1/4 of the diameter, down to 50 nm thick. Profiles were taken across a number of lenses for each diameter to measure the average Blu-ray line pitch. The magnification and contrast of two different $f\#$ lenses is shown in Figure 4.9. The $f\#$ given is not the true $f\#$ of each of the lenses, which was not understood at the time the micro-lens mould was manufactured, but is that of the parabolic reflective dish that produces that lens. Theory suggests that lenses made from moulds of similar $f\#$ should be comparable, but we discuss whether this holds true in Section 4.6.
Comparing the diameters by $f\#$, the magnification decreases with an increase in the diameter in both cases. The decreasing magnification is not so surprising, as for a given $f\#$ the focal length of the larger lenses is pushed further into the surface. This would agree nicely with our current theory, that the position of the focal point below the surface is a controlling factor. The plot of the contrast shows that, the higher magnification comes with higher contrast, as the contrast trends both follow the downward slope of the previous graph, as well as having higher contrast in the lower $f\#$ lenses. Our previous section looking at lacy carbon grids also suggested that, for structures at the diffraction limit, the magnification and contrast are both strongly linked.

If we consider the trend of a decrease in contrast with respect to the $f\#$ of the lens, a decrease in $f\#$ (gathering power) appears to decrease the contrast. However the trend, where a smaller diameter gives a higher contrast, follows a similar pattern to the magnification.

Currently, we do not know if the trend of the contrast following the magnification is correlation or causation. If we suppose, for a moment, that the magnification of the micro-optical element controls the contrast, then it follows that it is a controlling factor in the minimum resolution. We know that the focal position below the surface is important to the magnification factor, hence a shorter focal length (thicker) lens, or a lens sitting upon a pedestal would be the ideal to maximise resolution.

As we discussed in Chapter 2.2.9, the smallest features are formed by the largest values of the wavevector, $k_x$ & $k_y$. When the combination of these values goes above the total wavevector $k$, the propagating wavevector $k_z$ becomes imaginary, and hence the wave decays. If the magnification is what allows the evanescent waves to be converted into propagating waves, by decreasing the size of $k_x$ & $k_y$, it must increase the image size to within the resolution limit of the microscope. This would allow us to derive an equation.
for the minimum magnification, $M$, required to resolve a specific feature of height $h_o$, at a given wavelength, $\lambda$, by combining Equation 2.4 (from Chapter 2.2.1) with 2.21:

$$M > \frac{0.61\lambda}{h_o NA}$$

(4.2)

This means that for the total pitch of a Blu-ray, as viewed by a 0.95 NA lens under green light (500 nm, $d_R=321$ nm) the lens needs to magnify by only 1.003x, very little. It suggests the lens serves more to overcome the microscope flaws than the diffraction limit. To consider the individual tracks clearly resolved requires a 1.6x magnification (for the ridges), or a 2.6x magnification (for the troughs). Under a yellow hue of illuminating light (as we have used), where $\lambda \approx 580$ nm this would require a higher magnification of 1.16x for the Blu-ray pitch (which we have achieved), but 3.1x to consider the smallest feature resolved.

As our average magnification is at around 1.2-1.3x, it suggests that only the overall 320 nm pitch is being resolved, but measurements of the individual features that create that pitch would not hold up to scrutiny. If we applied this same calculation to the smallest resolution features seen under white light, by Wang et al. [5], they saw 50 nm features, which would require a 6.4x magnification to resolve. As they demonstrated an 8x magnification, this can be considered clearly resolved.

The next logical step would be to look at the design parameters of the ideal micro-lens. Firstly, the evanescent wave needs to enter the higher refractive index medium as soon as possible, before they decay, so a curved bottom to the lens or sphere actually reduces the range of wavevectors that can be saved. The higher refractive index of the lens allows the evanescent wave to propagate further before decaying. Secondly, the distance from the entrance point (bottom of the lens) to the magnifying curved profile is important. The wave is still decaying within the lens until it reaches this interface, and hence a shorter distance allows the smallest length waves, which decay the fastest, to reach the interface. Thirdly, the total magnification power of the top surface controls the absolute limit of the resolution.

As a higher degree of curvature gives a greater magnification, when combined with the first and second points it indicates we want a small diameter, deep lens, which agrees well with our results so far. We also need to minimise the sample/focal-point distance, and hence sitting the lens on a small post, tuned to minimise distance but maximise magnification would be ideal.

Figure 4.10 shows the control variables over the lenses optical properties that are available to us, due to our manufacturing process. By controlling the lens diameter, thickness and curvature we can control the absolute magnification of the lens, maximising our
imaging power. We can then vary the pillar thickness (if present) or film thickness to place the focal point just below the sample surface. This also has the benefit of control over the propagation distance of the evanescent wave to the magnifying (curved) surface.

4.4.1 Wavelength dependence

Figure 4.11 shows 3µm lenses magnifying a Blu-ray disc through a) a green filter and b) a blue filter. Image a) is a good demonstration of the increased resolution produced by these lenses. The smallest feature size visible by a conventional microscope under 500 nm wavelength light is 321 nm according to Rayleigh, or 215 nm according to Sparrow (See Chapter 2.2.7 for a full description). As the visible features of the Blu-ray are ~200 nm and ~120 nm, this puts each individual feature below the Starling limit, and the combination at the Rayleigh limit. Due to the area surrounding surrounding the lens being bare, it again shows the feature cannot otherwise be resolved under blue or green light.

The image under blue light is nearly identical, with clear lines visible within the lenses. The blue light ($\lambda \approx 450$ nm) is Rayleigh limited to 289 nm, with the Sparrow limit at around 194 nm. This still leaves the individual components of the feature below the Rayleigh limit, although the overall size is very slightly above.

The magnification is still 1.3±0.1x, suggesting the individual features are not being clearly resolved, only the over-all pitch can be considered as resolved.
Figure 4.11: 3\(\mu\)m lenses atop a Blu-ray disc under a) green light and b) blue light. The lines seen within the lens are the ridges of the Blu-ray disk. Due to being diffraction limited, they cannot be seen outside of the area of the lens. Insets are expanded images of the lowest lens in each image.

We have discussed how the diameter, thickness and objective microscope power affect the magnifying ability of the lenses. We have also looked at how the colour of the light affects the image. Next we move on to investigate another property of the light, the polarisation.

4.4.2 Angular Polarisation Dependance

Now we move on to look at the effect of polarisation on the image. When a single polariser is introduced between the sample and eyepiece (or camera) and rotated, we see a definable change in the contrast of the lines. Figure 4.12 a)-s) show an image series of a 3\(\mu\)m lens as the polariser is rotated, in steps of 20\(^\circ\). Looking along the images, the most notable changes are a darkening of the overall image, along with an apparent thickening of the dark-rim of the lens. The lines within the lens darken as we move to image h), causing them to appear to thicken, before they quickly fade again in image i)-n). From o)-s) the lines return to their original contrast.

Figure 4.13 a) is a graph of the contrast seen in the images in Figure 4.12, plotted against the polariser angle, along with the peak intensity seen within the lens. A large double-peak can be seen between 100-220\(^\circ\), which is around the angle of the Blu-ray grating (indicated at \(\sim 155^\circ\) by a blue line on the graph).

If we consider the nature of the sample, the lines on a Blu-ray form a regular grating, which has a polarising effect on the light reflected from it.

The large peak we see in the centre of the contrast plot, 4.13.a), is reached when the polariser angle is the same as the angle of the surface grating. The fact it is a split peak, with the lowest point at the angle of the grating suggests light is scattered most
Figure 4.12: Image series of a 3µm diameter lens as a polariser is rotated. Each image shows a 20° step in the 360° rotation. Whilst the overall transmission through the lens drops off for e-k, the contrast appears increased.
at angles just off of the grating angle. This could be an effect of the curvature of the lens.

When compared with the second line in the figure, we see that the contrast increases with a decrease in peak intensity. This means that, whilst less light is travelling through the lens, the peaks are becoming more distinct, as the background light is being removed.

The second figure in Figure 4.13, b), shows the peak position of the (nominally four) Blu-ray ridges visible within the lens, as a function of angle. The central two lines move very little with respect to one another, but do move up together, with respect to the lens edge, over the first 100°. This suggests a stable magnification within the centre of the lens as the polarisation is changed.

The bottom line (black-squared), corresponding to the left ridge in the image, varies greatly in distance from the central two with polariser angle. This suggests that changing polarisation distorts the edge of the image.

The data for the top line (green-downwards triangles), the right-most ridge in the image, is discontinuous, due to the ridge disappearing from the image in several cases. Where it is continuous, it to moves nearer and farther from the central lines, almost mirroring the movement of the black-square line. This suggests that the image distorts in a continuous manner with the change in polarisation.

Moving on to look at the effect of cross polarisation, the image below, Figure 4.14 shows two images of hexagonally close-packed hexagonal lenses a) without a polariser and b) with a pair of crossed polarisers. In 4.14 a) the Blu-ray ridges can be seen within each of the lenses. Due to the sample not sitting perfectly planar under the microscope, the transition from the top-left of the image to bottom right shows a variation in focus. The inset shows an expanded view of seven lenses, with the ridges clearly visible within them.

It is interesting to again note the work by Vlad et. al.[49]. They saw that three close packed spheres could act as a single lens, which they named a trimer. Our close-packed lenses appear too widely-spaced to act in this fashion, with a 500 nm gap between them. It would be possible, in future work, to test how the spacing of the lenses affected the trimer like quality of combining focus.

Figure 4.14 b) shows an identical image taken with a set of crossed polarisers in the microscope; one between light source and sample, and the second between sample and eyepiece. We note that the contrast of the lines has increased, with them becoming clearer at every level of focus across the image. The inset shows the same set of lenses as before. Note that we can now see distortion of the lines within the lens. Looking carefully at the central lens seen in the inset of b), there appears to be a slight warping of the lines toward the south-west. Whilst all seven lenses are (in an ideal world) identical, the warping of the lines indicates they are not identical in profile. As AFM
Figure 4.13: Shows a) a graph of the contrast in the image a certain polariser rotation, and b) the position of the 4 peaks apparent in the images. Whilst the outer two peaks (top green triangles and bottom black squares respectively) show significant movement and occasionally disappear altogether, the inner two peaks (blue triangles and red circles) are positionally stable over the course of rotation.

has previously confirmed that the milled dishes, moulds and similar PMMA lenses are identical in profile (Chapter 3.3.1 & 3.3.6.2), suggesting this uneven distortion is caused by warping in the supporting film, due to the stress induced by direct sample contact.

Comparing 4.14 a) and b) the higher contrast is clear. Numerically, the contrast with crossed polarisers is 1.98 times greater, which can be explained, as follows: the incident
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Figure 4.14: Images of 3µm hexagonal close-packed lenses taken with a single, and then a pair of polarisers. The polarisers in the second image were at 90° from one another, so that only light altered in polarisation by the sample was viewed. The insets show expanded images of the same set of hexagonal close packed lenses viewed without (top) and with (bottom) a second polariser. The contrast is higher with two polarisers in place.

Looking at the polarisation of light seen through a micro-lens, we have shown how the edges of the lenses can distort images, and how stress on the supporting PMMA film itself can distort the lens profile itself. A single polariser introduced into an image has the maximum effect on increasing contrast when in-line with the surface grating, whilst
crossed-polarisers gives the largest possible contrast boost. Next, we use a different method of using light to image the lenses, using a confocal microscope.

4.5 Confocal Scanning

Taking the discussion away from polarisation we move on to 3D confocal laser-scanning microscopy. A confocal scanning microscope uses very narrow depth of field optical lenses to image narrow sections through the depth of a sample, building a 3D image, layer-by-layer. A set of \textit{confocally positioned apertures} are used to remove high angle light, minimising the point spread function, and taking the microscope to the diffraction limit in $x,y$ and $z$. For these lenses, it effectively can map the intensity of the light rays propagating light rays through them.

4.5.1 Micro-sphere

Using such a microscope (Zeiss Z2, 50x objective 0.95NA, $\lambda=405$ nm) an image was built of a micro-sphere (Bangs lab, $3.2\mu$m av. diameter) sitting on a Blu-ray disc. Image 4.15 a) is a single xy-slice of an individual sphere showing the ridges of a Blu-ray. The profile taken across the sphere (yellow line) is shown in 4.15 b) where three peaks are clearly visible within the area of the sphere, and four (smaller) peaks are visible outside. Measuring across the three central peaks, we get a distance of $1.316\mu$m equating to a magnification of $2.1\pm0.1x$. The small peaks surrounding the sphere in the profile are the Airy rings, created by the sphere acting as a circular aperture, which visibly surround the sphere in image a).

Image 4.15 c) shows an xz-slice through the the sphere parallel to the yellow profile line in a). Looking to the surface at the edges of the image, indicated by a red arrow, we can see texture, where the confocal microscope is just able to pick up the hint of the ridges. In the centre of the image we see above the surface a bright area where lines are emanating from the surface. This is the reflected image of the surface as seen through the sphere. The ridges are visible, seen as the straight lines emanating directly upwards, and marked with a green arrow. The lines are surrounded by the Airy pattern formed by the sphere aperture, which spread moving away from the sphere. The drop-off in intensity of the Blu-ray image lines suggests that the sharpness of the image drops off as the distance from the sphere increases. Below the surface of the disc is the back focal plane of the sphere, indicated with a yellow arrow. Around this region we again see the ridges of the disc, however magnified to a lesser extent.
Figure 4.15: 3.2μm diameter silica sphere on a blue ray, taken by a confocal scanning microscope (405nm wavelength). a) shows slice 85, at the imaging plane of the sphere (Above the sample) showing 2x magnified lines, as confirmed by the profile in b). c) shows an XZ cross section, where the back and front focal imaging planes can clearly be seen. The red arrow indicates the top of the Blu-ray surface, the green arrow shows the top imaging plane of the sphere, whilst the yellow highlights the back focal plane. The red ellipse and blue sphere suggest the position of the sphere adjusting for the refractive index in z-only (red) and xyz (blue).

4.5.2 Confocal Micro-Lenses

Replacing the spheres with a set of lenses, in Figure 4.16 we see five lenses of 3μm diameter, each marked with an apparent magnification seen through the lens. The magnification increases moving from left to right from 1.3-1.8x getting close to that seen through the micro-sphere. The thickness of the lenses varies as we move along the set, increasing in steps of 50 nm from 0.55μm up to 0.75μm (left to right). Whilst this
may account for the change in magnification, looking at the surface we see that there is a change in focus across the surface, suggesting the sample was not perfectly flat. As we saw with the lenses suspended above a lacy-carbon grid, in image 4.5, changing the microscope focus, with respect to a fixed lens/sample distance, alters the apparent magnification. This focal change, along with the increasing lens depth combine to form the increase in magnification.

![Figure 4.16: Confocal scan of Blu-ray disc taken through a set of lenses. The lenses (increase in depth), and show variation in the magnification.](image)

Figure 4.17 shows a single 3µm lens with a) xy-image b) xy-profile, and c) xz-image in a similar form to Figure 4.15. 4.17 a) shows the magnifying abilities of the small lens, with good contrast highlighting the ridges. The wider peaks (200nm wide) are bright, whilst the troughs (120 nm) are dark. As the magnification increases, the difference in size between the bright and dark lines becomes clearer, allowing them to be more clearly resolved. This fits with our earlier assumption, that magnification is the key to sub-diffraction viewing.

The profile taken re-enforces the good contrast achieved by a 3µm lens, with five clearly defined peaks on show. Measuring across them gives a distance of 1.706µm or a magnification of 1.3±0.1x. A re-measurement across the central 3 peaks gives a higher magnification, 1.5±0.1x, indicating again that there is distortion at the edge of the lens pulling the two edge lines inwards.

Both of these values for magnification are lower than the average we see with the micro-spheres, however, as was indicated by our discussion of Figure 4.7, the lens thickness is significantly lower than that of a sphere. This leads to the focal point being pushed farther into the surface of the sample, lowering the magnifying power of the lens, as well as reducing the image sharpness. It is likely that a thicker 3µm lens, possibly on a small pedestal to mimic the reflowed spheres of Vlad et. al.[49] would increase the magnification.

The xz image shown is not nearly as clear as that of the microsphere, due to a much coarser step height used. However the ridges of the surface can be distinguished where the lens is present, and a focal point below the surface can also be identified.
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4.6 Computational Modelling of Lenses

We begin with a figure showing the MEEP simulations of 3-10µm lenses, suspended on a 1µm film. Figure 4.18 shows the contour plots of four lenses, their diameters marked in red. Each lens shown was chosen for having a theoretical f# of 1, as calculated from our derived plano-convex formula, Equation 2.16 in Chapter 2.2.4. For the sake of easy reference we will restate Equation 2.16 here:

\[ f = \frac{2fd}{(n-1)} \]  

The four images show that for every lens size, light is concentrated to a thin, but long focal spot. In imaging terms, this suggests good xy resolution, with a long depth of field. In all four images, the light passing through the lens strongly interferes before creating the spot, seen most clearly in the 10µm diameter lens.

To test our derivation of the plano-parabolic lens equation, Equation 4.3, a further 20 models were run, for varying lens thicknesses of the different diameters. The meep-simulation was run at a similar wavelength to the 3D confocal laser (405 nm), for a glass-like substance \( (n = 1.5) \), and a film-thickness of 1µm. The measurement of the focal length from each model is shown, in Figure 4.18 b)-e). Each plot shows the lens focal lens, \( f \), against the focal length of the same depth reflective dish, \( f_D \).

Equation 4.3 indicates that for a refractive index of \( n = 1.5 \), the relationship between the two should be \( f = 4f_D \), which has been plotted as red circles in each graph. The black square shows the measurements of \( f \) from the MEEP-simulations. Two lines have been fitted to the data, a blue parabolic fit, and a green linear fit that excludes data we
believe to be bad. The bad data comes from the focal point coming into close contact with the rear PML layer, in some cases cutting the end of the spot off, and changing the profile of the focal spot, moving it back towards the lens.

In the 3, 5 and 10µm lenses, the parabolic fitting is a very good match of the data, however the green line suggests that a linear fit is also a good match for the shorter values of \( f_D \) (deeper lenses). In every case, the data suggests a longer focal length than Equation 4.3 gives. The slope of the linear fitting gives the relationship between \( f \) and \( f_D \), which ranges from 8.4, for the 3µm lenses, up to 11.8 for the 7µm lenses. This is 2-3x greater than expected.

To get a better understanding of our findings, a 3D confocal scan was taken through a set of 10µm lenses, of 1.3-2.5µm thickness, as an experimental comparison sample. XZ-scan images have then been compared to MEEP-simulated models of identical lenses, shown in Figure 4.19. The experimental data in a) - c) show lenses with \( f_D \) of 5, 3.75 and 2.5 respectively, and are matched to the top three MEEP-simulated lenses shown in d).

At a glance, the focal length of the MEEP-simulated lenses appears significantly longer than the experimental data suggesting an inaccuracy in the model. Table 4.1 shows the values for the theoretical, simulated and experimental focal lengths. The theory and the experiment agree, suggesting our derivation of an equivalent lens-maker equation for parabolic lenses, Equation 4.3, was correct. However, as we noted from the images, the simulated focal length is 1.5-2x higher than the expected value. Looking back to our models in Figure 4.18, we saw similar trends at 2-3 times the value.

This suggests an error lead to our values being incorrect. Having carefully checked through the control file (shown in Appendix A.3.1) human error in defining the geometry or light source have been ruled out. As we showed excellent results in Chapter 5, which we corroborated in Chapter 4, this leaves us with an unknown. It is possible that our definition of the focal length was not correct, or somehow that the boundary conditions interfered with the process of the simulation. However, further experimentation, beyond the scope of this project, would be needed to confirm exactly why.

Looking at 4.19 a)-c) it appears that the back focal point (below the lens) gets shorter compared to the front focal point, as the thickness of the lens is increased. This is due to the form of measurement. As light is the measurement tool and the confocal microscope assumes the medium is always air, it does not compensate for the lens material, causing it to measure a shorter distance than would otherwise be the case.
Figure 4.18: Lens focal length plotted against the focal lens of parabolic dish of identical profile, for diameters of 3μm, 5μm, 7μm and 10μm lenses. Red lines show the theoretically calculated values ($f = 4f_d$), whilst the blue line is a polynomial fit of the data. The green line is a linear fit, in which possible bad data points have been excluded. This bad data is caused by interference of the back PML layer.
4.7 Biological Imaging

A major aim of most super-resolution techniques is to allow viewing of subcellar biology and viruses. As we highlighted in the literature review, improvements in biological imaging can lead to huge leaps forward in medical understanding, and so any improvement in microscopy would have very definite impact in the biology and medical sciences.
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Table 4.1: Table of 10µm diameter lens data. The lens depth and dish focus $f_D$, are shown along with the theoretical, simulated and experimentally measured focal lengths of such lenses, where $n = 1.497$. The theoretical result from Equation 4.3 is closest to the experimental result, suggesting an inaccuracy in the model. The back $f$ is the back focal length, the focal length measured below the lens.

Currently the smallest resolved biological feature, imaged using micro-spheres, are of 75nm adenoviruses [47].

The final images we show in this section are of biological samples. Whilst our images are not of such small structures, they begin to indicate the potential for super-resolution biological imaging. We have imaged very simple, safe samples to show how our lenses could, in principle, be used in such areas.

The first of the two figures is 4.20 where a *Streptococcus thermophilus* bacterial chain, suspended in water can be seen a) next to and b) beneath a set of micro-lenses. In this instance, to separate out the bacteria, yoghurt was centrifuged at 6000rpm for 2 minutes, separating solid from liquid. A small amount (~50µl) of liquid was taken from the yoghurt and dropped directly onto the back face of a lens film. A 5mm glass cover-slip was then placed on the back of the lens film, held on by the capillary force. In the pair of images shown in Figure 4.20 the bacteria swam under the lenses allowing the second image to be taken. Measuring across the width of each cocci (section within the chain) and comparing with & without the lens we get an average magnification of 1.2±0.1x, just slightly lower than what we achieved viewing Blu-ray tracks.

Two important facts about this image to consider are that the bacteria was live, and locomoted itself under the lens, and that it was unstained and yet still clearly visible. The imaging micro-lens has a diameter of 10µm and a depth of only 0.8µm. As calculated using Equation 4.3, this gives a focal length of 31.25µm, a long focal length when discussing the area of near-field optics. Relating this to an ($f#$), this puts the lens at around $f# = 3.1$. In terms of the focal position of the object compared to the lens, the bacteria must have been very close compared with the long focal length, minimising the magnification power. Our greatest magnification in this chapter was achieved with lenses of much smaller ($f#$) (closer to 0.25).

The final image in this chapter is 4.21, showing a yeast (Saccharomyces cerevisiae, Alinsons) next to and then under a lens. The image was taken using rig #1, allowing the sample to be moved around below the lens. The image demonstrates a 1.4±0.1x
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Figure 4.20: Images of a *Streptococcus thermophilus* bacterial chain, extracted from yoghurt. a) shows the bacteria free from the lenses, whilst b) shows the bacteria having moved itself underneath the lenses. A 1.2±0.1x magnification is seen through the lenses, due to the long focal length of the lens.

The yeast was prepared by fixing it to a 5 mm cover slip (for use in Rig 1, as described in Chapter 3) and staining with crystal violet. As the cell was attached to the glass cover-slip, our 3-axis piezo stack was able to freely move the sample below the lenses. The fine positioning of the piezo motors allowed the cell to be positioned centrally below the cell, an impossible feat to achieve with micro-spheres.

If we examine the cell image outside and within the lens, the external wall of cell appears sharper within the lens. Whilst no internal structure is visible, this is due to using a wide-spectrum stain, staining the surface wall, rather than the internal structure.

Returning to discuss the magnification, the lens had a diameter of 10µm with a depth of 2.2µm, giving a lens f# of 0.56, around twice that of the deepest lenses we made. Comparing it to the magnification of similar lenses shown in the graph of Figure 4.7, also viewed under a 5000x objective, the 1.4±0.1x magnification sits about where we would expect it between the 1µm and 2.5µm depth lens.

Overall this image serves to demonstrate firstly, the magnifying and image sharpening ability of the lenses, and secondly the ability to control the sample below the lenses.
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Figure 4.21: Images of a yeast cell being moved beneath a micro-lens. The scale bar is 10µm. The yeast was stained with crystal violet, and shows 1.4±0.1x magnification.

4.8 Conclusion

In this chapter we have looked at super-resolution imaging using micro-lenses and spheres. There is a need for imaging beyond the diffraction limit of light in biology, and many other areas. We demonstrate that a thin-film of micron-scale lenses can be used to image sub-wavelength structures.

During this chapter we compared the imaging ability of 3µm diameter silica spheres with polymer micro-lenses of 1-10µm diameter. We saw a 1.9±0.1x magnification from the micro-spheres, with a poorer quality microscope giving much lower imaging contrast.

Comparing with similar Blu-ray samples imaged using micro-lenses, we saw on average a much lower magnification, between to 1.1-1.5x. Whilst the super-resolution effect was seen, the magnification was notably lower through the lenses in every case. We came to the conclusion that the lens thickness, and lens/sample distance were playing a role in limiting the magnification.

Imaging alternative samples with micro-lenses, such as lacy-carbon grids that had holes spaced as little as 95 nm apart, we saw that the 95 nm gap could not be resolved, but a 200 nm gap could be. This gave us a lower-limit to the resolution of our micro-lenses.

We also saw a trend in both the lacy-carbon and Blu-ray images that suggested that, when imaging diffraction limited samples, the magnification and contrast where either causally or correlatively linked. Above the diffraction limit, an increase in magnification comes at a loss of contrast, however we saw the opposite case in our images. We put forth
a speculative argument, based on the hypothesis that the magnification controlled the contrast. We assumed that due to the magnification of the lens reducing the size of the wavevector, that the magnification itself was allowing light to turn from an evanescent into a propagating form. The stronger the ability of the lens to magnify, the larger the wavevectors that can become propagating, and hence more light is available within the image, causing the increase in contrast. This also means the magnification controls the minimum resolvable feature.

Based on this understanding, and the evidence of our experiments, we suggested that thicker lenses, having a shorter focal length and higher magnification, would make the ideal lens. By controlling the lens/sample distance by controlling the film thickness, we could maximise our magnification, to hit peak resolution.

Using confocal microscopy we confirmed our derivation of Equation 2.16 in Chapter 2.2.4, although our computational models did not agree due to an unknown error.

Finally, we imaged two, biological samples, one using Peizo positioning and the second in a simple cover-slip setup, immersed in water, to show our lenses are viable for biological imaging. We demonstrated a $1.2 \pm 0.1x$ and a $1.4 \pm 0.1x$ magnification of biological samples, although sub-wavelength features were not visible due to sample preparation.

Summarising this chapter, we have shown that our fabricated micro-lenses can do a similar job to a micro-sphere, and allow us to image sub diffraction limited samples using white-light. Due to being fixed to a thin film, the lenses are manoeuvrable and samples can be scanned in both x, y and z. The magnification shown in this chapter is generally between 1.2-2.5x, but higher magnifications should be achievable by increasing the lens thickness, increasing the film thickness and by improving the relative control of the focal point below the substrate. Our bespoke manufacturing method gives us control over all of these properties, and should allow us to maximise resolution. The smallest feature we have resolved is $\sim 200$ nm, around $2/3^{rd}$s of the Rayleigh criterion for the illuminating wavelength.

Having discussed our super-resolution lenses in detail our next chapters move on to a slightly different area of optics. We look into nano-jet forming curved profile micro-axicon lenses, used to form micron scale Bessel beams. Again these have a potential application in biology, used in optical tweezing and scanning techniques.
Chapter 5

Nano-jet Computational Modelling

Summary
In this chapter, we continue our computational modelling introduced in Chapter 2, and used in Chapter 4 to model lenses. We introduce introducing a new structure designed to form nano-jets, high-aspect, non-diffracting beams of intense light. We use computer models to demonstrate how changing the trough profile alters the beam length, intensity and width. We also look at how the width of the structures apex, as well as the refractive index of the medium change the beam properties. We discuss how offsetting the trough profile either side of the centre allows the beam to be deflected, showing control to $\pm 5^\circ$ of deflection, before finally discussing how the beam forms. To end the chapter we demonstrate a possible use for the structure, by simulating coupling between a fibre-optic cable and a waveguide. The Bessel-beam forming design shown within this chapter is subject to a patent application (number GB1415763.0).

5.1 Introduction
During our work on micro-lenses, and their use in super-resolution, we began experimenting with the idea of using micro-lenses in photolithography. As lenses focus light down to a very small spot, the question arose whether a lens, as part of a lithography mask, could be used to produce sub-wavelength patterns. This would allow much smaller features to be mass produced, without necessitating the complex and expensive
extreme-UV systems currently used by companies such as Intel, to reach their smallest feature sizes\cite{128}. Conventional UV photolithography is used to mass produce most of the world’s electronic goods, due to its ability to quickly produce small-scale structures. Integrating sub-wavelength features would allow smaller features to be produced without reducing the wavelength, a process that introduces as many difficulties as it solves.

When we planned how to produce micro-optics for photolithography, we considered the plano-concave lens. We could produce such a lens by directly milling a trough, or dish into photolithographic mask. During this discussion, we failed to account for the effect of the high-to-low refractive index, causing a beam passed through such a lens to become divergent. This lead to the opposite effect to what we anticipated. Whilst we expected a focal-spot below the centre of the plano-concave lenses, we instead observed that much of the light was collected between the sets of plano-concave lenses.

When tested photolithographically, which we will discuss in Chapter 6, we found interesting features in the photoresist, other than those expected from the regular focusing effect of a lens or a reflecting-dish. This lead to us computationally modelling the masks using the software MEEP (see description in Chapter 2), discovering an interesting effect, known as nano-jetting.

We first touched on the idea of such a jet in our literature review, Chapter 1.2.1.4, where nano-jet emission had been described\cite{57} and observed\cite{129} from micro-spheres. Such microsphere nano-jets have since been demonstrated for use in photolithography\cite{130}. A nano-jet is a region of high-intensity collimated light where the length is significantly longer than the width. For visible light such a jet usually has a full-width half-maxima (FWHM) of a few hundred nanometres, often less than the illuminating wavelength. The \textit{diffraction length} of the jet, as described by Devilez et al. can be a few microns long when coming from a sphere \cite{131}. We term this quantity the \textit{full length at half maximum} (FLHM).

In this chapter, we use computational modelling to describe how nano-jets with width similar to the wavelength, but a length of 16 or more microns can be produced. We look at 2D \textit{finite-difference time-domain} (FDTD) models of how light propagates through micron scale troughs, changing the dimensions of the troughs to find how the intensity, FWHM and FLHM are affected. This work will be further discussed in Chapter 6 where we experimentally test our structures, and compare them to our modelling.
5.2 Notation

Before starting let us define the basic quantities and shape of the optical feature we will be modelling.

Figure 5.1 shows the basic layout of what we will be describing. The white region is considered air or vacuum (assumed to be n=1.0), whilst the dark blue is a higher refractive index, usually mimicking glass or plastic with a refractive index $n \approx 1.5$.

The modelled troughs are parabolic in profile, unless specified otherwise. The depth and diameter of the plano-concave lenses use the same notation as the dishes discussed in Chapter 2.2.2, as indicated in Figure 5.1.

When modelling this structure we have stayed with a size we can make using the FIB, keeping the diameter at 3$\mu$m, and the depth to between 0.2-3$\mu$m.

What we denote the pitch is the centre to centre spacing of the troughs which, for a given trough diameter, sets the size of the apex. Due to the comparison with SNOM, an internal-reflection based technique, in Section 5.10 we discuss how much of the light in the jet is contributed by the light transmitted through the apex. We demonstrate that light is contributed by the full width of the pitch.

The small gold-coloured areas directly below the aperture show the position of a metal film (when present), usually 100 nm thick and, in the simulation, made of silver (Ag). In the real structures we test in Chapter 6 we use gold (Au), chromium (Cr) and palladium (Pd).

Note that the structure is simulated in two-dimensions. In three-dimensions it would be representative of a set of close pack troughs, elongated out of the page. Such a structure would form an extended light-sheet, rather than a single beam. We test exactly such structures throughout Chapter 6.

The alternative for beam-forming would be a rotationally symmetric structure centered on the apex, to which the simulation should also be relevant. This would form a single light-beam. Whilst the models are applicable to either of the structures mentioned, we will refer to the concave region as a trough for the purpose of our discussion.

The red, green and blue beams show the nano-jets under varying wavelength illumination. They are which are usually sub-micron in width, but few, or sometimes few-tens, of microns long.

In their paper, Devilez et al. define a nano-jet with four basic quantities, starting with the focal-length\textsuperscript{[131]}. During our discussion it becomes clear that, whilst our jets are similar to nano-jets from spheres, they are actually created by the interference between two, and can not truly be said to be focused. Hence we will be using the term Peak
Intensity Length (PIL) to describe the distance between the structures apex and the beam’s peak intensity.

The second quantity he describes is the intensity enhancement at the peak intensity position. As all figures for intensity we give are normalised to the intensity of the input Gaussian beam, we shall just refer to this as the intensity. The third quantity is the full-width at half-maxima (FWHM) of the beam, taken from the y-axis profile at the peak intensity position. Finally, the diffraction length, as was termed by Devilez, describes the full-length at half-maxima of the beam (FLHM), as taken from the x-profile through the peak intensity position. For preference, we will be calling this the FLHM.

The nano-jets we highlight in the diagram are what we refer to as the primary or zeroth-order beams, however other, weaker effects will be discussed which we refer to as first, second or third order effects.

**Figure 5.1:** Diagram of jet forming structure, highlighting the notation we use when discussing it. The illumination of the entire pitch region contributes to jet formation.

Moving on to the modelling, we will start by looking at the effect of the shape of the trough, looking at whether it is a diffractive effect or not, and then move on to how various parameters of the trough (curvature, depth, etc) affects the jet.
5.3 Effect of Curvature

Figure 5.2 shows a set of 2D contour plots, made in MEEP similarly to those of the lenses in section 4.6. The images model a glass photolithography mask (n=1.5), with 100 nm silver coating, that has had three 3µm diameter troughs of 0.75µm depth milled into it, with an 100 nm apex.

The mask is illuminated with 350(±100) nm light (with assumed zero absorption at the propagating wavelength) from left to right. Figure 5.2 a) shows such a set up. Blue colouration indicates a low concentration of light, whilst red, and deep red, indicates very high light concentration.

We see in 5.2 a) that the highest concentration of light is not, as we originally expected, at the focal point of the lens-like-troughs, but in the area between the troughs. The curved shape of the trough, along with the high-to-low refractive index change appears to have guided the incoming light to concentrate below the the 100 nm apex. The light forms an optical nano-jet, a very thin, long region of high-optical intensity that turns-on a few hundred nanometres from the surface, but extends for over 10µm.

One very important point to take from the image is that the very strong optical-jets are forming below a thin-metal layer. Whilst the metal thickness is only 100 nm, this would make it almost opaque to look upon. This could make such an optical feature useful in situations where guided light is needed below a conductive layer, such as in camera CCD and CMOS chips, as well as solar-cells.

Our original belief was that this was an effect of light-guidance down the sub-wavelength feature, making this structure more akin to a SNOM like approach for nano-optics (as discussed in 1.2.1.3). However, as we will describe in Section 5.11, we now understand that the light is being concentrated by the curved surfaces either side of the apex, in a setup more akin to an axicon lens. These are used to form a Bessel beam, which we covered in our literature review, Chapter 2.2.10. These are beams formed by interference effects, and have several novel properties, including the ability to self-heal, which in our case allows the beam to traverse a metal cap on the apex.

Figure 5.2 b) shows an identical set-up, without a metal coating. Strong nano-jets indicate the presence of the metal is not needed to form the jet, showing it is not a plasmonic effect. In both 5.2 a) and b) we can still see the presence of the central focal-point of the three troughs (seen as yellow higher-intensity regions following right along the y-axis from the centre of each trough). Before modelling, we had anticipated that these focal points would be the regions of highest optical intensity. It is clear that the centre focal point is still of higher intensity than the surrounding region, but nowhere
near the intensity of the jets. The top and bottom focal points are both weaker and appear deflected and have merged into higher order effects emanating from the region between the troughs. These focal points are the result of the structure acting like a grating. Comparing it to 5.2 e) in which we have a square grating set in the glass, we see similar points, although they are positionally shifted in 5.2 a) and b) by the presence of the nano-jets.

Comparing the intensity of the jets a) with and b) without metal, the setup without any metal has a very slightly higher intensity, 2.943 compared with 2.940, a 0.1\% difference. This change is tiny, and immediately suggests very little light is passing through apex.

Figure 5.2 c) shows a mask with a continuous metal layer, stretching across the milled troughs and leaving an air gap. Whilst the nano-jets are still present, their intensity drops by 35\% compared to structure without metal, to 1.908. Strong, but smaller, reflected nano-jets can be seen emitting backwards, into the glass mask, with an intensity of 2.909, showing where the lost light has gone. Later, in Section 5.10 we will test opening an hole in the metal at the apex, looking at how light emanating from the trough-area contributes to the nano-jet intensity.

Figure 5.2 parts d) and e) show a metal-only grating, and a square grating (with metal layer) being tested. As neither of them show any nano-jet type effect, we can attribute that most of the effect comes from the shape of the glass/air interface. 5.2 f) shows a triangular grating, giving an approximation between the parabolic troughs, and the square grating. Strong jets are visible, meaning that a triangular shape is enough to cause the light-guiding effect. However, the length of the jets is decreased, compared with Figure 5.2 a) or b). What we now understand is that two flat plates, in such a setup as Figure 5.2 f), are known to create a Bessel beam, and is commonly called an axicon lens. We discussed the principles of a Bessel beam in the literature review, Chapter 2.2.10, and will further discuss them throughout this chapter, including Section 5.11, when we discuss the theory of how our structure works.

With confirmation that the shape of the glass/air interface is the key to the light-guiding effect, we will continue by looking first at how the depth of the trough affects the Intensity, FLHM, FWHM and PIL of the jets. We will then move on to look at how the profile controls the beam properties by varying the depth of the trough, as well as examining what effect the size of the apex (varying the pitch) and wavelength play. We will then move on to looking at the effect of offsetting the depth of the troughs with respect to one another, in which we discover we can steer the angle of the beam. Then we examine how light passes through the system by introducing metal-slits to block light. Finally we will have a brief look at modelling our structure in a real world system, for light coupling into a waveguide.
It is worth noting that, due to the large amounts of data needing fitting/extracting, a series of Matlab scripts were written to extract much of this data, the details of which are in Appendix A.3.

5.4 Effect of profile change through varying depth

To ascertain the effect of a changing profile on the jets, the depth of the trough was varied. Models where run on a set of three, 3µm diameter troughs spaced 100 nm apart with a 100 nm metal (Ag) layer on top, varying the depth of the troughs between 0.1875µm to 3.0µm. Figure 5.3 shows the contour plots of the 0.1857, 0.75, 1.25 and 2.5µm deep troughs.

As a qualitative look at the data, the plots show some very useful information. The 0.1875µm troughs have two long, wide jets of relatively low intensity, peaking around 12µm from the structure. The 0.75µm deep troughs show strong, very long jets, where their peak intensity radiates for 10µm. The 1.25µm depth trough has more intense peaks then any other depth, but that intensity appears to radiate for a shorter distance than the 0.75µm trough. The 2.5µm deep trough appears to radiate very little from
the aperture, with the highest light intensity found trapped at the glass/metal interface between the troughs. We highlight why this happens when we discuss the theory in Section 5.11, noting how the angle of curvature changes the refraction/reflection of the light.

Figure 5.3: Contour plots for glass (n=1.5) troughs of various depths, including: a) 0.1875\(\mu\)m b) 0.75\(\mu\)m c) 1.25\(\mu\)m and d) 2.5\(\mu\)m. Illumination wavelength was 350±100 nm.

Figure 5.4 holds four plots of the trough depth against a) Peak Intensity, b) Full Length at Half Maxima (FLHM), c) Peak Intensity Length (PIL) and d) Full Width at Half Maxima (FWHM).

The intensity/depth graph, a), shows that a depth of 1.25\(\mu\)m gives the best performance in a 3\(\mu\)m diameter trough. Beyond 1.25\(\mu\)m the peak intensity drops off again. A secondary, smaller peak can be seen at the 2.5\(\mu\)m mark. The secondary peak is caused by there being very little jetting in the 2.0\(\mu\)m and 3.0\(\mu\)m depths. In both cases the light drops off from the metal interface, and almost no peaks are seen beyond that point. For this reason, their focal lengths are close to 0, as the measured intensity is at the peak of the radiation, next to the metal. In the 2.5\(\mu\)m trough the nano-jet intensity is still significant, with a distinct peak beyond the metal that reaches a higher intensity than the light emanating from the metal itself. To get the most out of these light-jets, tuning
the depth of the trough structures to maximize the peak intensity and nano-jet length would be useful.

The FLHM and PIL show very similar patterns, a drop off from $0.375 \mu m$ down to $2.0 \mu m$, with a sudden spike at $2.5 \mu m$. Together they tell us that the peak intensity region grows shorter and nearer to the metal film as the depth increases. In the case of the depths below $1.5 \mu m$, the drop off can be said to follow an increasing peak intensity. This suggests more of the light is being focused into a single, small area, with a higher drop off rate. The very longest jets, $0.1875-0.375 \mu m$, both reached the maximum length our model could show, of $17 \mu m$.

The small peak seen at a depth of $2.5 \mu m$ is difficult to explain. In general, below $2.0 \mu m$, there is a drop in intensity in the x-profile just after the metal layer at the tip of the apex, before a sudden rise to the peak intensity. At both $2.0$ and $3.0 \mu m$ the light intensity drops off from the apex metal, and then never rises again, whilst the $2.5 \mu m$ does increase to a peak. Whilst we cannot explain exactly why this happens, this at least explains why we have a peak at $2.5 \mu m$.

The case of the FWHM is interesting, where the $0.1875 \mu m$ trough has a much wider beam than the average of around $0.5 \mu m$. Looking at the cross-sectional profile of the data, seen in Figure 5.5 b), it appears the $0.1875 \mu m$ trough has a distinct peak of a regular width, with strong shoulders either side contributing to its width. They likely come from first and second order effects that haven’t fully split from the main peak. As for the FWHM of the other depths, the $1.5 \mu m$ trough has the smallest jet-width, at $225 \text{ nm}$, with the highest intensity $1.25 \mu m$ trough at a slightly wider $303 \text{ nm}$. As the structure was illuminated with $350 \text{ nm}$ wavelength light in this case, it means the nano-jets can reach widths of $2/3$rd the central illuminating wavelength. This is very promising for any area where squeezing a lot of light into a small area could be used, such as in optical communications, getting light from a few micron optical fibre into a few hundred nanometre waveguide. We’ll discuss this idea further in Section 5.12.

Below in Figure 5.5 is a plot of the transmission through the structure. The black-squared line labelled ratio-large takes into account an area the full width of the feature (three troughs and two apertures) before and after the structure. The red-circled line labelled ratio-small looks at an area the size of a single aperture in line with the aperture. As the depth of the troughs increases, the total light transmitted drops off from $0.77$ to $0.5$ (ratio-large). In this case it could be explained by a reduced area where transmission from glass to air is possible, due to the critical angle for total internal reflection. We’ll discuss this idea further in Section 5.11, where we look at the theory of what the structure is doing.
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**Figure 5.4:** Graphs showing the variation in a) peak intensity b) FLHM c) PIL and d) FWHM as the trough depth is varied. In d) the wavelength is indicated by the green line, demonstrating the FWHM drops below the wavelength between 1-2µm.

For the smaller area, the total light gathered into the area of the beam peaks at 1µm before dropping off. The progression is similar to that seen in the intensity plot in Figure 5.5, however the peak seen at 1.25µm is shifted slightly, with only a small shoulder peak in its place. This means that whilst a depth of 1.25µm gives a higher maximum intensity, at 1µm more light can be coupled through the structure. As we will discuss in Section 5.11, a deeper trough leads to a steeper curvature, more quickly reaching angles in which refraction of light is impossible, stopping the transmission of light.

### 5.5 Variation of the pitch (apex width)

Altering the size of the pitch, whilst keeping the trough diameter constant allows us to change the width of the apex. We have run models for apexes of 0µm to 3µm in width. In Figure 5.6 we show a) the peak intensity, b) FLHM, c) PIL and d) FWHM. The trough-depth was set at 1.25µm, to maximise the peak intensity, with a diameter of 3µm. The usual 100 nm of Ag was present. The illuminating wavepacket again was 350±100 nm, looking toward uses in photo-lithography.
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Figure 5.5: Transmission percentage as the trough depth increases. The absolute transmission (black line), decreases with the depth, demonstrating more light is internally reflected. The transmission into the beam area (red line) increases, to peak at a depth of 1µm before falling off. Figure b) shows the areas over which the data was normalised. The blue-line shows the starting point of the incident wave. The large ratio was normalised by dividing the large, green area seen to the right of the troughs, by the green area to the left (accounting for the area difference). The small ratio was normalised over the red areas, by dividing the right by the left. These show the total transmission through the structure (large ratio), and transmission into the beam (small ratio).

The maximum intensity is reached with a 200 nm apex, but remains consistently high until 1.5µm (5.6 a). The PIL and FLHM both increase with the apex width, pretty much linearly in the case of the PIL (which indicates the distance from structure to peak intensity). It suggests that the larger 0.5-1.5µm apertures may be ideal for real-world uses, as the peak intensity is also stable in this region. However, the FWHM increases with the apex width, indicating that increasing the jet-length also increases the width, which may mitigate the structures usefulness at large pitches. The peak-FWHM reached is 519 nm, whilst the smallest is 206 nm, all under the same 350 nm wavepacket. Below 0.5µm the FWHM repeatedly dips into the sub-wavelength region (Below 0.35µm), possibly indicating a resonance effect. If line were to be fitted between the data, rather then taken as absolute values, the trend would still take the FWHM below the wavelength, indicating the structure can form sub-wavelength width beams.

Looking to the transmission in Figure 5.7, a pattern similar to the increasing depth emerges. The ratio of total transmission through the structure drops linearly from 0.63 to 0.52, whilst the transmission in-line with the apex increases to 2.0 times, showing the light gathering power, before dropping logarithmically to 0.8.
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**Figure 5.6:** Effect on jet properties by varying the apex width. a) peak intensity, b) FLHM, c) PIL d) FWHM. The stability of the peak intensity, whilst FLHM and PIL increase, between 0.5 to 1.5 µm, suggests an ideal zone for manufacturing a structure of this diameter and depth.

**Figure 5.7:** The Transmission through the structure as a function of the apex width. The large ratio indicates total transmission, whilst the small ratio indicates transmission into the beam area. The beam area peaks at twice the incident light within the beam, for a pitch of 100-200 nm

### 5.6 Effect of Wavelength

Next we need to look at how the central wavelength of the wavepacket affects the jetting, for a given structure. Remembering back to our introduction to *meep* in Chapter 2, the
light source we use in *meep* is a Gaussian wavepacket of wavelengths (or frequencies). Whilst we quote the *wavelength* as the central wavelength used, the drop off on either side is Gaussian. For these next experiments, we use a wider-envelope wavepacket (±250 nm) as it looks at a more realistic, optical source.

The structure we use is the standard 100 nm apex, chosen to minimise the FWHM at a time before the full spectrum of apex widths had been run. The diameter is 3µm by 0.75µm deep trough. The metal is 100 nm thick silver. Note that we use an approximation of glass/plastic for the refractive index (∼1.5), but we have not modelled any dispersion, so that we may see exactly how the wavelength interacts with a structure of this size.

![Figure 5.8: Effect on jet properties by varying wavelength. a) peak intensity, b) FLHM, c) PIL & d) FWHM.](image)

Looking to the intensity plot in Figure 5.8, we immediately see the exponential drop-off in intensity above a wavelength of 300 nm. This is almost certainly caused by the ratio between the size of the structure and the wavelength decreasing. As light is incident on an object smaller than it, the effect of scatter and absorption lessen, meaning longer wavelengths don’t interact with smaller objects as strongly. If we bring the transmission into the discussion, show in Figure 5.9, we can see that the total
transmission (RatioLarge) is more or less continuous at 0.7, whilst the total light within the beam (RatioSmall) drops off with a similar exponent to the peak intensity. This indicates that transmission through the structure is unchanged by the wavelength to structure ratio. But, how the light is guided, specifically into the beam, is affected.

The FLHM increases dramatically with the wavelength up to 400 nm, before slowly decreasing, whilst the FWHM increase is largely linear, staying consistently above the wavelength. A linear FWHM is not surprising in the context of a linearly increasing wavelength. Comparing the change in FWHM from varying the wavelength to those seen in the depth (5.4.d) and apex size (5.6.d) simulations, it appears that most of the modification to the FWHM that is achievable comes from varying the structure for a given wavelength.

![Figure 5.9: Graph demonstrating the transmission of light through an area the width of the pitch (RatioLarge), and an area the width of the beam (RatioSmall), before and after the metal layer. The large ratio shows that the total light transmitted does not vary with wavelength for a given structure, whilst the small ratio shows that the light in the beam is strongly dependent on the wavelength.](https://example.com/figure-5.9)

5.7 Variation of the Refractive Index

Whilst we have looked at how the wavelength, aperture size, and the depth of the structure alter the nano-jets properties, we still need to look into an important aspect, the refractive index. Up till this point, a refractive index of $n_1 = 1.5$ has been used for the structured region, and $n_2 = 1.0$ elsewhere.

In discussions held before running these simulations, we had assumed that it was the absolute change in refractive index that defined the jet properties. To test this hypothesis, models were run for range of values of $n_1$ and $n_2$, as noted in table 5.1. The diameter and depth of the troughs were set to 3$\mu$m and 0.75$\mu$m respectively, and an illuminating wavelength of 500±250 nm was used.

The values for $n_1$ were chosen to mimic commonly used optical materials, with $n_{\text{water}} = 1.33$, $n_{\text{glass/plastic}} \approx 1.5$, and $n_{\text{BTG}} \approx 1.8 - 2.1$ (BTG = Barium Titanate Glass). The
values for $n_2$ were selected to keep the absolute change in refractive index constant at values noted in the second row of the table. For each index change, several points are mapped, excluding $\Delta n$ of 1.1, which was only run once. The key indicates the two refractive indices, $n_1$ and $n_2$, that make up the point.

We shall start by examining the peak intensity with respect to the change in refractive index, labelled a) in Figure 5.10. Each point represents a pair of refractive indices, $n_1$ and $n_2$, and have been plotted based on the refractive index change between them ($\Delta n$). The close clustering of the points suggests our hypothesis, that it is the difference in indices that control the beam properties. Fitting of the results suggests a logarithmic increase in intensity with the value of $\Delta n$. A quick look at the three other graphs shows similar clustering of the points, which seems to confirm that it is not the absolute refractive indices that matter, but the change in index that controls the gathering power of the structure.

Moving to the Full Length at Half Maxima in b), values of $\Delta n$ of 0.8 and below all have a diffraction length longer than the computational-cell, which caps out at 16.5 $\mu m$. It appears the drop-off of the intensity with distance is very slow in every case, but that of a $\Delta n$ of 1.1.

In Figure 5.11 a) we have plotted the profile along the length of the jet for every value of $\Delta n$ for which $n_1 = 1.8$. In each case, the intensity remains high for the full length, not dropping below the intensity seen to the left of the metal layer for most of its length. Simplifying slightly, we can consider each profile as having 3 peaks. Peak-one just below 10$\mu m$, peak-two near 14$\mu m$ and peak-three roughly around 20$\mu m$. As $n_2$ increases, peak-one drops off rapidly, becoming a small shoulder to peak-two. Peak-two lowers in value more slowly and moves towards the metal layer. Peak-three first increases before decreasing. Being near the back wall of the computational-cell, this peak may have contributions from light reflected off the back barrier. Whilst the rear of the cell should
be a perfectly-absorbing layer, in reality the absorption is imperfect, and some reflection may be contributing to the third peak.

If we were to look at the intensity of only peak-two, a good representative peak that appears strongly in all profiles, we would see a similar, but possibly steeper, drop off to the intensity plot in Figure 5.10 a).

![Graphs of beam intensity, length and width with varying refractive index of both structure and medium.](image)

**Figure 5.10**: Graphs of the beam intensity, length and width with varying refractive index of both structure and medium. The graphs show the a) Peak Intensity, b) FLHM, c) Peak Intensity Length and d) FWHM, for a large set of simulations, run with respect to the refractive index change between structure and surroundings. The legend shows the values of $n_1$ and $n_2$ for each point, in the form of $n_1n_2$. The close clustering of the points demonstrates our hypothesis, that the refractive index change between structure and surroundings is more important than the absolute values to forming a jet.

Moving on to Figure 5.10 c) and the PIL we see a semi-random distribution of points, clustered towards longer values for $\Delta n$ of 0.5 and below (excluding a single point at 0.5), and towards shorter values for one value of $\Delta n$ 0.5 and above. Thinking back to Figure 5.11 a) we can conclude that the longer/shorter divide comes from a difference in strength in the three peaks, changing which peak is measured. Longer values will look at peak-three, whilst shorter values look at peak-two. An interesting trend that developed is that the values for which $n_2$ is vacuum have the shortest focal length for their value of $\Delta n$. This may be an effect of the increased speed of propagation through a vacuous medium.
In a similar way to the intensity, the full-width at half-maxima, Figure 5.10 d), shows a strong trend, in this case an exponential decay. As the value of $\Delta n$ increases, the FWHM reduces. Whilst the FWHM never drops below the wavelength (0.5$\mu$m ) this is not unexpected for the depth of concavity used (see 0.75 depth, Figure 5.4). The only unusual behaviour is seen at $\Delta n = 0.1$, where the average width is around 8.5$\mu$m. Looking at Figure 5.11 b), in which y-profile (width) of the $\Delta n = 0.1$, $n_1 = 1.8$ is shown, we see that the reason for this is that the peak is no-longer well defined at half intensity. Instead the base of all central peaks have merged to form a single peak. For this reason we excluded all the values of $\Delta n = 0.1$ when creating the fitting in Figure 5.10 d) (blue-dotted line). Extrapolating the fitting back to $\Delta n = 0.1$ suggests the individual peak width would be around 2.25$\mu$m.

Knowing that the refractive index change is the key to beam formation may be an advantage in industrial applications. In our literature review, Chapter 1.2.1.4, we discussed reflow of polymers to form lenses. A simple method of producing our structure would be to reflow a low index polymer to form lenses extended along one axis, and then deposit a higher index polymer on top. This would form a simple beam-former whilst minimizing production cost and complexity. Such a product may be found useful in CMOS lenses, or solar cells, as a cheap way to maximise the light reaching sensing regions.

Figure 5.11: Figure shows profile plots along a) the x-axis (length of the beam), for $n_1 =1.8$ and all values of $\Delta n$. The position of the metal layer at the tip of the apex, and the first, second and third intensity peaks (as referred to in the text) are indicated. b) shows the y profile of only $n_1 =1.8$, $\Delta n =0.1$, where the zeroth order beam has merged with the first order, so that the FWHM appears widened to 8.5$\mu$m. We estimate a more realistic value to be around 2.6$\mu$m.

Finally, we have plotted the intensity of a summed area before and after the structure, to get an idea of the amount of light transmitted. Figure 5.12 shows the ratio of the intensity after the structure compared with before. The large-ratio is an area the full width of all 3 dishes and apertures. The small-ratios are only as wide as a single aperture. The top and bottom labels refer to them being in-line with the top or bottom aperture.
Firstly, the large-ratio shows that as the change in refractive index increases, there is a nearly-linear drop off in transmission. This agrees well with the theory, as light prefers to travel from a low to a high index, rather than from higher to lower.

For the small ratio we see that the top and bottom apertures agree perfectly, with each blue triangle matched by a red circle. As there should be perfect reflection symmetry through the centre of the cell along the x-axis, this is a useful indication that our model has run correctly, as have the data extraction routines.

The value of the small-ratio, a measure of the light within the jet, increases with the refractive index change, peaking at 0.8 before decreasing. This suggests that as the value of $\Delta n$ increases, the gathering power of the structure increases. Above $\Delta n = 0.8$, the difficulty in getting light to leave a higher index medium overcomes the increase in gathering power which, looking at the FWHM, suggests is no longer any greater. Had we more data points higher than 1.1 we may have been able to deconvolve the two progressions, and look at the strength of each effect.

One final analysis is to look at the value of the absolute refractive index of the structure, $n_1$, within the clustered sets at each $\Delta n$. In each case the progression was clear, with the lowest value of $n_1$ being either the top or the bottom point in the set, and increasing upwards or downwards in order from there.

The black arrows in the image indicate the direction of an increasing value of $n_1$ for the total transmission (ratio-large). The arrows all point up, indicating the highest absolute value of $n_1$ (2.1 in these simulations) gives the highest rate of transmission. The blue arrows indicate an increasing value of $n_1$ for light transmitted into the beam (ratio-small). When $\Delta n$ is 0.5 and below, the trend is for the lowest value of $n_1$ to give the maximum intensity within the beam, however when $\Delta n = 0.8$, this effect is reversed. This means that, in general, a higher absolute value of $n_1$ will allow more light intensity through, whilst gathering less into the beam, except for the case of $\Delta n = 0.8$.

### 5.8 Single Structure

As we have tested repeating units of nano-jet emitters, we have an idea of how they work. However in a real world application we may only want a single structure, such as in optical communications putting one on the end of a fibre to aid alignment. Further simulations have been run to look at whether a single structure works in a comparable manner to an array of structures.

To achieve this, a single protruding tip was made, with the effective curvature of a $3\mu$m diameter by $0.75\mu$m deep trough. The apex width was varied from $0.125-2.0\mu$m. In
Figure 5.12: The transmission through the structure, as a function of \( \Delta n \). The absolute intensity decreases with an increase in \( \Delta n \), whilst the intensity within the beam increases, peaks at 0.8, then decreases. This shows that beyond 0.8, the structure becomes highly internally reflecting, stopping light transmission. The arrows indicate the progression of the smallest to largest value of \( n_1 \), the structures refractive index (purple for small ratio, black for large ratio). The higher values of \( n_1 \) have a higher absolute transmission, but lower transmission into the beam.

these models we wanted to move away from thinking of this as a structure only for photolithography, and hence have used a wavelength of 500±250 nm. The contour plots of the 250 nm and 1000 nm apexes are shown in Figure 5.13. They demonstrate that the effect does not require an array of tips, a single tip will do.

Observationally, we see that as before, the thinner apex leads to a smaller jet width, whilst the jet length, and peak intensity position appear longer on the wider apex. Surrounding the central (zeroth order) jet are the first and second order jets, visible from both apex sizes. In Figure 5.14 f) is a cross section of the y-profile of the 250 nm apex jet. We can see the strong, central peak, which reaches an intensity 3 times greater than the incident gaussian wave. Surrounding the central peak are smaller peaks, decreasing in intensity as you move to higher orders. We have commented before in this chapter that our beams are "Bessel Like", in that they do not diffract over reasonably long distances (up to tens of microns). The profile of a Bessel beam (shown in Figure 2.6 from Chapter 2.2.10) follows a Bessel function of the first kind. It has been noted that in a circularly symmetric Bessel beam the energy is evenly distributed between the rings, hence the more rings that are visible, the more energy is lost from the zeroth order jet [68]. The curved surface of our structure appears to minimises the number of additional rings in the Bessel beam, placing most of the light into the core beam. This leads to the very intense beam, with small FWHM, that we tend to see.

Looking at Figure 5.14 the graphs are similar in shape to those see in Figure 5.6. This
Figure 5.13: Contour plots of a single tip structure with a) 250nm apex b) 1000nm apex

is exactly what we would expect, and shows an array is not necessary to the intensity jet. Similar to the earlier version, the peak intensity is reasonably consistent from a small to a large apex. In this case the intensity *linearly increases* from 3.1 to 4.1 (values normalised to the intensity of the incident wave). These values are similar the previous model, and the difference in the shape of the trend can be accounted for by the change in wavelength. The FLHM and PIL (Figure 5.14 b & c respectively) are similar to the previous results, shown in Figure 5.6, in both shape and value. For the FWHM, as fewer data values were used, we cannot comment on whether a similar resonance effect would be seen for the smaller apexes. However we can note that the FWHM is sub-wavelength from apexes that are of the same size as the wavelength and lower (0.5µm).

In a real world situation, a single tip may be much more useful than the arrays we had previously been discussing. In Section 5.12 we use a single structure to direct light into a simplified waveguide. Before that, we move on to looking at offsetting the depth of the troughs to direct the angle of the beam. Then we move on to introduce metal apertures in Section 5.10 to look at how the light is transmitted through the structure. Before we finish with the waveguide models, we discuss the theory of how the structure forms the beam in Section 5.11, using the evidence built up throughout the chapter.

5.9 Offset lenses and directionality

An interesting phenomenon was observed when simulating ‘offset’ trough depths. By varying the depth of two troughs of similar diameter, we found that the nano-jet deflected. Figure 5.15 a) has a diagram indicating what we mean. By increasing or decreasing the depth $c_2$ with respect to $c_1$, we can steer the jet, with it angling towards
Figure 5.14: Figure demonstrates the change in properties of a jet from a single tip, with a varying size of apex. The graphs show the a) Peak Intensity, b) FLHM, c) Peak Intensity Length, d) FWHM, e) Transmission and f) the y-cross section of the beam from a 250 nm apex. The cross section shows the first and second order jets, and show the Bessel-like property of the beam.

the shallower of the pair. When discussing our data, we assume the angle $\theta$ is positive when angling toward $c_1$.

Figure 5.16 contains the contour plots of a series where $c_1$ was held constant at 0.75$\mu$m, whilst $c_2$ was varied from 0.1875$\mu$m to 3.0$\mu$m. The apex was held at 100 nm, and the incident light was a gaussian wavepacket with central wavelength 500 nm ± 250.
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Figure 5.15: Offset troughs. a) shows a diagram, labelling the troughs as $c_1$ and $c_2$. The beam angle is considered positive when angling towards $c_1$. b) is a plot of the jet angle by $c_2/c_1$ ratio. It shows $\pm 5^\circ$ of tilt is easily achievable, but $20^\circ$ or higher is plausible.

Qualitatively reviewing the images, the shallowest value of $c_2$, seen in a), corresponds to a depth ratio $c_2/c_1$ of 0.25. It shows a reasonable deflection of the jet. Similar to the depth-test series, having a shallow dish seems to have created a very long jet with a low peak intensity, possibly caused by the increase in peak-width. When the depth ratio is 1, we are back to our normal regime, and the angle is restored. Increasing the depth ratio above 1 appears to increase the angle of the jet, whilst also reducing the length and width of the jet.

Figure 5.15 b) shows the change in angle with the ratio of the depths. The trend is linear with $\pm 5^\circ$ attainable for ratios between 0.25-2. Above this, whilst we see the trend in angle is still largely linear, we begin to compromise on length of the jet (measured in Figure 5.17 b) as well as getting strong secondary jets next to the main peak.

Bearing this in mind we move on to look at Figure 5.17, showing a) the peak intensity, b) FLHM, c) peak intensity length (PIL) and d) FWHM for varying depth-ratios. The peak intensity increases from 1.9 to 3.6 as the ratio increases. In the previous depth graphs (fig 5.4) the intensity peaked for a depth of 1.25 (which $c_2$ reaches at a ratio of 1.66) before dropping off. The reason for the constant increase past this point is that the jet starts to circumvent the metal, and emits from the dish itself. This also causes the drop in FLHM and PIL seen at these depth-ratios.

The drops in FLHM and PIL follow a very similar pattern to the depth plots, however the jet lengths seen are significantly longer in the offset lenses. The FWHM on average
is larger than the one we saw when testing the depth, some of this is accounted for by the longer wavelength, and wider band wavepacket used.

With the ability to steer the jet comes some interesting possible uses in both science and industry. In science, Bessel beams are commonly used for light tweezing and trapping [7]. Being able to accurately form a light trap off the axis of the incident light may be useful in microfluidics, for trapping small particles below a viewing window, or in optical atom traps. In industry, a device such as a solar cell may benefit from guiding light under non-transparent conductors. Whilst the Bessel beam is self-healing around small metal points at the apex, the ability to direct the light below a metal opens up the possibility of much larger conductors.

We now move on to the next section, where the look at using metal-apertures to get an idea of where the light is propagating from.
5.10 Metal Apertures

With a good idea of how a change in profile of the structure affects the properties of the beam, including intensity, FWHM and transmission, we now need a better idea of how the light travels through the structure. By introducing metal slits and covers, centered on the apex, to block the light, we can get an idea of how the light is propagating. Figure 5.18 shows a diagram of where the metal is positioned with respect to the structure when used as a slit (top) or cover (bottom). The metal used was silver, at 100 nm thick, designed to act as a reflecting layer.

Figure 5.19 shows four contour plots of a set of three 3\(\mu\)m wide troughs of depth 1.25\(\mu\)m, illuminated under 500 nm wavelength light with a wavepacket size of 500 nm. In each image, two slits have been opened in the metal, centred around the two central apexes. Figure 5.19 a) has no slits, b) has 300 nm slits, c) 1\(\mu\)m and d) 3\(\mu\)m (the diameter of a trough).

Where no apertures are present, 5.19 a), we get a continuous layer. We first showed a continuous metal layer in Figure 5.2 c), and mentioned how most of the light is reflected back into the structure, forming strong internal jets. The small amount of light that
Figure 5.18: Diagram of the position of the metal slits and covers. The jet forming structure (dark blue) has a thin metal layer (silver, 100 nm) placed in front of it to create a set aperture (slit) or a cover, of the dimension specified.

makes it through the layer does produce weak jets, but at a significant loss compared with its non-metallised counterparts. Here we will consider it alongside metal layers with slits in, discussing the peak intensity, as well as FWHM, FLHM and Peak Intensity Length.

In Figure 5.19 b) the 300 nm slits have allowed enough light through to form small, high intensity regions more similar to those we have seen before. Looking between b), c) and d) we see that as the size of the slit increase from 300 nm to 1µm , to 3µm wide, the length and width of the intense region appears to increase, as more light is allowed through at lower angles. 5.19 e) and f) are small (100 nm) and large (3.1µm ) covers that block the apex region. The small cover is similar to previous setups, with the metal exactly covering the apex, but does not have metal on the flat region, above or below the structure. The large cover blocks the width of the area indicated as the pitch in Figure 5.1. With the large cover present, powerful reflected beams can be seen within the glass region, similar to the continuous metal layer (5.19 a). Comparing f) with a), we can say that very little of the light from above or below the cover (outside of the pitch of each apex) is scattered into the area where the beams form. This indicate that the region we denote the pitch represents the extent of the aperture of our modified axicon lenses.

Moving to Figure 5.20 the a) peak intensity, b) FLHM, c) PIL, d) FWHM and e) transmission are graphed. In 5.20 a) through d) the black square data points represent an increasing aperture diameter, from 0.0µm (a continous film) to 3.0µm. The red circle data points show two points for metal covers, where the dimension represents the width
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Figure 5.19: Metal aperture contour plots, where continuous metal layer has been placed over the structure, and slits have been opened at the position of the apexes. The size of the apertures in each image is a) no slits, b) 300 nm, c) 1µm and d) 3µm. The final two images show the inverse, where metal covers of two widths have been placed over the apexes. Image e) is 100nm covers, showing the standard form we have used before, and f) has 3.1µm covers, which block the entire "pitch" region of each apex.

of the cover itself. Only 100 nm (same size as the aperture) and 3.1µm runs were made, giving a general indication the change on the individual graph.

Looking at the intensity plot in a) the maximum intensity increases as the slit opens, peaking for a 1.5µm slit, around half the diameter of the troughs. That the intensity drops off past 1.5 micron suggests some diffractive element may have come into play scattering additional light into the path of the jet, enhancing its intensity. As the slit size is increased beyond half the pitch of the two troughs, the diffraction from the slit reduces, whilst the additional light allowed through is no longer at an angle to re-enforce the beam, hence the drop off in intensity. The plot for the cover indicates that increasing the amount of metal blocking the light reduces the intensity, as would be expected.

The full-length at half-maximum intensity (FLHM) shows an increasing trend for a aperture larger than 300 nm. Both the smaller 'slits' (0 and 100 nm) are longer due to having such low intensities that the drop off rate is low, creating much longer jets. The covered variant shows only a small change from around 8µm to around 5µm as the
Figure 5.20: Graphs of metal slit and cover runs. a) Peak jet intensity, b) Full Length at Half Maximum intensity, c) Peak Intensity Length, d) Full Width at Half Maximum intensity, e) transmission through the sample

metal diameter increases. However, no comment can be made on the trend as a peak or trough in the middle is possible.

The peak intensity length increases from zero, for the 500 nm slits, up to 3.75µm for the largest slits. This indicates the peak intensity is moving away from the features surface as more light is allowed through. The cause of this may be that with the opening of the aperture, higher angled light is allowed to reinforce the beam from the surrounding region. This would enhance the beams length, as well as pushing back the peak intensity, both of which we see in our graphs.
The FWHM for a continuous film (0.0\(\mu\)m slits) is large, suffering from a similar merging of the peaks seen in Figure 5.11 b). For the 100 nm to 1.5\(\mu\)m slits the width stays below the wavelength in size, bottoming out at 285 nm. Only for the largest slit does this increase above the wavelength. In a physical sense, this is because as light is only allowed through the aperture at certain angles, the width of the jet is controlled.

In this case the version with the metal cover is not sub-wavelength at either end of the dimensional spectrum.

The final part of Figure 5.20 shows the transmission through the structure, as a function of the slit or cover size. The two lines titled large ratio refer to ratio of light for the full size of the feature (all three troughs plus two apertures). The small ratio lines show the ratio of light before and after the structure in line with the apexes, and at the width of the apex (100 nm).

There is an increase in transmission caused by increasing the size of the aperture in the metal. Whilst the overall transmission through the sample increases as we open holes in the metal, the amount of light found within the jet-region increases much faster than the overall increase in transmission, showing that the gathering power of the structure comes into play as the apertures open up.

These models have given us a strong insight into the mechanism forming these long nano-jets. Introducing apertures that cut out high angled light reduces the beam length considerably, and introducing an aperture of the right diameter enhances the signal due to diffractive effects. Next we will use this to discuss why the geometry works to form an extended beam.

### 5.11 Theory

Having run a large number of models, we can now discuss the theory behind how the beams are formed. In Figure 5.21 is a diagram of the path of rays passing through the structure. Whilst ray tracing is not entirely accurate for near-wavelength scale geometries, due to ignoring diffractive effects, it can still give an idea as to how light may propagate.

The structure appears to cause the light passing on either side of the apex to converge and constructively interfere above the apex, forming a beam. This is similar to how a Bessel beam is formed[132]. A Bessel beam is a non-diffractive beam of light that in profile follows a Bessel function of the first kind. Normally they are formed using an axicon lens, a triangular lens similar to that seen in Figure 5.2 f). We have discussed
them in some depth in Chapter 2.2.10, but will highlight the important points again here.

1. The beam does not diffract
2. It is formed by interference
3. It is self healing, and will reform around an obstruction
4. The beam energy is distributed evenly between the central and surrounding rings.

Although an ideal Bessel beam requires a theoretically infinite amount of energy, experimental examples of quasi-Bessel have been created using axicon lenses illuminated by wave-fronts of a Gaussian profile.

Unlike in a standard axicon lens, where the interfaces are flat, the structure we have investigated is made from two curved profile interfaces. This changes the behaviour of the beam from that of a standard Bessel beam. We saw in Figure 5.2 how f) a standard axicon lens compared with b) our curved profile plano-concave lenses. Adding curvature shifted the peak intensity closer towards the structure, whilst also elongating and thinning the beam.

To form a true approximation to a Bessel beam using a standard axicon lens requires the incident wavefront to be Gaussian in profile. Whilst we have not tested a setup with such an incident wavefront, we can surmise from the comparison between Figure 5.2 b) and f), that our structure would form such a Bessel beam, and that due to its unique structure it may be thinner and more tune-able by modifying the profile.

If we look at the y-profile of a single curved-axicon, such as we showed in Section 5.8 for the jets intensity, it has a similar form to a zeroeth order Bessel function of the first kind, $J_0(x)$. Whilst peaks surrounding the central peak are evenly spaced, as would be expected in a Bessel function, the intensity within each surrounding peak is below the background, suggesting a greater percentage of the light is being channelled into the central (zeroeth order) peak.

To begin to relate the data we have gathered to how the light propagates through the structure, we first need to mention Snell’s law for refraction.

$$n_1 \sin \theta_1 = n_2 \sin \theta_2$$  \hspace{1cm} (5.1)

In the case where $n_1 > n_2$ at a certain value of $\theta_1$, $\theta_2$ increases above $90^\circ$, meaning all light internally reflects. This angle is known as the critical angle $\theta_c$, and for glass in air is at around $42^\circ$ from the surface normal.
Looking back to Figure 5.21 we can see that the yellow line near the centre of the trough, where the angle between the incident ray and the surface normal is small, the ray is refracted into the path of the beam.

As the slope progresses towards the apex, the ray/normal angle increases until it reaches the critical angle. The orange ray is internally reflected towards the opposite trough where, due to the angle, it will either refract, but not enter the path of the beam, or internally reflect again back towards the direction it came.

Moving further towards the centre, the red beam again internally reflects from the incident trough, but is then able to reflect from the second trough back into the path of the beam. As the light emerges from the aperture of the apex, it will diffract due to the scale, which may give an indication of why we saw so little light from the apex contributing to the beam (Section 5.10).

This gives us three ways light can react to our structure, the first and third forming the beam and the second losing light. From our models where we varied the trough depth in Section 5.4, we saw that as the trough got deeper, the rate of change of the angle of the profile increases, leading to a smaller area which refracts, and a larger reflecting region that will scatter light outside of the beam. We saw this as reduction of the transmission when increasing the depth of the troughs.

As we saw from the models in which we varied the refractive index, in Section 5.7, the absolute difference in refractive index, between the structure and the surrounding medium ($\Delta n$), controlled the beam properties. As the geometry was kept constant, this means that the variations in the beam’s length, width and intensity, were caused entirely by the difference in refractive indices. Whilst the peak intensity increased with refractive index change, and the FLHM stayed high for all values below $\Delta n = 1.1$, the absolute transmission dropped off linearly with an increasing refractive index difference. This is caused by a reduction in the value of the critical angle as $\Delta n$ increases. This means that more light is internally reflected and lost (orange rays in Figure 5.21) meaning more light is reflected rather than refracted.

In order to best exploit our structure, the scattering region of the trough must be minimised, whilst controlling the rest of the profile to maximise intensity, jet length and minimise jet width.

Finally, having discussed the theory behind how the jets form, we look at a simple model of a real world example, that of light emanating from an optical fibre into a silicon waveguide.
Chapter 5. *Nano-jet Modelling*

Figure 5.21: Ray diagram of the optical structure. Yellow rays indicate refracted light, orange rays are internally reflected and scattered, whilst red rays follow the path of total-internal reflection that re-enters the jet path. As the red and yellow rays overlap the light is concentrated in an elongated, non-diffracting region.

5.12 Waveguide Example

Silicon photonics is an area of research involving guiding longer than optical wavelengths (1.1µm +) through silicon, a region of the electromagnetic spectrum silicon is transparent to (n≤3.5,k≈0). It has applications in optical communications and possibly in the future of quantum computing. Whilst manufacturing optical chips is a process becoming more and more refined, one big issue in silicon photonics is getting light onto and off of the chips [133]. The current procedure involves carefully aligning an optical fibre (5µm inner core and 200µm outer cladding diameters) to the very small (200 nm) waveguide. This is a difficult procedure, and involves some trial and error looking for a peak in the signal as the relatively large fiber is moved next to the waveguide. Current coupling solutions are often complex in the manufacturing and alignment phases.

We suggest using our structure to increase the amount of signal entering the waveguide. The structure could either be milled directly onto the waveguide (during test stages), or for large scale manufacturing to be photolithographically made as part of the waveguide optical chip. This would give a larger area to align to. Figure 5.22 shows a perfectly
aligned optical fibre butted against a 200nm waveguide. In a) the optical fibre is unaltered, whilst in b) our structure has been attached. The wavelength of illumination is 1.5\( \mu \)m, with a 200 nm packet width. The silicon waveguide has a realistic refractive index for the wavelength, whilst the optical fibre is a 3.5\( \mu \)m glass fibre clad in reducing refractive index materials.

Looking at the images, the scale bar on the right side immediately highlights the significant increase in intensity within the waveguide. Whilst the light emerging from the fibre in a) diverges, with a significant portion going above and below the waveguide, in b) the light is less divergent, meaning more light couples into the waveguide. In absolute numbers, for a flat fibre end only 30% of the light enters the waveguide. With a tip this raises to 49%, which may be increased even further by idealising the geometry for the wavelength. For a silicon photonics researcher this would result in a stronger signal detected at the other end of the waveguide.

![Contour plots of optical-fibre/waveguide coupling](image)

**Figure 5.22:** Contour plots of optical-fibre/waveguide coupling, with and without our structure as coupling mediator. Figures show an optical fibre, made of a single core surrounded by cladding (indicated in yellow), emitting light into an aligned waveguide a) without and b) with our optical structure milled into the optical fibre. A nearly 66% increase in intensity within the waveguide is apparent. The smaller insets in each image show the first 1-2\( \mu \)m of the waveguide. In b) the surrounding colour is a deeper blue suggesting greater contrast and more light within the waveguide.

### 5.13 Conclusion

In this chapter, we have simulated a structure for producing nano-jets, high-aspect ratio beams of light. We introduced the idea of using such things in photolithography, something that will be demonstrated in Chapter 6. The structure consists of two side-by-side curved parabolic troughs that form a jet of light between them. We began our discussion of the models with a simple look at our curved-profile structure compared with a metal-grating, structured glass-grating and flat triangular structure. We saw that the grating alone was not enough to form any sort of beam, meaning they are not formed through diffraction. We also saw that both the curved and triangular
structures formed jets, but the curved structure formed longer, slightly thinner jets closer to the surface of the glass.

We then moved on to look at how varying the curvature changed the nature of the jet, through varying the depth of the troughs. The highest intensity reached in this way was for a depth of 1.25\(\mu\)m, whilst the longest jets where found for the smallest depths at around 0.375\(\mu\)m deep. As the total intensity within the jet in 1.25\(\mu\)m was twice that of 0.375\(\mu\)m, it leads us to conclude that the very high length comes from a shallower drop off in intensity over the length of the beam. They may have had similar intensity 16\(\mu\)m from the structure surface, but as we measure the drop off to half-maximum, the result is a more intense peak will read as shorter.

The FWHM was seen to drop below the wavelength between depths of 1.25-2.0\(\mu\)m. The transmission through the structure was seen to linearly decline with an increase in depth; however a peak in the ratio of light collected into the jet was seen for a 1.0\(\mu\)m depth. Over-all we saw that controlling the profile of the troughs would allow us to tune the light transmitted into the jet, as well as the length and width of the jet.

Next, we discussed how varying the size of the apex (as defined in Figure 5.1) affected the beam. Whilst the peak intensity varied little for sizes between 100-1500 nm, the jet length increased dramatically as the apex width increased. For the smallest apex widths, < 500 nm, the FWHM was seen to dip above and below the wavelength, possibly caused by an interference effect, based on the internally scattered light.

When varying the wavelength, we saw that the maximum intensity dropped with an increasing wavelength, whilst the focal length increased to peak at 400 nm, before slowly decreasing. The FWHM increased nearly linearly, always remaining above the wavelength in width. We surmised this was due to the geometry not being idealised for any single wavelength.

When looking at changing the refractive indices of the structure and surrounding medium, we came to the conclusion that the change in the refractive index controls the ability of the structure to form a jet. As the refractive index change increased, so did the peak-intensity in the jet, whilst the FWHM reduced. As the transmission was seen to drop at a linear rate with \(\Delta n\), it indicated that, whilst the total amount of light allowed through decreases, the gathering power of the structure increases, allowing it to form more intense, narrower beam.

We then changed the structure to a single protrusion, and in doing so showed that it acts similarly to an array. By then varying the profile either side of the apex, by varying the depth, we then investigated how the angle of the beam could be varied. We found that as the relative depth of one side was increased, with respect to the other, the beam
deflected towards the shallower of the two. The change was linear between ±5°. The trend stayed linear even at 20°, but the length of the jet was diminished and it began to emerge from within the smaller trough, rather than above the apex.

By introducing metal slits, we showed that very little of the light in the beam comes through the apex. Most of the light is refracted in from the curved trough surface. We also showed that reducing the range of angles that can enter the beam (by reducing the aperture size), it not only drastically reduces the beam length, but also reduces the beam width.

We then described how the jet is formed, along with a little discussion of the nature of the jet. The jet is a quasi-Bessel beam, with the characteristic x and y profiles of such a beam. As light is refracted towards the centre of the structure from either side of the apex, it interference and forms a beam. In our structure, the curvature controls how the light interacts at the high/low refractive index boundary. It can either refract into the path of the beam, internally reflect and be lost to the beam, or totally internally reflect back into the beams path. We have shown that the totally internally reflected component makes up 5% of the beam or less. By changing the profile, via the depth or the diameter of the trough structure, refraction into the beam could be idealised to deliver the maximum intensity possible into the beam.

Having an idea how the beam was formed, we finally ran one further pair of simulations, looking at a possible use in gathering light into a photonic waveguide. When an optical fibre was butted directly against the waveguide 30% of the light was trapped within the waveguide. With the addition of our structure to the end of the fibre, this number raised to 49%, which could easily be pushed higher by idealising the profile for the longer, mid infra-red wavelengths that are commonly used in photonic applications.

In the next chapter we will move on to looking at experimental verification of our modelled results. This includes our early results in photolithography, as well as later results which came from direct transmission imaging of milled structures.
Chapter 6

Micro-optical light-guides

Summary
In this chapter we look at experimental verification of the nano-jets we observed computationally, showing good agreement between the modelled and experimental results. We begin by examining the structures formed in photoresist when a jet is used in photolithography. We then move on to direct imaging of glass and sapphire masks in various refractive index media, comparing the modelled and experimental results. We then show our ability to manufacture structures of arbitrary shapes and footprints by producing posts, S-bend and doughnut shaped jets. Finally, we demonstrate a light-sheet former made in silicone-rubber via a scalable reflow and replication technique.

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6.1 Introduction

During our work on modelling micro-lenses, we also started investigating their applicability to lithography. Whilst the bulk of our work was in viewing beyond the optical limit of light, we wondered if, by combining photo-lithography and micro-lenses, we could make sub-wavelength sized features.

The project started by producing lithography masks in the FIB, by milling dishes into metal-coated glass, using the method discussed in Chapter 3.2.1. The mask was then used in conventional UV photolithography to see what patterns emerged in the photoresist below the structures. In Section 6.2 we show and explain images of the patterns that emerged. From these images we noted how the light appeared to be removing the material between the milled structures, were we expected them to be at the focal point of the milled structures.

By studying the computer models, seen in Chapter 5 we realised we had stumbled across a method of producing a modified version of a Bessel-beam. These beams are different to the optical nano-jets observed from micro-spheres [57][129] having depth of fields several times longer than the spheres, with significantly less divergence as one would when using a focusing lens, as characterised by the numerical aperture. These are very long (along the z-axis) regions of high intensity light, with a very high aspect ratio. As noted in our literature review in 1.2.1.4, one of the current theories of how micro-sphere assisted microscopy works is through producing optical nano-jets.

Summarising what was seen in Chapter 5, we looked at sets of closely spaced 3μm diameter troughs, varying the depth, separation, illuminating wavelength and refractive index. We saw that varying these properties altered the length, diameter and maximum intensity within the jets. Throughout this chapter we will use this knowledge to compare experimental data to that predicted computationally. Included in our models were simulations of off-set troughs (Chapter 5.9), showing clearly that we can steer the beam by ±5°. In Section 6.6 we explore this further, by manufacturing off-set troughs and measuring the beam angle.

We begin by looking at our earliest experiments using milled structures for photolithography.

6.2 Photolithography and Mask Making

To understand the propagation of light through our structures, several masks were made by milling a set of patterns into a metal coated transparent substrate, where the metal
coating is thick enough to block light transmission. These were initially used in photolithography, creating an image in the photoresist of how the light propagates through the mask. The masks are then later examined in a transmission optical microscope, to directly view the transmitted light pattern.

The substrates we used included silica-borite glass cover-slips and single crystal sapphire ($\text{Al}_2\text{O}_3$). The metal layer was beneficial for two reasons: firstly as a method of stopping light in the un-milled regions (as is standard in photolithography) and secondly as an electrical conductor, reducing surface charging when milling structures into the insulating glass/sapphire substrates.

The cover slips were cleaned with a standard triple-clean, before metal was deposited using a sputter-coating system (JLS, MPS). Three types of metal coatings were used, the first being 75 nm of gold (Au), with a 5 nm adhesion layer of titanium (Ti). The second was coated in 80 nm of chromium (Cr), and the third a 100 nm layer of palladium (Pd). All three metals were chosen for their availability, the later two were chosen to limit plasmonic effects and for their hardness.

A range of patterns were milled into the masks made. Figure 6.1 shows images of two sets of patterns; a) shows a Cr on glass mask, with 7 sets of patterns. From left to right these are octagonal, hexagonal and pentagonal close-packed dishes, then three 3\(\mu\)m wide by 10\(\mu\)m long troughs with increasing spacing between troughs moving down the rows, and increasing depth (i.e. shorter focal length) moving along the columns. The final set is a single 10\(\mu\)m wide by 10\(\mu\)m long trough, increasing in depth moving down along the rows.

The second image, b), is Pd-coated sapphire, with some of our later, and more unusual geometries. The first, $S$-shaped pattern to the right is a pair of highly curved troughs. These demonstrate the advantage of our FIB manufacturing technique, in that any footprint or curvature is possible. In cross section, it is similar to a pair of side-by-side troughs, and in Section 6.6 we show how this forms a curved nano-jet.

The three circles to the right of the ‘$S$’ are rotationally symmetric posts, made such that a single beam forms above the centre of the circle. The centre set of troughs are identical to the second column of troughs in image A). These are used to directly view the light sheet formed, and then test the effect of changing the refractive index of the medium in which it sits, in Section 6.5.

Between the troughs and the circular doughnuts is a shallow, flat square, milled into the metal to give an area to normalise the transmitted light intensity to, when possible. The final patterns are doughnuts made of two troughs, creating a circular jetting region, which could be used as an optical trap in the manipulation of ultra-cold atoms and small
particles. The comparative depths of the inner and outer troughs has been varied in the top and bottom doughnuts, creating circular offset troughs, similar to the models we created in Chapter 5.9, in which Figure 5.15 b) showed that angling the lens by \( \pm 5^\circ \) is easily achievable by offsetting. We will discuss the doughnuts, along with the circular-posts and s-shapes in Section 6.6.

![Figure 6.1: Images of the a) chromium on glass and b) palladium on sapphire masks used throughout this chapter. Shown are various milled structures, including close packed structures in the left of a), along with the troughs used throughout this chapter. b) contains an S-shaped structure and three doughnuts that create curved light sheets, as well as posts, that form single beams.](image)

A Mask with features identical to that shown in 6.1 a), but gold coated on glass, was used in standard contact photolithography. Using a spin-coater, 2ml of surface preparer, Hexamethyldisilazane, was applied to a glass substrate at 4000 rpm for 10 seconds, accelerating at 200rpm/s. The photoresist AZ5214e was then spun on at 4000rpm, acceleration 200rpm/s, for a further 30 seconds, leaving an even coating. The substrates where then baked at 95°C for 1 minute, before being allowed to cool.

When cooled, they where placed in a mask aligner (Suss, MA1006), with the mask laid on top of them. Unlike in conventional mask alignment, the mask was not pushed into contact, which will come to explain some of the variation in results we will see. The samples where then subject to a 6.0s flood exposure of 362 nm UV light.

Development was carried out by dipping the samples in a 1:5 ratio mixture of microposit developer and de-ionized water (DI water), for 35 seconds, before a dunk in a DI-water kill-bath, a wash with fresh DI water, and drying with a nitrogen gun.

With the samples prepared, we examined them using optical and electron microscopy, as well as AFM.
6.3 Three trough photolith

Part of the standard mask included three 10µm long troughs, with a 3µm diameter of curvature. Under these structures we saw patterns of lines in the photoresist, as seen in Figure 6.2. The focal point of the parabolic profile of the trough was set to be at the surface of the mask. Image a) shows an SEM image of the photoresist after developing, b) displays a 3D AFM heightmap of the same sample. In the photoresist, we can equate the post-development depth with the light-intensity, in a similar way to FIB depth equating to ion-dosage, discussed in Chapter 3.

We had originally expected three lines with similar intensity, positioned under the centre of each trough and indicating the focal point of the dish-type lenses. Instead, we see several pairs of lines of similar depth, demonstrated by the AFM profile in d). As there is reasonably strong reflection symmetry down a plane between the central two lines, it appears the lines more likely to coincide with the edges of the troughs.

In b) we have overlaid three blue-dashed rectangles to indicate the position of the mask. The middle two lines in the photoresist agree with the position of the inner and outer edges of the troughs, showing that the highest intensity light was between the troughs, and not at the centre of the troughs, as originally expected.

The third and indistinct fourth sets are well outside of the mask region. They appear to be from the second and third order scattered light seen in the models run throughout Chapter 5. Figure 6.2 d) shows the comparison of the AFM profile with a plot of the square of a first order Bessel function of the First Kind ($J_1(x)^2$). The Bessel profile has been inverted, for ease of comparison. Whilst the depth of the higher order troughs in the resist does not perfectly match the function, the two are similar in both spacing between the peaks, and in rate of drop off. This is a good indication that our FDTD models correctly simulated the structure.

To get an idea of how the nano-jets change with distance from the mask, a series of images were taken under a transmission optical microscope (Zeiss axiophot), starting from just below surface focus, and increasing the distance between microscope and mask in 1µm steps.

Figure 6.3 a)-i) shows nine transmission optical images taken at various points through the focus of the top central trough set seen in Figure 6.1 a). In Figure 6.3 in a), the image was taken with the microscope focused at the surface of the mask. Moving to b) the image was taken 1µm above the surface. Two very bright lines have appeared, in line with the gaps between the troughs. This corresponds to our nano-jet. In images c)-i), we increase the distance between the imaging plane and the mask from 4µm up to
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Figure 6.2: Images of photoresist exposed below a three-trough structure. a) shows an SEM image of the resist post develop, b) is the AFM contour plot of the resist, with the position of the mask overlayed (blue dotted line) c) shows a transmission optical image of the mask, demonstrating the path of the light through the mask. Graph d) shows the AFM profile across the resist, plotted alongside the $J_1(x)^2$ Bessel function. Whilst the intensity of the higher order peaks is greater in the resist, the spacing between peaks conforms well.

$17\mu m$, in steps of $2\mu m$ and then $3\mu m$. As the distance increases, second order effects spread further from the centre of the trough set, before the beams eventually begin to merge. In 6.3 c) and d) we can see a single central line, which corresponds to what we saw as the diffraction pattern of the three glass apertures in Chapter 5.3, Figure 5.2. At the same time, the bright lines corresponding to light jets emerging between the troughs are still there, and there is no bright-dark contrast reversal typical of going through the maxima and minima when changing the diffraction conditions.

The furthest images from the surface, g-i), show that the jets are still well distinguished at distances longer than $10\mu m$. Only at $14\mu m$ (image h) do the tails of the two central jets begin to meet, causing a brighter line between them. From $17\mu m$ (image i) the lines begin to merge more quickly, and are indistinguishable at $20\mu m$ (not shown). In our models, Chapter 5.4, we often saw the beams from shallower troughs reaching very long distances (for example, see Figure 5.3). $10\mu m$ is the length we expected from a $0.75\mu m$
deep trough. As the beams are still reasonably distinct to $17\mu \text{m}$ away from the mask’s surface, we can say that the structure exceeded our expectations.

![Images taken at various focal points moving through a Glass mask in air. The red number in the top corner indicates the distance above the mask surface, from 0-17\(\mu\text{m}\). Two clear bright lines appear between the troughs at 1\(\mu\text{m}\), indicating jetting, and stay distinctive for over 14\(\mu\text{m}\), upon which the jets merge.](image)

**Figure 6.3:** Images taken at various focal points moving through a Glass mask in air. The red number in the top corner indicates the distance above the mask surface, from 0-17\(\mu\text{m}\). Two clear bright lines appear between the troughs at 1\(\mu\text{m}\), indicating jetting, and stay distinctive for over 14\(\mu\text{m}\), upon which the jets merge.

Looking back to Figure 6.2, image c) is from the set of transmission optical microscope mask images. By measuring the distance between the lines in the resist, and comparing them to the optical images, we estimated the air gap between the mask and surface at 8\(\mu\text{m}\). Due to the photolithography being performed by placing the mask on the sample, without a constant force applied, this distance varied greatly from sample to sample, from 2-3\(\mu\text{m}\) up to 20\(\mu\text{m}\). In conventional photolithography, the distance between the mask and surface is carefully controlled for exactly this reason.
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Taking the profile across the images in Figure 6.3 allows us to build up a picture of how the nano-jets change, micron-by-micron. Figure 6.4 shows a set of such profiles, next to a MEEP model of an identical structure. The structure was 750 nm deep and metal coated. Comparing the nano-jets from the optical images with the modelled data, we have a very good match.

We can see a smaller, secondary peak after the first in both experimental and modelled data, which we have highlighted with a red arrow in Figure 6.4. The ratio of the smaller to the larger peak taken from the experimental setup is $0.64 \pm 0.01$ whilst the ratio from the model is $0.84 \pm 0.02$. The error was evaluated using the peak values at $\pm 1\mu m$ from the maximum intensity. Whilst the two values are not within error of one another, they are still close enough so that we may be able to indicate the source of the difference (within 20%). Two factors that may contribute to this lower peak intensity are: firstly, the limited collection angle ($\sim 70^\circ$) in the microscope, compared with knowing what 100% of the light does, within the model. The second reason is that, whilst the model was run at a wavelength of 350 nm, absorption in the glass was considered negligible, as well as having a wider wave-packet than a UV lamp would provide ($\pm 100$ nm). The value for absorption is low, but is 50% higher than for visible wavelengths[134], resulting in lower peak intensity than the MEEP model.

The first and second order effects, discussed earlier in Section 5.1 & Section 6.3, are clearly visible in both the model and the experimental plots. Whilst the model does not match the experiment perfectly in this respect, we know that the microscope is not ideally suited for collecting off-angle beams, with its limited collection angle.

If we look at the y-profile of the beam, shown in Figure 6.4 c), it is a similar, yet inverted, copy of the AFM profile seen in Figure 6.2. Having directly viewed the light intensity, we can confirm it is very similar to the square of a higher order Bessel function, with two peaks surrounding the centre, followed by evenly spaced peaks of decreasing intensity[135]. In Figure 6.4 c) the intensity has been compared with a $J_1(x)^2$ plot (red line), where the x-axis of the Bessel function has been multiplied by 0.77, squeezing it to give a better fit. The peak fits well in both peak height and spacing, out to the second order peaks (third peak from zero). The furthest out peak in the intensity profile is positioned closer to the centre and is significantly less intense than the Bessel function, with the probable cause being a lack of intensity in the input beam. The *ideal* Bessel beam requires infinite energy, and so a finite energy beam must diverge from this profile at some point.

The profile along the axis of propagation, shown in d) also shows a typical Gaussian-Bessel beam profile for the z (propagating) axis, with a dramatic rise to the peak, which
sits close to the surface, before a gradual dropping off. We showed such a peak in Chapter 2.2.10 Figure 2.6.

Whilst simple trough structures are able to produce light sheets, which could be industrially relevant to photolithography, sensing and solar cells, arrays of more complex footprint dishes produce more complex light patterns. So, having discussed our three-trough structure in photolithography, we now move on to looking at using close-packed hexagonal structures.

### 6.4 Hexagonal Photolith

Along with the milled troughs, close-packed hexagonal structures were milled into our photolith masks (seen in Figure 6.1 a). Figure 6.5 shows the AFM height maps of
three patterns made in photoresist using the same hexagonal mask. Overlaid in blue-dashed lines are the outlines of the mask above them (based on rotational symmetry). The images below contain transmission optical images taken through the mask in a similar method to the troughs in the previous section. From this we estimated the photoresist/mask air gap at $1 \mu m$ for the first image, $6 \mu m$ for the second and $9 \mu m$ for the third.

The first image has a deep, star-like pattern with six arms that surrounds a hollow circle of material. It suggests that between the hexagons there were very-high intensity, thin beams of light. As the original intent of the experiment was to assess whether the photoresist could be formed into subwavelength features, we measured across the centre of the arms, finding an average width of $1.17 \mu m$. From a profile across the central ring, we see that the ring is a $1 \mu m$ tall circular peak with a full width at half-maxima of $1.3 \mu m$. The full width at 0 was not possible to measure due to the centre of the ring being the only place in the pattern to go all the way through the photoresist to the glass substrate below. This shows that the lithography is well above wavelength scale, by 3-4x, suggesting the technique may not be ideal in its current form.

Also visible in image a) are points within the centre of the outer ring of hexagons, as well as a pattern made up of 3 points just outside of the outermost edge of the hexagons. The centre points, similarly to the troughs, are caused by diffraction through a grating. The three points appear to have originated from the combined contribution of the hexagon corners, as well as second order light scattered from that edge.

The second image, 6.5 b) shows another AFM height map of photoresist, which by comparison with e), allowed us to estimate a $6 \mu m$ mask-resist air gap. The pattern is clearly different, where before there was a ring with arms, now there is a set of deeply indented spots at the corners where 3 hexagons meet. The transmission optical image 6.5 e), taken at $6 \mu m$ from the mask surface, shows an identical dot pattern, 6 bright spots surrounding a central point, with a further 6 less intense spots. Again, the less intense spots relate to the diffraction effects at the centre of the dishes. The centre spot of the central hexagon appears more intense than the edge ones, due to re-enforcement from light scattered from surrounding hexagons.

In 6.5 b) strong first and second order effects can be seen in the resist surrounding the pattern. These correspond to the just-visible scattered light in 6.5 e). The pattern of three points at the edge of the dish region, visible in the last image, a), are now a single spot which has moved further away from the edge of the mask. The change in distance from the centre of the pattern is alone enough to suggest that the mask is further from the surface of the photoresist, as the higher-order effects always move away from the centre of the pattern.
Figure 6.5 Image c) shows an AFM height-map of the photoresist with a much larger sample/mask distance. We show the transmission optical image for a 9µm air gap for comparison, but a larger distance (10-15µm) may be a better fit. Due to the diffuse nature of the light, causing large areas of material to be removed, it was difficult to accurately gauge the distance with our usual method. The images serve to show how dramatically the pattern of the light changes with distance, meaning any photolithographic method based on such an optical system would need very precise control over the sample/mask distance.

Figure 6.5: Figure shows the photolithographic pattern created by a hexagonal array of dishes. a), b) and c) are lithographic patterns made using identical masks. The change arises from a change in distance between the mask and photoresist. c), d) and e) are transmission-optical images of a mask helping identify the mask-resist distance as 2µm, 6µm and 10µm respectively (indicated in red in the images).

From our data so far, we can conclude that we have intense jets between the trough structures, and not at the focal points of the optical elements, as predicted by our models. The light intensity was high enough to preferentially expose photoresist. We have seen that, over 17µm, the beams are straight, and have very little divergence, widening slowly with respect to a conventional focal point. This property may be very useful in industrial photolithography, allowing for deep, straight walled shapes to be
quickly manufactured, provided strict control is exercised over the mask-photoresist air gap.

Comparing experimental data to the simulations from Chapter 5, we saw very good agreement between the data sets, including the zeroeth, first and second order peaks. The x and y profiles verified that the nature of the jet is that of a Bessel beam, as we discussed in Chapter 5.11.

The next step is to experimentally verify that the difference between the refractive indices ($\Delta n$) controls the shape of the beam, rather than the absolute values for the indices. In Chapter 5.7 we discussed this in detail, and concluded from our models that, not only does the difference in refractive indices control the beam profile, but that larger refractive index changes gave a more intense, thinner beam. In the next section we study, using transmission imaging, the properties of a glass and a sapphire mask immersed in liquids of various refractive indices.

### 6.5 Refractive Index

Up to this point, all of the masks examined have been made of borosilicate glass ($n = 1.52$), and imaged in air ($n \approx 1.0$). As we saw in our simulations, Chapter 5.7, the difference between the refractive indices of the structure and surrounding medium controls the jet properties; the absolute values of the two indices does not matter. In this section we investigate this experimentally, using two masks, one of borosilicate glass, the second of sapphire ($n = 1.77$). We vary the index of the mask, and then image in different media, including: air, water ($n = 1.33$) & glycerol ($n = 1.47$). These two masks and three media give us 6 different values for the refractive index difference, $\Delta n$.

If the experimental results agree with the model, we expect to see similar trends in the peak-intensity and FWHM as we saw in our models, where the intensity increased logarithmically, whilst the FWHM decreased exponentially. In our model, we had duplicate values of $\Delta n$ using different refractive index masks which, due to the clustering of values, allowed us to draw the conclusion that $\Delta n$ controls the beam properties. As we lack exact duplicates experimentally, we will have to approximate where we have pairs of close values.

Having already imaged with a refractive index change of $\Delta n = 0.52$ with glass/air, we move on to look at sapphire in air $\Delta n = 0.77$, then each lower value of $\Delta n$ in turn.
6.5.1 Sapphire-Air $\Delta n = 0.77$

As before, a set of three 3$\mu$m diameter troughs of 750 nm depth were imaged in transmission, under an microscope. Figure 6.6 shows images taken whilst focusing through the mask, as was previously done in Figure 6.3. Images are shown at a) surface focus b) 1$\mu$m above the surface, c) 3$\mu$m, d) 5$\mu$m, e) 10$\mu$m & f) 15$\mu$m, as indicated in each image in red.

Image a) shows a reasonably clean image of a mask, very similar to the glass/air image shown previously, with the exception of a piece of dirt sitting in the mid-left on the top metal track. Image b) shows the turn on of the jets however, unlike with the glass example, a strange structuring of the light is apparent, with additional bright lines either side of the jets, including thin lines along the centre of each trough. There is a noticeable chromaticism in the image, the light from the troughs is red tinted, whilst the beams themselves are blue. This demonstrates a dispersion effect, but as sapphire is not very dispersive over the range of visible wavelengths$[136]$, it suggests that some water may have been left on the lens after immersion imaging (discussed in section 6.5.2 & later).

Image c) is more like the expected result, with two distinct lines over the metallised regions, brighter than their surroundings. Unlike the air-example, no first or second order scattered lines can be seen. The zeroeth order jets are still well separated at 5$\mu$m, but begin blurring immediately after, so that by 10$\mu$m (image e) the jets are no longer distinct. Beyond 10$\mu$m from the sample, the jets merge, shown in 6.6 f).

In Figure 6.7 we show side-by-side experimental intensity profiles at different heights from the mask, next to modelled data. Near the surface of the structure the two images are similar in many ways, with two large zeroeth-order beams, surrounded by higher order beams, but these are quickly lost, merging into the main peak. The two central beams merge after $\sim 11\mu$m, which is around the position the two smaller peaks within the zeroeth-order beam appear in the simulation.

Some of this merging may be due to blurring of the image, caused by the lens improperly drying after liquid immersion.

6.5.2 Sapphire Water $\Delta n = 0.44$

Immersing sapphire in water creates a difference in refractive indices of 0.44, close to our Glass-Air value of 0.52. We have imaged a sapphire mask through an aqueous medium, shown in Figure 6.8. The images shown here are 0-15$\mu$m from the surface, as indicated in red within each image.
A droplet of water was put on the mask, and then the microscope was moved over the sample. As the mask and microscope lens came into close proximity the droplet wetted the microscope, forming a continuous medium between them. We must add that the
lens used was not idealised for imaging in water, and so dispersion, as evidenced by chromaticism, was present in our images.

Image a) is already slightly blurred by the presence of the water, but clearly no jets are present. In image b) they turn on and grow stronger in c). Moving to d) the distinct lines begin to split in two, caused by the dispersion of the water, and in e) the beams have split into many thin lines, with the colour shifting from white to red to blue.

In the sapphire-air example, where a trace amount of water was present on the surface of the lens, dispersion occurred and the components of the light were separated at the objective lens, forming an image with red troughs and blue lines, based on the angle of the incoming white light. In the sapphire-water example, the splitting appears to occur at the sapphire/water interface, causing the beams themselves to split apart by wavelength (colour). Figure 6.8 d) clearly shows three-peaks, with a blue, green tinted and red visible.

This strong splitting comes from the dispersion in the water’s refractive index along with the larger refractive change between the two media. Referring back to Snell’s law, Equation 5.1, described in Section 5.11, we see that as $n_1$ increases with respect to $n_2$, the angle of refraction, $\theta_2$, increases. The large change causes stronger bending of the light’s path, whilst the dispersion in the water means that not all wavelengths are bent to the same angle, similarly to how a prism creates a rainbow, and the effect of chromatic aberration in lenses.

In f) the light has merged into two amorphous white blobs, meaning the separated wavelengths of light have spread to the point of merging back into a single diffuse spot.

Whilst over the optical wavelengths, the change in the refractive index ($n$) of the water is small ($\sim 1.36-1.33$), the change in absorption ($k$) is large, dropping by 2 orders of magnitude and rising back again between 200-700 nm. When combined with the large range of angles found within the trough, this causes the dispersion we see.

The comparison between intensity profiles as a function of imaging height and modelled data in Figure 6.9 still indicates that two jets propagate for a long distance, although due to large overlapping tails they are less-distinct. Examining 6.9 a), the first 10µm are chaotic, with the main jet peaks somewhat hidden by the additional peaks caused by dispersion. At around the 5µm mark the central two peaks appear to split, showing the effect we saw in Figure 6.8 e). The splitting changes in the images around it, causing the most intense peak to dance around, so that unlike the glass-air example, no clean line can be followed along the z-axis, as we image away from the surface of the mask, along the peaks.

The peak intensity of the jets is found close to the surface of the structure, within the
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Figure 6.8: Set of images taken moving through the focus of a Sapphire Water structure. The images were taken between 0-15µm above the mask surface, as indicated in red. Chromatic aberration from the structure can be seen, caused by the dispersion in the water, most visible in the 10µm image where the jet has split into 3 colours.

first 2µm. In contrast, the absolute length of the jets is very long, with the two still distinct from one another at 18µm from the surface.

As with previous examples, the secondary peaks in the main jets (seen in 6.9 b) appear at the point the jets begin to overlap, reaching maximum intensity at around 9µm in the z-axis in both experimental and model.

6.5.3 Glass-Water $\Delta n = 0.18$

Next we move on to look at a glass mask immersed in water. The images in Figure 6.10 were taken at 0-4µm from the mask surface. Only the first 4µm are shown, as the beams merged and flattened unusually quickly in this case, compared with the 10+µm beams the modelling predicted.

The first image, a), shows the surface of the mask. Unusually, the jets are already visible at the surface, suggesting that, as with our deepest troughs in Chapter 5.4, the jet is beginning at the surface of the metal and decaying.
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Figure 6.9: Comparison of a) experimental and b) simulated light intensity profiles through a Sapphire mask in water. Two clear peaks can be seen in the experimental data, however they partially merge 11 µm from the surface, making them less distinct.

Above the surface, the beams are still present, widening substantially. The low refractive index difference ∆n should direct significantly less light into the jet than when the medium was air, which we see by the beams never becoming very intense. Strong secondary peaks are present coming from within the troughs.

As with the sapphire-air example, we see a colour distinction between jet and trough. The images were white balanced by the microscope software, equalising the RGB histograms. The jets appear red-tinged by the effect, whilst the light seen scattering from within the troughs is blue/white tinged, indicating the wavelengths have been spatially separated. As we illuminate with a broadband white light (tungsten filament), the variation in colour must come from dispersion from the water, as this effect was not visible when imaging borosilicate glass in air.

In Section 6.5.2, we saw chromatic aberration where a single peak is spatially split into its colour components at the structure/water interface. Here what we see is more likely dispersion between the water and imaging lens, splitting the colours so that only certain wavelengths are at acceptable angles to be captured by the CCD. This changes the local colour of different sections of the structure, without splitting apart the image.

The intensity-slices in Figure 6.11 show how the jets drop off almost entirely beyond 5 µm from the surface. In the background, nearest to the surface, there are multiple peaks that don’t follow a very clear progression. The additional tall peaks confuse the issue by moving across the profile. Only when the jets have strongly merged does the pattern become slightly clearer, with hints of two central jets (green region).

Interestingly, a very minor second order peak can be seen to the right of the main cluster (orange region). This peak begins at around 2 µm and continues for a few microns before
Figure 6.10: Images taken moving through the focus of a glass mask in water. Red numbers indicate the height above the mask surface the image was taken. Spreading out.

Figure 6.11: Comparison of a) experimental and b) modelled data for a Glass mask in Water. A steep drop-off in intensity is seen in the experimental data, which does not agree well with the simulation. Small peaks can be made out in the background. Simulation axis is measured in pixels, scaled at 35 pix/µm.
6.5.4 Glass - Glycerol $\Delta n \approx 0.05$

Finally we move on a glass mask imaged in glycerol. Glycerol was chosen for being optically transparent, whilst having a refractive index of $\sim 1.47$, just below that of the glass used. This gives a $\Delta n$ of 0.05, the smallest change we measured. A second advantage of Glycerol is that its refractive index is linearly tunable by mixing (by weight) with water, which would allow for more measurement of interim indices, if needed.

Figure 6.12 shows the mask imaged between 0-8$\mu$m from the surface, as labelled on the images. The surface image a) is already slightly out of focus due to the addition of the glycerol. As we move up, brighter lines are apparent at the position of the metal tracks between the troughs, although not very distinct. These continue, peaking in intensity in the 3$\mu$m image, and fading at around 8$\mu$m from the surface.

This progression clearly shows that the glycerol has dramatically reduced the light gathering ability of the structure, nullifying its jet forming capabilities. This demonstrates that refraction of the light must be the main jet forming mechanism, as we concluded from our modelling.

The experimental profile plots in Figure 6.12 a) agree well with the modelled data, shown in b), with specifically less-distinct peaks being formed. It is worth noting that the model shown shows a 0.1 refractive index change, whilst the experimental data has closer to a 0.05 change. This suggests that the peaks should be less defined in the experimental data, which appears to be the case. In the experimental data, two small peaks (indicated with red arrows) can be followed along the body of the background light. Passing $\sim 10\mu$m the two peaks drop off entirely and only the background is left. In the model the two central jets merge with the secondary jets to form two wide, yet still distinct peaks. These peaks are likely to have formed due to reflection off the backwall of the computational cell, so it is not surprising we don’t see them experimentally. Looking back through the experimental intensity-profiles so far, we can see that as the difference in refractive indices reduced, the background light tended to increase relative to the peaks, suggesting the gathering power, the ability to form an intense jet, is decreasing.

6.5.5 Refractive index change summary

Now we come to quantitatively compare our experimental data to our models. In Figure 6.14 we show the peak intensity, FLHM, focal length and FWHM as they vary with the refractive index difference, $\Delta n$. 
Figure 6.12: Images taken focusing through a Glass mask submerged in glycerol ($\Delta n = 0.05$). The distance above the mask surface ranged from 0-8$\mu m$ and is indicated in red in the image. Whilst two bright lines can be seen running through the centre of the structure, they are very indistinct due to the low gathering power of the structure when $\Delta n$ is so low.

Figure 6.13: Comparison of a) experimental and b) modelled intensity data of light passing through a Glass structure in glycerol. The experimental value for $\Delta n$ is 0.05, whilst the model represents a $\Delta n$ of 0.1. The two data sets are similar, with small peaks protruding from a largely unmodified background, suggesting the structure has very little gathering power.
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The trend for the maximum intensity is to slowly increase as the refractive index change (\(\Delta n\)) increases. The drop in intensity of the \(\Delta n = 0.44\) (sapphire in water) before suddenly rising at \(\Delta n = 0.52\) (glass in air) suggests that dispersion in the water had a big effect on the intensity. As we saw in Figure 6.8, the sapphire-in-water jets are split by colour, so that the intensity of each individually coloured peak was low. By recombining the central peak of the sapphire-water with the two split peaks that surround it, the intensity of the peak increases from 74% of the glass-air intensity up to 91%, creating a trend more reminiscent to the one from the simulation data, in Chapter 5.7 Figure 5.10.

The FLHM in Figure 6.14 shows two lines. The first (blue triangles) is the measured length over which the jet remains above half of the maximum intensity. In this case, all but glass-water (0.185) remained intense over the full range they were measured. However, in most cases the jets merge well before they drop below half maximum intensity, forcing us to introduce a second line (orange-diamonds), which measures the length over which the beams are distinct.

The blue-triangle line shows the FLHM is long for the largest refractive-index-difference, \(\Delta n \geq 0.4\), but reduces for the lower values. The glass-water example (\(\Delta n = 0.185\)) appears to have suffered from scattering of the light in the water, more so than was seen in the sapphire-water example. This very quickly dropped the intensity, over only a few microns, creating a steep drop off in the FLHM. The reason only this sample suffered it may be because of the combination of dispersion, and the very small difference in refractive indices, reducing gathering power. The lowest value of \(\Delta n\) (0.05), did not suffer from such issues due to the lack of dispersion in glycerol.

Bringing the orange line into the discussion, which shows the length at which the jets merge, it drops as \(\Delta n\) increases, with the lowest value found in air, at \(\Delta n \approx 0.52\). Comparing both lines with the simulated data (from Chapter 5.7, Figure 5.10b), the modelled FLHM was longer than the computational cell (17.5 \(\mu m\)) for all refractive index differences, up until \(\Delta n = 1.1\), where it dropped to 2 \(\mu m\). In each case, the jets within the simulations remained distinct for the length of the FLHM.

The peak intensity length, a measure of the distance from the structure to peak intensity position, is between 2-4 \(\mu m\), showing that the peak intensity stays close to the surface. This suggests the peak position is less of a function of the refractive index, and more of the profile of the troughs, as we saw in Chapter 5.

The FWHM remains above the average wavelength (\(~550\) nm) in every instance, with the glass-air (\(\Delta n = 0.52\)) coming the closest at 773 nm. In the computer models there was a drop off from \(~1.5-0.5\) \(\mu m\) full-width as \(\Delta n\) increased from 0.3 to 1.1. In the modelled data \(\Delta n = 0.5\) gave a FWHM of between 0.9-1.2 \(\mu m\), so the experimental data has actually exceeded our expectations from modelling in that respect. However the
increase in FWHM to $\Delta n = 0.8$ is surprising, as models predicted a decreasing towards 1.1. The increase to 1.5$\mu$m at 0.8 suggests an issue during measurement, which as we noted may have been due to the lens not fully drying from previous submersion in water.

As with the models, the FWHM of the lowest refractive index change is significantly larger than any other value, reaching 6.5. Again, this is caused by overlapping of the peaks causing them to merge.

In this section, we set out to test whether the absolute values of the refractive index matter, or whether it is only the difference in refractive indices that effect the jet properties. In our simulated data, from Chapter 5.7, we saw various pairs of refractive indices with identical values of $\Delta n$ give similar results. The clustering of data points seen in the peak intensity and FWHM were enough to conclude $\Delta n$ the controlling factor. In our experimental data we do not have multiple data-points for a single value for $\Delta n$. Instead we must compare nearest neighbours. We have two values of $\Delta n \approx 0.1$ (pairing 0.05 with 0.185), two values of $\Delta n \approx 0.5$ (pairing 0.44 with 0.52).

In intensity, the lower pair are nearly identical, giving credence to our theory. The higher pair are very different, with $\Delta n = 0.44$, 26% lower than $\Delta n = 0.52$. However, as we noted earlier in this section, recombining the peak after dispersion gives us a value only 9% lower, again hinting our theory is correct.

We showed earlier our ability to manufacture unusual geometries, when we made hexagonal close-packed dishes for photolithography. Taking this one step further, we now move on to exotic beam formers with varying degrees of curvature.

### 6.6 Exotic shapes & offset troughs

Using the manufacturing technique we described in Chapter 3, the FIB can produce jet-forming structures of varying size, curvature and footprint. Whilst gathering light into a long light sheet could be useful to many areas, such as gathering light into a solar cell, there are many other places where light structuring into more complex shapes, at the wavelength scale, may be useful. As optical cold-atom traps have been demonstrated using structured surfaces of a similar scale to those we make[137], the beam formers may find a use in that area. We will now look at more complex structures, that form curved light-sheets and single point beams.

Figure 6.16 shows several of these shapes, including a rotationally symmetric post, a set of doughnuts, made of a set of inner and outer circular troughs, and an S-shaped structure. The features we discuss can be more clearly seen in Figure 6.15, where 3D models of the light forming structures are shown in cross section. The post, shown in a),
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Figure 6.14: Graphs of a) peak intensity b) Full Length at Half-Maximum c) Peak Intensity Length d) Full Width at Half Maximum for experimental data where the difference in refractive index was varied

and the doughnut, shown in b), are both circularly symmetric, and hence the removed half is the same as that visible. The S-shape is more or less shown in full, but with the ends of the trough left open, for visibility of the troughs. The doughnut and S-shape both form a light-sheet between the troughs, whilst the post forms a single beam above the structures centre.

Beginning with the post, shown in a), the set shows the microscope imaging from below the surface, to above the surface of the mask. The post is a rotationally symmetric half-trough, similar in many ways to the conical axicon lenses often used to form Bessel beams [132]. Figure 6.16 a) begins below the surface, imaging within the post. Moving up (still within the post) a tiny bright dot appears, showing focused light trapped within the glass post.

Moving past the surface (marked 0 in red) a single, bright point appears above the apex, which has a FWHM of $2.1\mu m$. The bright spot continues for a further $2\mu m$, before merging into the total light coming through the aperture. Whilst the FWHM is much larger than the wavelength in this example, our models in Chapter 5 suggest that fine tuning the apex size ($\sim 1\mu m$ in this example) and trough profile would allow us to push this down in size. A smaller FWHM would be of interest when coupling light into a
Figure 6.15: A set of cross-sectioned 3D models of the S-shape, Doughnut and Post beam formers, in cross section. The sections of the doughnut and post removed in the cross section are identical to that seen.

small aperture, such as the wave-guide example we modelled in Chapter 5.12, whilst a larger FWHM like the one produced here would be of more use in light trapping and manipulation of micron scale objects.

In Figure 6.16 b) is a pair of s-shaped troughs. The S-shape serves to demonstrate that these Bessel-like sheets can be curved, forming them from very exotic, arbitrary shapes, which may make them useful in various situations. Curved Bessel sheets may be useful in microfluidic optical traps, guiding particles in a suspension along a given pathway. As before, when moving above the surface the jet appears. In this case it follows the curve
of the two troughs perfectly to form an S. Moving slightly further from the surface to \( \sim 4 \mu m \) shows two additional bright points in the middle of the bends of the S. These form due to the light scattered from the innermost side of the inner trough, which overlaps due to the tight curvature of the trough.

Moving on to the image set of the doughnuts in Figure 6.16 c), a pair of concentric circular troughs of equal depth. As before, above the surface, marked +1 in the figure, we see jets turn-on, growing thicker as we move away from the surface of the structure. In Chapter 5.9 Figure 5.15, we showed that by offsetting the depths of two near-neighbour troughs, the angle of the beam can be steered. Here we investigate this experimentally. In Figure 6.17 we have plotted the profiles across three different doughnuts. The first is where the outer trough is twice as deep as the inner trough, steering the beam towards the centre. The second is for two troughs of equal depth (seen in 6.16 c). The third is from an inner trough that is twice as deep as the outer one, steering the beam away from the centre. In each case, the shallow and equal troughs were always 0.75\( \mu m \), whilst the deeper trough was 1.5\( \mu m \) deep. Modelling predicts a change in angle of the beam of around 5\( ^\circ \) from the surface normal.

In all three images the red profiles show images focused below the surface, whilst the black is above the surface. In each image two peaks can be seen below the surface for each jet, showing the back focal points of the troughs themselves. Moving above the surface a peak can be followed emanating from between the troughs, showing our jet.

In a), where the central trough is shallower, the paths of both light beams are directed towards the centre. The angle of the beam is 24\( ^\circ \) from the normal to the surface, significantly higher than the 5\( ^\circ \) predicted by our modelling.

The angle of the jets in c) is 34\( ^\circ \) towards the centre of the doughnut, as measured over the full 14\( \mu m \) above the surface. However, eliminating the last two points (where the beam has spread the most) reduces this angle to 15\( ^\circ \), and eliminating a further two puts the angle at 3.6\( ^\circ \) away from the doughnut centre. This shows that the angle of the beam is actually outwards for the first 10\( \mu m \), before moving inwards as the beam spreads.

In c), where the inner trough is the deepest of the two, the path of the beam appears very straight. The beam angle is 12\( ^\circ \) away from the centre over the length of the beam. However, as before, the beam angle is outwards for the first few microns, and then somewhat more inwards for the last few microns, caused by widening of the beams.

In all three images we saw an over-all tendency for the jet to angle towards the centre. For instance, a) is 24\( ^\circ \) towards the centre, whilst c) is only 12\( ^\circ \) outwards, even though the troughs are mirrored in cross section, and should direct the jet to the same angle.
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in reverse. This is also seen in the identical troughs of b), which also angled the jet inwards.

It appears the circular nature of a doughnuts (or other curved shape) directs the light towards it’s centre, similar to how the $S$-shapes of 6.16.b) gathered light within the inner bend of the troughs, instead of just between them. As our models were entirely run in two-dimensions, we could not have predicted this from them. In our theory section of Chapter 5.11 we discussed how the overlapping rays of light, refracted through the structure, form the beam. The nature of a three-dimensional curved trough means that rays passing through the inner-most curved region will overlap with one another, re-enforcing the light within the curve and angling the beam towards its centre. In Figure 6.16 b) and c) we can see this effect; a separate peak within the doughnut is visible, which eventually merges into the jet. This may or may not be useful as a control variable when forming light-sheets. As we now understand the effect, removing the contribution of curvature to the jet angle is as simple as creating a single tip structure, rather than a full trough, as we did in Chapter 5.8.

The doughnuts serve to show that control of the beam angle is possible, by controlling the trough profile, and curvature of the structure. As the models in Chapter 5.9 predicted, the light angles towards the shallower of the two troughs, but can be steered by more than the ±5° predicted. As the modelling worked well in the case of the conventional trough structures, to overcome this discrepancy between the model and experimental setup, we would need to adjust the models to include three dimensions. As we have highlighted before, the ability to direct the beam may be very useful for maximising the light under a non-transparent structure, such as a metal conductor in a solar cell or CMOS detector.

### 6.7 Reflowed Structures

The final investigation into this structure looks at the possibility for large scale manufacturing. Whilst all previous structure have been made from ion-milled transparent substrates, we wondered if a structure could be made by more conventional means, such as for CCD/CMOS lenses. Lines of photoresist were prepared via conventional photolithography 10μm deep by 10μm wide and several centimetres long on a single silicon wafer. The wafer was then diced into several 1.5 cm squares. Figure 6.18 shows an edge on view of of one of these wafer pieces, showing the square cross section of the tracks. The samples were then placed in an oven at 150°C for 40 minutes, which is just above the glass transition temperature of AZ9260, which is ~140°C (post-exposure) [138]. As can be seen in Figure 6.18 b) this has allowed the polymer to reflow, forming into droplets
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**Figure 6.16:** Sets of images taken through various geometry at different heights above the structure surface (noted in red). Images show a) a circularly symmetric post, b) a curved pair of S-shapes and c) a doughnut of two troughs.

**Figure 6.17:** Profiles across 3 doughnuts in the ratio of the trough depths were varied. In a) the outer is twice as deep as the inner, in b) they are equal and in c) the inner is twice as deep. The Peak intensity of each image moved towards the shallowest trough. In a) this is 24° towards the centre, b) 3.5° outwards, and c) 12° outwards. This shows the jet deflection can be controlled.

extended along one axis, similarly to how reflowed lenses were formed in our discussion of reflow, Chapter 1.2.1.4.

PDMS was then poured into the AZ9260 lines, and cold cured for 24 hours. When peeled from the surface the photoresist, the PDMS formed spherically curved troughs similar to our milled structure.

The troughs are 18µm diameter and 5µm deep with an apex width of 1.2µm. We can compare these troughs to our previous experimental and modelled examples by $f\#$. Using equation 2.5 from Chapter 2.2.1 gives a value of $f\# = 0.45$. This is comparable to the 3µm diameter by 0.75µm depth troughs, whose $f\#$ is 0.5.
We will need to bear in mind that the refractive index of the PDMS is 0.1 lower than that of the glass examples we’ve used previously, when discussing the data. In scale with the 3µm trough, the apex of the PDMS structure would be 200 nm, close enough to the 100 nm we often used for comparison.

![Image](image1.jpg)

**Figure 6.18**: Preparation of replicated troughs. a) photolithographically defined lines, b) lines after reflow c) PDMS replicant of lines, d) close up of a single track. Images a) and b) show the change in profile before and after reflowing. c) demonstrates that our replication process has successfully re-created our structure in PDMS.

Scanning a laser in 2D slices in x and y, moving the focus up in the z-axis builds up a 3D volume. Figure 6.19 a) shows a slice through one of these 3D volume images. In the image the green arrow indicates the tip of the apex. Above that we see a bright line, indicated by a red arrow, which is the light jet itself. The width of the jet is 1.038µm, which stays consistently thin for a full 10µm in distance. Comparing with our closest reference from the simulations (diameter 3µm, depth 0.75µm), the jet length was 10µm, with a width of 0.5µm. In both cases the jet was non-divergent over at least 10µm. Whilst the absolute value of the FWHM is lower for the smaller trough, the ratio of FWHM to the apex width is actually lower in the replicated structure. This leads us to conclude that the wavelength of light sets the limit of the FWHM. The similarities between the large (18µm) replica, and smaller (3µm) model indicate that the structure is scale invariant, which would indicated the f# is a good indicator of the structure’s light-guiding capability.
Below the tip of the apex is a bright area, found between the jets. This corresponds to the light reflected from the surface of the troughs, and below that the back focal-point of the trough can be seen (indicated with a yellow arrow). Image 6.19 b) shows a profile plot of the beam shown in a), demonstrating how thin the line stays.

Due to the differences in intensity seen when scanning through the xy planes, the microscope had to auto-adjust the light-sensors sensitivity. Therefore, the data for each xy plan was normalised by the area under the profile (total intensity), and so the image does not give the full picture of how the intensity drops off. This explains why the expected, Gaussian-Bessel z-profile is not seen.

Near the surface (the closest profiles in 6.19 b) the only peak visible is the single zeroeth order jet. Moving further from the surface, the jet width remains low, but very small, blue, peaks indicating noise appear in the surrounding area. These are more visible due to the normalisation process noted earlier. On the furthest left and right are additional regions of high intensity light. From 6.19 a) we can see that they are positioned above the centre of the troughs. In our models, Chapter 5.3, we saw that glass apertures, spaced similarly to our troughs form a diffraction pattern above the aperture centre. We noted this effect in several examples where troughs were used, and so can conclude these bright spots to be the diffraction pattern of the series of apertures formed by the troughs.

A higher quality confocal scan, as seen below in Figure 6.20 can more dramatically show the higher-order emanations. The image shows the PDMS replica placed trough-down onto a glass slide. Three markers show the level of three interfaces; 1. the top of the PDMS, 2. the PDMS/glass interface, 3. the air/glass interface. The difference in height between 2 and 3 is caused by the shorter path-length of light when travelling through the higher-index PDMS ($n \approx 1.41$).

The light around the apex of the troughs forms into two triangular shapes. The lower of the two shows the light just below the surface of the apex (found in level with marker 2), where the jet emerging from it can be seen. The higher triangle (above 2) is formed by light reflected from the surface of the curved troughs. We predicted such reflection in Chapter 5.11, caused by the curvature reaching the critical angle for internal reflection, whilst the structure not being idealised to use total internal reflection.

Around the lower of the two triangles, very weak lines can be seen coming from the trough region. Again these show light internally reflected off the trough that is scattered away from the jet.

The inset shows a single jet from the same data set, where the intensity of each z-slice has been normalised. This highlights the beam (indicate by the red arrow) and the
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Figure 6.19: Low resolution 3D laser scanning confocal image, demonstrating the jets (red arrow) emerging from the apex of the structure (green arrow). Below the level of the green arrow, between the jets, is a bright area of reflected light, below which is the back-focal point of the troughs (yellow arrow). The plot shows the intensity profile of the jet, demonstrating that it stays thin for a long distance (40+µm).

emanations below the beam. The beam itself is thin (2.1µm), indicating non-divergent, for a distance of ~13.5 microns, after which it diverges at a low angle (~7°).

The confocal data has shown the Bessel-like nature of the beam, and demonstrated the scattering of the light from the trough, as we suggested would happen in our theory section of Chapter 5. Having discussed our experimental results, we now need to conclude this chapter.
6.8 Conclusion

In this chapter we have experimentally demonstrated the ability of our curved axicon lenses to form a Bessel beam.

We began by manufacturing glass and sapphire photo-masks for UV photolithography, using the techniques described in Chapter 3. Each mask had various structures on them, including a 3D analogue of our 2D-modelled data, a set of three troughs. Alongside these were close-packed hexagonal dishes. As we described, other shapes were also made into similar masks, including single posts, doughnuts and S-shaped bends, but these were directly imaged in a transmission optical microscope, and not used in photolithography.

When using the masks for photolithography we showed that light passing through the three-trough structure can structure light to preferentially expose resist. The resist was most heavily exposed directly below the metallised tracks between the troughs. From the modelling discussed in Chapter 5 we surmised that the structure was forming a thin light-sheet. An atomic force microscope profile of the photoresist showed the resist had been most exposed directly below the two tracks between the troughs, but had also been exposed outside of the area of the mask, corresponding to first, second and third order jets surrounding the zeroeth order. Equating the depth of the resist to light intensity, we saw that, in profile, it was similar to a higher order Bessel beam of the first kind ($J_n$, where $n \geq 1$).

Comparing a set of intensity profiles, taken from images moving through the focus through the mask, a three-dimensional intensity image of the light moving through the three-trough structure was built up. When compared to the computational model of...
an identical structure, the intensity graph looked almost identical to the modelled data. The x and y profiles across a jet agreed with the beam being Bessel-like in nature, with the profile seen in the resist conformed to the intensity.

The photolithography of the close-packed hexagons showed a very complex pattern formed in the photoresist. This pattern was due to light-sheets forming between the dishes, along with a myriad of higher order jets. The strongest jets had formed at the meeting points between the dishes, but variation in the resist/mask distance caused the pattern surrounding the mask area to change. For a small mask-resist distance, intense light sheets were formed between the dishes, similar to those seen between the troughs. For larger mask-resist distances single jets formed at the meeting points of the dishes, creating patterns of dots. Whilst complex, a modified version may be a useful method of splitting a signal to couple into an optical fibre bundle.

As the initial idea for the study had been to test a method of performing sub-wavelength photolithography, we were disappointed that our initial tests had not created sub-wavelength structures in the photoresist. However, as the resist was exposed in proportion to the light intensity, it may be possible that with further development of the structure, as well as control of the mask/resist distance, would allow a sub-wavelength structure to be exposed.

Next, we looked to experimentally compare our data with our modelled data in a more robust way. By varying the refractive index of the mask & medium, we intended to see whether our prediction that the difference in refractive indices of the mask material and the surrounding medium controlled the formation of the jet, and not the absolute values of the two indices. We looked at two mask materials Glass (n=1.52) & Sapphire (n=1.77) in various media, including Air, Water (n=1.33) and Glycerol (n=1.47), to look at how the difference in the refractive index affected the gathering ability of the structure. This gave us a comparison with our work on refractive indices found in Chapter 5.7

We discussed each image set individually, starting with the highest refractive index change, sapphire in air ($\Delta n =0.77$), and finishing with the lowest, glass in glycerol ($\Delta n =0.05$). Whilst each material and medium formed jets, they grew less distinctive as the refractive index difference, $\Delta n$, decreased. The images taken in water showed dispersion, due to the microscope lens not being idealised for liquid immersion. The sapphire-air example showed clear peaks which merged more quickly than expected from the model. The sapphire-water example showed strong chromatic aberration, caused by dispersion, seen as the peak splitting into several colours. Glass-water had the steepest drop in intensity, with the jets merging into the background after only $4\mu m$. The glass in glycerol mask, having the lowest value of $\Delta n$, barely formed jets, but looked very similar to the modelled data. The visual comparison between the modelled and
experimental data indicated that the models gave an excellent representation of how light passed through the structure, suggesting that data taken from the models could be used to calculate the structure needed to form a jet of given properties.

The plots of Intensity, FLHM, peak intensity length and FWHM trends were somewhat different to what we anticipated from the simulations. The Peak intensity had a slightly increasing trend with $\Delta n$, which the $\Delta n = 0.44$ (sapphire-water) did not fit. From this we concluded that the peak splitting due to chromatic aberration, seen in the sapphire-water example, had reduced the peak-intensity by spatially separating the total peak intensity. By adding the contributions of the coloured peaks we concluded that the jet intensity would increase from 76% to 91% of the value of $\Delta n = 0.52$ (glass-air), which would better fit the expected trend.

When examining the FLHM, we saw that the intensity in the jets did not drop below half peak intensity before they began to merge. As the FLHM is defined as the Diffraction Length of the beam, the length over which the beam is non-diffracting, we added an additional measure, the jet length before merging. Whilst the measurement of FLHM showed that higher values of $\Delta n$ had very long beams, agreeing with the simulations in that respect, the true distance before merging was lower in every case, except for that of glass-air where the beams faded before merging.

When we discussed the modelled data, in Chapter 5, we concluded that jets with a higher peak intensity declined at a steeper rate, leading to a shorter measurement of FLHM. In general this caused us to find less intense jets being measured as longer. However, in our experimental measurement of the jet length before merging, we saw a trend towards increasing length with increased gathering power (higher $\Delta n$). In this case we can only conclude that by introducing a medium the light in the jet is scattered in the medium itself, increasing the divergence angle and causing the beams to merge. The modelled data assumed a perfectly uniform medium with no absorption or diffuse scatter, hence could not have predicted this effect.

In the experimental data we saw that the FWHM did not drop below the wavelength. However the glass/air example did exceed expectations, the FWHM was measured to be 773 nm, whilst modelling predicted a minimum 900 nm width. This suggested that, by tweaking the trough profile, a sub-wavelength width jet would be possible.

Our final conclusion on our theory that $\Delta n$ controlled the jet properties was that whilst the data was not of high enough quality to conclusively prove it, there were hints that it is correct.

The post, $S$-shaped structure & doughnut demonstrated our ability to manufacture arbitrary structures, with high curvature, that can form single point beams or curved
light-sheets. The post formed a 2µm long jet with a FWHM of 2.1µm, both wide and short for our structures, however at 1µm diameter, the apex was significantly wider than ideal for beam forming. The S-structure demonstrated curvature along the plane of a light-sheet. Such a structure may be useful in unusual optical tweezing and light scanning setups. Whilst the light sheet had good FWHM and length, additional gathered light was seen within the centre of radius of the curving trough pairs. This lead us to conclude that curvature in the third dimension, unaccounted for by our models, focuses the light into and additional beam or spot.

Using the doughnuts we tested our modelled prediction that offsetting trough depths allows control over the beam deflection. Chapter 5.9 Figure 5.15 b) suggested that a ±5° deflection should be possible with the trough profiles we used, with the beam deflecting towards the shallower trough. The experimental measurements of the doughnuts confirmed that the beam always deflected towards the shallower trough, angling between +34° to -12°, significantly greater than the model predicted. Both the increase in deflection angle, and the directional preference towards the centre of the doughnut suggest the third-dimensional curvature played a role in the beam angle.

The doughnuts had proved that control of the beam angle could be achieved via offsetting the profile of the two troughs. Along with the post and S-shape, curved light-sheet formers could play a useful role in optical tweezing, allowing small objects to be guided along extended light paths acting like tracks.

The final piece of work in this chapter showed a scalable process for manufacturing our structure, using a simple, two step, reflow and replication process. This overcomes ion-beam lithography’s size limit, and makes the structure industrially viable to integrate into other devices. When examined via confocal microscopy we found that our 18µm diameter by 5µm deep trough structure formed jets with similar properties to that of a smaller structure with the same f# we had modelled previously. This included the jet length (10µm) and width (1.1µm), but the higher order jets were not present in the lower quality images. High-resolution confocal clearly showed the internally reflected light forming an intense region within the structure, as well as scattering out of the troughs, and being lost to the jet. We predicted these two mechanisms of loss when discussing our theory in Chapter 5, noting how a steeper trough profile (deeper trough, smaller f#) creates more internally reflecting angles causing more light to be lost.

We have manufactured curved micro-Bessel beam formers using both ion-beam and reflow based techniques. As part of this work we have modelled their formation and compared this to real experimental examples. From the work we have concluded we can predict how the profile and refractive index of the structure will affect beam formation. Such structures could find uses in many areas, including in optical tweezing and atom
trapping, coupling of light to and from optical fibres, and fluorescence microscopy, where Bessel beams are often used to control the light/sample interaction to perform sub-wavelength imaging[139].
Chapter 7

Conclusion & Future Work

Over the course of this thesis we have looked at two, closely-linked areas: Micro-lens super-resolution, and micro-optic beam forming. The linking factor is our manufacture technique. By using an ion-beam microscope, along with a replication technique, we can produce very unusual geometrically precise lenses. We described our manufacture technique in Chapter 3, touching briefly on how it could be expanded into a larger scale production method.

Therefore, conclusions need to be drawn on three areas: the manufacture of micro-optics with wet-etching of substrates; the characterisation of super-resolution micro-lenses; and the characterisation of curved Bessel-beam formers. We will take each in turn, and then draw the conclusions together with future work that may be carried out in each area, and how they may be used together.

Before we begin the concluding discussions, it is worth summarising the output of this thesis. Currently one paper has been published (Langridge et. al, Micron[9]), and two patent applications have been filed (GB1418180.4) & (GB1415763.0), both of which are subject to the earlier patent describing 3D milling (WO2010112827 A2). The paper details the manufacture of 3D geometry using a focused ion-beam microscope, along with chemical etching to remove ion-beam damage (as we described in Chapter 3). The first patent application (GB1418180.4) describes the replication of 3D milled geometry to form micro-lenses, as we described in Chapter 3 and characterised in Chapter 4. The second patent application (GB1415763.0) describes a curved axicon structure used to form extended quasi-Bessel beams, as we details in chapters 5 & 6.
Chapter 7. Introduction

7.1 Manufacture

Beginning with our manufacture method, we described how to produce curved surfaces using a focused ion-beam microscope. Our method mathematically defined the curved profile by radially splitting the pattern into concentric, overlapping shapes, and giving each an increasing depth for each larger radius such that the summation of the ion dose in each of the concentric shapes defined the profile. A parabolic profile defined this way has a linear increase in the dose between the entities, whilst a spherical profile requires non-linear ion-dose steps.

As the method is based on concentric entities, the footprint as well as the profile can be easily modified, allowing for the manufacture of reflecting troughs, hexagonal close-packed arrays, and highly-curved trough pairs, such as those seen in the S-shapes in Chapter 6.6.

This method, compared to the conventional methods of micro-lens manufacture, such as polymer reflow, has several distinct advantages, and only one major drawback. Reflow is able to produce large areas of lenses, positioned via conventional lithographic methods and then reflowed to form spherical-profile lenses of either circular base, or in long tracks (as we demonstrated in Chapter 6.7). Our method produces a smaller numbers of precision lenses, but they can have of any profile or footprint we can mathematically define. Whilst the lenses are of extremely high-quality, with low roughness and excellent conformity to the intended profile, manufacturing large numbers of lenses on a single film is a slow process, requiring serial milling of the moulds.

As part of our discussion of FIB in Chapter 2.4 we noted the most recent development in the field of ion microscopy, Ion-Plasma microscopes. The exceptionally high mill rates would allow a mask for larger scale lenses, or a larger number of lenses, to be produced much more quickly than is currently possible. As the milled mask is replicated, and can be considered tooling in the manufacturing process, this should improve the speed sufficiently to allow for large scale mass manufacture. The ideal scenario would be to produce a single, large array of varied lenses, on a significantly greater scale than the 100 or so lenses produced in our current pattern, and produce multiple PDMS replica moulds from the original master. This would allow thousands of sets of lenses to be produced per day.

On a smaller scale, we have shown that a lens film can be produced from a single, silicon-rubber mask within 2 minutes, and that the mould does not degrade even after 50 uses. A small scale setup to produce and sell thin-film lenses could potentially manufacture a hundred films per day, adequate to supply a small market of specialist microscopists.
This means that, depending upon the demand, the process of lens manufacture is entirely scaleable. We would expect such demand to come from biological & materials researchers who work at the limit of optical microscopy, and often have to resort to electron microscopy. In Chapter 4, where we characterised the optics of these lenses, we demonstrated imaging below the diffraction limit of light using these lenses. We clearly resolved 200 nm objects and spacings, along with the 100 nm ridges of a Blu-ray disc. Magnifications of between 1.2-2.5x were observed using our micro-lenses. This included a 1.36x magnification of a fixed yeast cell, and a 1.2x magnification for live yoghurt bacteria in water. This magnification could be improved upon, as we discuss further on, granting biologists a system for studying living viruses, in-vitro, by imaging beyond the Rayleigh diffraction limit of the optical microscope.

When characterising the roughness of lenses produced by this method in Chapter 3.3.6, we showed that it increases from 4.0 nm in the original, as-milled silicon mould, up to 11.7 nm in the final PMMA lenses. This leads to 1% scatter of light from the lenses, due to roughness, at a wavelength of 147 nm, well beyond the point of 100% absorption in the polymers we use. This proved our optics were of a suitably high-quality for their intended purpose.

When defining our manufacture process, we discussed a method of wet-etching to remove ion-beam damage, which we published in Micron[9]. We showed, using atomic force microscopy, ion-range simulations and scanning transmission electron microscopy, that hydrofluoric acid etching of ion-beam exposed silicon removes the amorphised silicon down to the 31% amorphous material (for a 30 minute etch in 48% conc. HF). The aim was to remove not only the amorphous silicon, but the implanted gallium within the layer, which is the hardest part of healing ion-beam damage.

We discussed how, by increasing the ion dose when milling silicon, it increases the surface amorphisation, allowing the chemical etch to remove more of the surface, so that more of the implanted gallium is removed along with it. By tuning the ion milling dose, the amount of gallium left within the surface after etching can be minimised. The fewest ions left within the surface occurs at a dose of 3000 \(\mu C/cm^2\), leaving less than 0.5% remaining within the surface. Even when increased above this dosage, up to 20,000 \(\mu C/cm^2\), the number of gallium ions left behind by the etch was lower than the doses below 3000 \(\mu C/cm^2\).

This information from this study could be useful in many parts of ion-beam microscopy. We showed the huge extent of the ion-beam tails, at greater than 50\(\mu m\) area for a 33 nm spot diameter. From a qualitative test of the etch time, by testing the adhesion of PDMS, we saw that the duration of the etch changed the colour of the silicon, indicating a variation in the remaining layer thickness (Chapter 3.3.5, Figure 3.13). The methodology
adopted in this chapter could easily be applied to control the remaining amorphisation of the surface, and the ion percentage within it, to control the surfaces optical and electrical properties.

7.2 Super-resolution imaging

Our work on super-resolution imaging falls within a new, up and coming area of research that has been called Micro-sphere Optical Nanoscopy, although we prefer to drop the specification of sphere, opening the area to all optics. As the area is so new, there are still some big questions remaining to be answered. In our journal review we highlighted a few of these, including: how to form the micro-sphere or lens optics into a simple, workable system for general purpose use; what is the difference in resolution, magnification and contrast between a sphere and a lens; and what is the fundamental mechanism for imaging beyond the diffraction limit?

To overcome the drawback of positioning the lens with respect to the sample, two micro-optic rigs were built, one with piezo xyz control of the sample, and the second with piezo xy, and micrometer z. Both systems have been shown to work well, allowing a single feature to be viewed with three different thicknesses of lenses, as well as showing magnified and regular images of single features. The ability to move freely in the x, y & z axis have been crucial to the work in Chapter 4, where we characterised the micro-lens/microscope system.

When comparing a sphere to a lens, such as we produce, there were a number of characteristics to consider. The spheres are simpler to define, having only a diameter and a refractive index to define the focal length of the sphere. The lenses have separate diameter and depth, along with the refractive index, allowing the focal length to be freely tuned.

We saw in Chapter 2 that, in conventional geometric optics, a lens forms a virtual image when the object is closer than the focal length, with the maximum magnification occurring when the object is almost at the focal point. During Chapter 4 we saw several indications that this is what we were also seeing using micro-lenses.

The difference in magnification observed between a lens and a sphere was small, but present, where on average the sphere magnified to a higher degree. The lenses showed between 1.2-2.5x magnification, depending on the sample, averaging closer to 1.5x. The spheres demonstrated 1.8-2x magnification showing a greater average magnifying power, which agrees with what has been observed across various journal papers.
For lenses & spheres that were often in direct contact with the surface, or near direct contact, the focal point of the sphere sat only a few tens to hundreds of nanometres below the sample surface, minimising the object/focal point distance and maximising the magnification.

The focal length of a lens, whether spherical or parabolic in profile, is significantly longer, as confirmed by both theoretical calculation and direct observation with confocal microscopy in Chapter 4.6, Figure 4.19). This pushes the focal point into the sample surface and creates a greater distance between the focal point and the sample, minimising magnification. In this section we confirmed that our derivation of the equation for the focal length of a plano-parabolic lens is correct, however our modelling predicted a focal length over twice the value experimentally measured. Correction of this will be discussed in our section on further work.

Much of the work we did with lacy-carbon grids varied this surface/focal-point distance, which we demonstrated by observing the variation in the magnification. Changing the sample/lens distance on a chequerboard pattern (Chapter 4.3) showed an increased magnification as the distance decreased, suggesting we were imaging a real image, and outside of our super-resolution zone. With a lacy-carbon grid, we showed that changing the micro-lens/objective lens distance also changed the magnification, allowing the micro-lens to be thought of as a part of the microscope’s optical setup, such that a change in the distance between any two lenses changes the ultimate focal length.

In our journal review, we highlighted the reflow work of Vlad et al.[49], who reflowed spheres and created lenses, and lenses on pedestals. The addition of the pedestal lowered the surface/focal-point distance, and increased the magnification. Adding a pedestal arrangement to our lenses would be very simple. Due to the linear depth/dose relationship we discussed in Chapter 2.4.4 all that would be necessary would be the addition of a single circle the size of the lens, with the dose set to mill the required thickness of the pedestal. This would allow a huge amount of variation in the pedestal thickness, allowing for accurate testing of the sample/focal-point distance in direct contact with extreme accuracy.

For the same reason, increasing the lens depth should also increase the magnification factor, as indicated by our experiments in Figure 4.7. This gives two ready modifications that can be made to improve our system. The advantage of increasing the lens depth is that the FWHM of the focal spot should be smaller. As we noted in our journal review, the sub-wavelength size of the focal spot from spheres is suggested to play a role in the recovery of the evanescent waves. A smaller lens focal spot should therefore increase the resolution.
When characterising the lenses, we also investigated the contrast of sub-resolution features. We saw that the contrast and magnification showed a tendency to increase and decrease together, suggesting a fundamental link between the two. This could hint that the resolution of the lens is controlled by its magnification, as the contrast is key to determining if an object is resolved.

During the chapter, we observed some Barrel distortion at the edge of the parabolic lenses, where the magnification decreased with distance from the optical centre of the lens. This was most evident in our confocal scan through a lens (Figure 4.17) and our polarisation angle example (Figure 4.13), where the distortion actually changed with the angle of the light. Whilst our parabolic lenses were very precise in profile, it appears a parabola may not be best suited for a micro-lens. A paper by Guo et. al.\cite{62} in our journal review highlighted that the spherical aberration at the micro-scale is counteracted by diffraction from the lens aperture, suggesting a parabolic profile is not entirely necessary to remove the aberration.

In future work, it may be worthwhile to move to the more common form of modified-spherical profile use in macro-scale aspheric lenses. These also reduce spherical error when correctly milled, but may reduce distortion at the lens edges. Due to our manufacturing process being very adaptable, all that would be necessary to move to such a profile is to calculate, from the radius of curvature, how to subdivide the new profile into discrete quantities.

If we continued with parabolic lenses, at least with confirmation of the focal length, the magnification could be tuned for direct contact, as was most often used.

### 7.3 Micro-Bessel beam formation

Using our manufacture technique for curved profiles structures with unusual footprints, we demonstrated a curved axicon Bessel-beam former. We demonstrated, using extensive models and experiments, how a beam is formed through interference of two overlapping beams. The structure could be formed of a pair of troughs, side by side, or a rotationally symmetric post, both of which were demonstrated in our experimental chapter, 4.

Bessel beam formers are conventionally used in two key areas: biological microscopy, where light sheets are scanned through samples or beams used to inject particles into cells; and micro-manipulation, where Bessel beams are commonly used as optical traps for small particles. These are usually made by large scale (few mm) axicon lenses, and require a Gaussian incident beam.
We demonstrated, by modifying the depth of a set of troughs, that the beam’s *diffraction length*, peak intensity and *full-width at half-maxima* can be controlled, to form a beam with a width $2/3$rd of the wavelength, and a non-divergent length of 17 or more microns. We showed that as the peak intensity within the beam increased, the length of the beam decreased, caused by a steeper drop off in intensity.

The differences between our structure and a conventional Gaussian-Bessel beam created by a flat-profile axicon are the intensity and length of the beam. In a conventional axicon lens, the profile angle of the axicon, along with the illuminating beam radius (limited by the axicon diameter) define the beam’s *diffraction length*. Unless the axicon angle is very shallow, the length of the beam is usually smaller than the beam diameter. An angle of $2^\circ$ or lower is required to reach the incident beam diameter, and commonly very few angles lower than this are available.

In our curved structure, the beam length has been shown to regularly reach close to 6x the diameter of the structure. This measurement from our model is actually limited by the size of the computational cell, and could be significantly greater.

By curving the axicon, the incident angle of the light on the interface between the high & low refractive index materials varies radially from the structure centre. In the ideal situation, the path of light passing through the centre of the lens-structure is changed only slightly, whilst at the lens edge it is more dramatically directed towards the peak intensity. It is the overlapping of the central beams that creates the very long *diffraction length*, whilst maximising the peak intensity.

It also has the advantage of creating a Gaussian-Bessel beam from an incident plane-wave, rather than a Gaussian wavefront. This dramatically simplifies the optical setup required before the beam former.

In our models, we demonstrated that the amount of light gathered into the path of the beam increases with trough depth, up until the angle of the trough becomes too steep, and *total-internal-reflection* occurs. This leads to light loss and a reduction of total transmission. This means exact control of the trough profile is needed, to minimise beam width and maximise length, whilst allowing maximum transmission.

By introducing metal covers within our computational models, we confirmed that most of the light travelled through the troughs, rather than through the apex, as would be expected from a total-internal-reflection effect.

We modelled and experimentally tested the effect that the refractive index played on the formation of the beam. From our computational models, we concluded that the strength of the refractive index change ($\Delta n$) controlled the light gathering ability of
the curved axicon lens. The lowest values we tested, ($\Delta n = 0.05$ experimentally or 0.1 computationally) showed that very little light is gathered into the beam where only a small change is present. The models showed that whilst the absolute transmission through the structure decreased with an increasing value of $\Delta n$, the intensity within the beam increased up to $\Delta n = 0.8$, before decreasing. This suggested that the gathering ability of the structure was increasing, until too much of the structure was above the angle for total internal reflection.

Due to a lack of high enough quality experimental data, we could not conclude definitively whether the refractive index change was the controlling factor, however our data did suggest this may be the case.

As a separate idea, we tested offsetting the depth of the two troughs that form the beam, in principle creating an asymmetric beam former. We demonstrated both computationally (Chapter 5.9), and then experimentally (Chapter 6.6) that this allows the beam angle to be deflected, giving another aspect of control over the beam path. The computer models predicted $\pm 5^\circ$ deflection, whilst the experimental results showed more like $\pm 23^\circ$. This suggests that some effect, such as the gathering angle of the microscope, was unaccounted for by the model, but is an exceptionally good result if interested in angling light around a corner, as might be the case for an optical detection layer buried beneath a metal contact.

When it comes to uses for the structure, several have been suggested over the course of this thesis. In Chapter 5.12 we modelled its use in coupling an optical fibre to a waveguide. The coupling of light to and from optical waveguides has been noted as one of the major areas holding silicon photonics back from wide-scale integration into current electronics. The difficulty in precisely aligning the few micron core of an optical fibre with a few hundred nano-metre waveguide is huge, and even a slight misalignment can lead to several decibel loss in signal.

We showed a non-ideal 5$\mu$m glass optical-fibre, butted against a 200 nm waveguide, and then again with our beam-forming structure milled into the fibre. The transmission into the waveguide for a 1.5$\mu$m wavelength was 30% without the structure, and 49% with. Whilst in a real waveguide coupling scenario, an efficiency of 90% or higher is aimed for, our structure is not idealised for the wavelength, and so wastes a lot of light. If this were to be used in a real example, further modelling to characterise a longer-wavelength structure would certainly improve the coupling efficiency. If the beam width can be idealised to well into the sub-wavelength regime, the coupling efficiency would improve dramatically, making the structure sought after.
Experimentally, in Chapter 6.7 we showed that the structure can be produced on a large scale by photoresist reflow and replication. As the materials are polymers and the methods are conventional, such a large area method could very cheaply be used to provide light guidance into a detecting structure, such as a CCD or a solar cell. Both electronic devices are mass produced on a massive scale, so any improvement must come at a tiny price per square metre.

To summarise the work in this thesis, we have detailed a method of producing extremely high-quality micro-lenses & curved micro-axicons for sub-wavelength illumination and imaging. The work within this thesis has been covered in one-journal paper, with subsequent papers to follow, along with one full-patent, and two patent applications (The author is a named inventor upon only the two patent applications).

7.4 Future work

Several areas of research discussed within this thesis would benefit from further research and discussion. We note a few of these areas, including altering the lens thickness, correcting mistakes in the computer simulations and incorporating our designs into a microfluidic system.

7.4.1 Lens Thickness

During our micro-lens characterisation, in Chapter 4 we noted how thicker lenses, with shorter focal lengths magnified the sub-wavelength features to a greater degree. We hypothesised that the magnification was the factor controlling the contrast and resolution limit of the lens. Should this hypothesis ever be verified, fine control over the focal position of a lens would be crucial to maximising its imaging ability.

We noted in our conclusion above how Vlad et al.[49] made lenses on posts by reflow, and how it would be easy for us to do the same. To take this work further, creating a mould with a large number of lenses, with varied diameter, thickness, and pedestal thickness would allow us to test the effect of the surface/focal-point separation to a highly accurate degree.

With the equation for the parabolic lenses focal length confirmed, and the few-nanometre accuracy in milling available using the FIB, we could create lenses of a given thickness, on pedestals of a known height, replicating the work seen by Vlad et al.[49] but with
the ability to define exact properties. This would give us control over the sample/focal-point separation to within a few tens of nanometres, allowing us to map it against the magnification and contrast for a number of small samples.

7.4.2 Lens Model Correction

In Chapter 4.6, where we computationally modelled the focal length of our plano-parabolic lenses, we saw an anomaly, in that our simulated data predicted 2-3x longer focal lengths than the analytically derived theory predicted. Once we confirmed that the derived theory agreed with experimentally measured data, it left the FDTD modelling data in question. Having confirmed that no human error was made when setting up the control file, it suggests an unknown fault in the FDTD modelling.

As our very similar models, used for the optical beam forming structure in Chapter 5, agreed very well with the experimental data that followed in Chapter 6, it suggests that the modelling software itself was not at fault. To extend and correct this work, a thorough investigation of the scripts used to run and then extract data from the models would be necessary. If the model was corrected, such that the results could be relied upon, further conclusions could be drawn in parallel to the spherical discussions seen within our journal review. By extending the models to output the Poynting vector, we could further form a discussion on exactly how the diffraction through the lens affected the focal properties. Throughout Chapter 4 we saw distortion at the edges of our parabolic lenses. Correct modelling may be able to predict the origin of the distortion, and indicate how best to correct the profile of the lens.

7.4.3 Three-Dimensional Curved Axicon Lenses

We looked at curved profile beam-formers which additional curved in the third dimension, examples being the doughnuts and $S$-shapes, seen in Chapter 6.6. We saw that the addition of curvature in another dimension, also concentrated light, in a way which we had not predicted. Our models in Chapter 5 had been entirely two-dimensional in nature. It would be useful to expand the modelling and experiments into the third dimension, to predict the nature of this additional light concentration allowing it to be either removed or incorporated into the useful beam.
7.4.4 Continuation & Micro-fluidic Incorporation

As a continuation of this project, we will look to incorporate both the super-resolution micro-lenses, and the Bessel beam formers into a micro-fluidic unit. Figure 7.1 demonstrates the core ideas behind the project.

A PDMS micro-fluidic structure will be formed using replication techniques, to copy ion-beam manufactured optical structures. Figure 7.1 shows a small particle A, entering a fluidic channel with two outlets. By employing differential pressure between the channels, the particle should preferentially flow straight downwards, and out of outlet E. When a beam former, shown as a yellow line and marked B, is illuminated, the particle should be controlled by an optical tweezing effect, and prefer to flow into the second channel. Here, a second horizontal beam former, indicated as D, with the beam shown in red, traps the particle below a microlens sitting above the observation chamber. This will allow particles in fluid to be selected for, and then observed under a micro-lens, in a fluid.

The uses for such a device are numerous. The device itself could be used as a biological analysis suite, whilst the individual components, including the particle selector and observation chamber, could be incorporated into a variety of other microfluidic devices.

*This is not the end, it is not even the beginning of the end, but it is perhaps, the end of the beginning.*

Winston S. Churchill
Figure 7.1: A schematic of a micro-fluidic device incorporating both the lenses and beam formers made in this thesis. A small green particle, indicated A, floats down the micro-tube, its direction vector is indicted by a red arrow. The yellow line, indicated B, is a curved light sheet formed by rear-illumination of a curved structure in the bottom of the tube. When illuminated the particle preferentially drifts down the left tube, controlled by an optical tweezing effect. This allows selection of small-scale particles. C indicates a micro-lens sitting on top of an observation area. Within the observation area is a planar beam-former used to capture particles under the lens.
Appendix A

Appendices

This section contains work that has been excluded from the main body of the thesis. This includes minor experiments and details of scripts and runfiles used during computational modelling.

A.1 Chapter 3 Appendices

A.1.1 Poorly Poured PDMS

During our experimentation with replication of PDMS lenses, described in Chapter 3.3.6.1, we noted that using an improper pouring method when depositing the PDMS directly onto silicon trapped air, misshaping the mould. Here we show a similar set of moulds to that made in Chapter 3.3.6.1, where the PDMS was poured directly onto the dishes, trapping air within them. Figure A.1 a) shows transmission optical images of the set of lenses, including histograms on and off the lenses, showing that they do concentrate light. A.1 b) shows an AFM heightmap of the lenses, demonstrating how the trapped air-bubble mis-formed the lens into a concave lens.

A.1.2 PMMA spin speed

To determine and calibrate the layer thickness, PMMA was spun onto flat borosilicate glass, and then scratched (removing some of the PMMA). This allowed us to use a profilometer (Tencor Instruments, Alpha-step 200) to measure the layer thickness. The machine works similarly to an AFM, in that a tip is dragged across a sample to measure height, but works for a greater height ranged (200µm compared with AFM’s 3µm ). The PMMA was spun between 4000-9000 rpm, keeping the spin time at 16 s, and acceleration
Figure A.1: Images of PDMS lenses misformed due to air bubbles. a) shows a transmission microscope image (labelled A), along with intensity map (labelled B). Below are two histograms showing the intensity within (i) and outside (ii) of a lens area, confirming the ‘lenses’ do concentrate light. Figure b) shows an AFM heightmap of one of these lenses, showing how an air-bubble mis-formed it into a concave, rather than convex, lens.

at 660 rpm/s. Figure A.2 shows a plot of thickness vs spin speed for PMMA. As with most photoresist, the thickness decreases dramatically as we move towards the higher spin speeds, before reaching a minimum level. Unusually the highest spin speed appears to create a thicker layer at 9000 rpm than 6000-8000 rpm, suggesting the higher speed forces material to gather at the edge.

Figure A.2: PMMA spin speed thickness curve. Shows the thickness varies from around 750 nm (at 4000 rpm) and drops to 625 nm (at 6000-8000 rpm)
A.2 Chapter 4 Appendices

This section contains experimental data not included in Chapter 4, for the sake of brevity.

A.2.1 Blu-Ray false-positive

When first examining micro-lenses on Blu-ray under the microscope, we saw what we believed was the ridges of the disc, shown in Figure A.3. However, unlike the micro-sphere images in Chapter 4.2, exactly two lines were seen in every lens, with no variation of position within the lens. If we compare the image to a later image, Figure A.4, where a set of our micro-lenses are in contact with a Blu-ray disc in a similar arrangement, we see real, magnified lines. In this image, the Blu-ray lines are close to the angle of the grid arrangement of the lenses. 3, 5, 7 and 10µm lenses are shown where the deepest is depth/4, and they reduce in depth in 50 nm steps.

![Figure A.3: Micro-lens false-images, taken under 20x microscope objective (NA 0.4). The lines shown in every image have been confirmed to be at a different orientation to the true blue ray lines (which run at ~45°, in a similar direction to the long axis of the grid of lenses.)](image)

By turning the sample with respect to the microscope, and seeing the direction of these lines unchanged, we can tell that they are a false positive. This tends to only be seen under a lower power lens (20x magnification). We believe them to be a Moire pattern, caused by the combination of the Blu-ray ridges and some part of the microscope. Whilst it was suggested the grid-structure of the imaging CMOS chip, the lines can be seen through the microscope eye-piece, and hence we conclude it more likely that a square aperture within the microscope being the cause.
Appendix

Figure A.4: Shows images of Blu-ray lines through a set of 7µm lenses at two angles, confirming the lines are real. a) shows the lenses at 0° with respect to the microscope, b) shows them after a 90° turn. As they turn with the sample, unlike the false positive in the previous figure, this confirms the lines are real.

A.2.2 Dish Confocal

The confocal XZ scans in Chapter 4.6 showed the path of light traveling through a lens. Here we show additional confocal performed upon micro-reflective silicon dishes. The scans were taken just above the surface of a silicon sample, and show the focal points of three 10µm dishes. The scale bars are not uniform in both x and z, and hence are indicated in image b). The focal length of each of the dishes is a) 5.16µm, b) 8.33µm and c) 11.0µm, which are close to the values calculated for lenses of their respective depths (1.25µm, 0.75µm & 0.5µm).

Figure A.5: Confocal XZ of reflective dishes, shows the light pattern formed above three milled silicon reflective dishes. The wavefront leading up to the focal point is curved, caused by diffraction.
A.3 MEEP programs

In this section, we show some of the MEEP control files & matlab scripts used in chapters 2, 4 & 5

A.3.1 MEEP control files

The control file detailed here has been used to run the failed lens models in Chapter 4. An earlier version of this script has also been used to run the full set of beam-former simulations run in Chapter 5, as well as a number of other scripts. The various geometric shapes tested throughout this thesis can be seen within this MEEP control file. It has been commented to allow for easier reading.

; MEEP control file for running low and high resolution simulations of LENSES and DISHES

; resets all the variables in meep
(reset-meep)

; The following section defines the COMPUTATION CELL
;; PHYSICAL DOMAIN DEFINITIONS ===============
(define-param sx 48) ;; physical domain size x
(define-param sy 24) ;; physical domain size x ; normally 24
;; PML x and y are the perfectly matched layers that form the BOUNDARY CONDITIONS
;; Here we set the size for each PML layer.
(define-param pml_x 1) ;; pml x
(define-param pml_y 1) ;; pml y
;; This adds the PML to the X and Y size to create the complete COMPUTATIONAL CELL
(define-param Com_Domain_X (+ sx (* pml_x 2))) ;; computational domain size x
(define-param Com_Domain_Y (+ sy (* pml_y 2))) ;; computational domain size y

; create the lattice in x and y (but not z)
(set! geometry-lattice (make lattice (size Com_Domain_X Com_Domain_Y no-size)))

;; end of physical domain definitions ===============
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;; Define Parameters for Geometry ============================
;; this section includes whether to use parabolic, spherical or 
;; elliptical lenses
;; also sets whether to use a metal or not

(define-param a 1.0) ; 1 unit in meep = 1 um, this is not used 
                     mathematically, but was worth noting *shrug*

(define-param D 3) ; D is the dish diameter
(define-param c 0.1) ; c is the dish depth
(define-param c2 1.5) ; an additional depth, used in offset 
                      troughs
(define Foc (/ (expt D 2) (* 16 c))) ; Define "Foc" the focal 
                                   length of the dish (f is in use)
(define Foc2 (/ (expt D 2) (* 16 c2))) ; the focus for the 
                                      second trough (if in use)

;; Define parameters for the dielectric material constants
;; to change between a lens and a dish, reverse the values of Si 
;; and Air.
(define-param Si 1.497) ;; 1.497 PMMA @ 500nm, 1.488 @ 700nm, 
                        ~1.555 @300nm (si ~12 at visible)
(define-param Air 1.0) ;; 1.435 - pdms at 500nm, 1.422 - pdms @ 
                        700nm
(define-param Glass 1.462) ;; A parameter for "Glass"
;; http://www.filmetrics.com/refractive-index-database/SiO2/
;; Fused-Silica-Silica-Silicon-Dioxide-Thermal-Oxide-ThermalOxide
(define R (+ (/ c 2) (/ (expt D 2) (* c 8)))) ;; Used for 
                                           calculating the RADIUS of a sphere from depth and diameter, 
                                           for spherical profile lenses.
(define-param shape 1) ;; used to choose the dish/lens shape. 
                         0=flat, 1=parabolic, 2=flat (with metal)
(define-param metaslits 0)
(define-param sourcedirection 0) ;; sets the direction of the 
                                  source. left to right is 0, right to left is 1.

;; This sets the resolution of the cell! very important for 
controlling CPU time and output file size!
(set-param! resolution 40) ;; base 10 for low res, or 25 for high 
                         res ; 35 for very high res (needed for 350 wavelength)
 ;; The runtime is defined by the wave frequency (Specified later) 
and this number!
(define-param runtime 120) ;; base 200
;; Captureevery is used to define how often during a timestep to
output data, can range from 0.1 to (runtime).
(define-param captureevery 0.3) ;; base 1.0 ; use 0.1 for
higher-resolution contour plot

(define-param xOS (- 0 12)); the x offset to move the dish/lens
in the x plane. currently sets the lens to the left of the
cell.

(define-param yOS 3.1); the y offset to move the dish up and
down, or add dishes above and below ; 6 is down, -6 is up,
etc. ; set to 0 for only 1 lens/dish

(define-param W 0.1); sets the distance between the two dishes
in SHAPE 2 (multiple dishes) where were looking at two dishes
different depth

(define-param mwdth 1); currently used to define the lenses FILM
THICKNESS ; previously used to define a thick metal film

;; Used for creating a thin metal film, with slits ; above line
was part of this, but has been reused

(define-param Mpos (/ sx 4)); metalslit position

(define-param thinfilm 0.1); used for a thin metal film

(define-param filmwidth 0.1)

(define-param slitspacing 2.0)

(define-param pitch (+ filmwidth slitspacing))

(define-param blocknumber (/ (* sy 1000) (* 2 pitch) ))

;; Used for single-sphere measurements to set the sphere radius
and position

(define-param sphere-diam 3.2)

(define-param position-x 0)

;; SOURCE PARAMETERS
;; This sets the source FREQUENCY (fcen) and FREQUENCY WIDTH of
the wavepacket fwth.

; the wavelength = 1/fcen ;; the +- on the wavelength is =
fwth/(fcen^2)

(define-param fcn 2.0); Centre frequency of source 2.0 =
500nm, 1.42857 = 700nm 3.3333 = 300nm (for a = 1um) ; 350nm =
2.857

(define-param fwth 0.2); width of frequencies to cover
(necessary for gaussian) Normally 0.2 for turn on/turn off

(define-param Source_Pml_Dist 1); distance from pml to source
Appendix


(define myAg (make dielectric (epsilon 1)
(polarizations
 (make polarizability
 (omega 1e-20) (gamma 0.0038715) (sigma 4.4625e+39))
(make polarizability
 (omega 0.065815) (gamma 0.31343) (sigma 7.9247))
(make polarizability
 (omega 0.36142) (gamma 0.036456) (sigma 0.50133))
(make polarizability
 (omega 0.66017) (gamma 0.0052426) (sigma 0.013329))
(make polarizability
 (omega 0.73259) (gamma 0.07388) (sigma 0.82655))
(make polarizability
 (omega 1.6365) (gamma 0.19511) (sigma 1.133))
)))

;;; Gold metal. for some reason never got it to work
(define myAu (make dielectric (epsilon 1) ;; from "Notes on Metals in meep" by Aaron Webster, 3rd nov 2011
(polarizations
 (make polarizability
 (omega 1e-20) (gamma 0.042747) (sigma 4.0314e+41))
(make polarizability
 (omega 0.33472) (gamma 0.19438) (sigma 11.363))
(make polarizability
 (omega 0.66944) (gamma 0.27826) (sigma 1.1836))
(make polarizability
 (omega 2.3947) (gamma 0.7017) (sigma 0.65677))
(make polarizability
 (omega 3.4714) (gamma 2.0115) (sigma 2.6455))
(make polarizability
 (omega 10.743) (gamma 1.7857) (sigma 2.0148))
)))

;;;;;;;

;;; GEOMETRY CREATION

;;;;
To create the parabolic geometry, sets up a variable \( f \) as a function of \( p \) to create the geometries shape. We call on \( f \) later when we build the geometry.

It creates a parabolic/spherical/elliptical dish in the \( y \) axis, with 2 flat "padding" regions at top and bottom.

For Parabolic: Basically says "if the \( \frac{(y^2)}{4 \times Foc} \) position is greater than the \( x \) position, make the value of \( f \) 1 instead of 12.

This conditional looks at the argument defined above "Shape" to determine what geom to define.

```
(cond
  ((= shape 0) ; FLAT INTERFACE
    (define (f p) (if (< ((vector3-x p) c) Air Si)) ;; AIR SI
      ;; Creates an air/silicon interface at \( x = c \)
      (set! default-material (make material-function (epsilon-func f)))
    )
    (set! geometry (list
      ;; standard PML clearing
      (make block (center 0 (+ (/ sy 2) (/ pml_y 2))) (size sx pml_y infinity) (material air)) ; top pml
      (make block (center 0 (- 0 (+ (/ sy 2) (/ pml_y 2))) (size sx pml_y infinity) (material air)) ; bottom pml
      (make block (center (- 0 (+ (/ sx 2) (/ pml_x 2))) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; right pml
      (make block (center (+ (/ sx 2) (/ pml_x 2)) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; left pml
    ))
  )))
```

This creates the parabolic shape within the function \( f \).
Appendix

((= shape 1) ;; SINGLE LENS/DISH, SINGLE APATURE
  ;; Define the parabola as a function f
  (define (f p) (if (> (vector3-y p) (/ D 2))
      (if (> 0 (- (vector3-x p) xOS)) Air Si) ;; The "+0" for the x part is so I can re-add an
      (if (< (vector3-y p) (- 0 (/ D 2)))
          (if (> 0
              (- (vector3-x p) xOS)) Air Si)
          (if (> (/ (expt (vector3-y p) 2) (* 4 Foc)) (- (+ (vector3-x p) c) xOS))
              Air Si)
          ))
  )

  (set! default-material (make material-function
    (epsilon-func f))) ;; use the function f to define the default
  ;; Further edit the geometry using standard
  blocks/spheres/etc.
  (set! geometry (list
      ;; Clear out PML layers, they must be
clear of material to work
      (make block (center 0 (+ (/ sy 2) (/ pml_y 2))) (size sx pml_y infinity) (material air)) ; top pml
      (make block (center 0 (- 0 (+ (/ sy 2) (/ pml_y 2)))) (size sx pml_y infinity) (material air)) ; bottom pml
      (make block (center (- 0 (+ (/ sx 2) (/ pml_x 2))) 0) (size pml_x Com_Domain_Y infinity) (material air)); right pml
      (make block (center (+ (/ sx 2) (/ pml_x 2)) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; left pml

      ;; RHS MATERIAL CLEARING, remove excess
  material to form a thin film.
      (make block (center (+ (/ sx 4) mwdth xOS) 0) (size (/ sx 2) sy) (material air)); clear out excess
  material to make right side clear
      (make block (center (+ (/ sx 4)) 0) (size
          (/ sx 2) sy) (material air))
      (make block (center (/ sx 4) 0) (size (/ sx 2) sy) (material air)); additional clearing)
(define (f p) (if (> (+ (vector3-y p) yOS) (/ D 2))
  (define (f p) (if (> (+ (vector3-y p) yOS) (/ D 2))
  ;; A modified version of the singlet dish/lens code, to create
  ;; three adjacent dishes, separated by flat areas, whose width is
  ;; defined by yOS
  (define (f p) (if (> (+ (vector3-y p) yOS) (/ D 2))
  ;; Define the geometry
  )
)
(set! default-material (make material-function (epsilon-func f)))

(set! geometry (list

;; PML clearing
(make block (center 0 (+ (/ sy 2) (/ pml_y 2))) (size sx pml_y infinity) (material air)) ; top pml
(make block (center 0 (- 0 (+ (/ sy 2) (/ pml_y 2)))) (size sx pml_y infinity) (material air)) ; bottom pml

(make block (center (- 0 (+ (/ sx 2) (/ pml_x 2))) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; right pml
(make block (center (+ (/ sx 2) (/ pml_x 2)) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; left pml

; Clear material just below the metal (c below ?) if using 2 refractive indices
(make block (center (+ xOS c layer) 0) (size sx sy) (material air))

;; Create a silver metal layer to the left of the three troughs.
(make block (center (- 0 (- xOS c)) 0) (size thinfilm sy) (material myAg)) ; metal block for mask
(make block (center (- 0 (- xOS c)) 0) (size (* 1 thinfilm) D) (material (make dielectric (epsilon Si))))

(make block (center (- 0 (- xOS c)) yOS) (size (* 1 thinfilm) D) (material (make dielectric (epsilon Si))))

(make block (center (- 0 (- xOS c)) (- 0 yOS)) (size (* 1 thinfilm) D) (material (make dielectric (epsilon Si))))

))

;; Create triplet of CONES to replicate a standard Axicon lens
((= shape 3)

;; background is defined as a flat interface
(define (f p) (if (> c (+ (vector3-x p) xOS)) Air Si))

(remove all dishes, flat glass)
(set! default-material (make material-function
  (epsilon-func f)))

(set! geometry (list
  ;; PML clearance
  (make block (center 0 (+ (/ sy 2) (/ pml_y 2))) (size sx pml_y infinity) (material air)) ; top pml
  (make block (center 0 (- 0 (+ (/ sy 2) (/ pml_y 2)))) (size sx pml_y infinity) (material air)) ; bottom pml
  (make block (center (- 0 (+ (/ sx 2) (/ pml_x 2))) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; right pml
  (make block (center (+ (/ sx 2) (/ pml_x 2)) 0) (size pml_x Com_Domain_Y infinity) (material air)) ; left pml

  ;; A metal layer is defined, and then three triangular holes are removed to form the axicon lenses
  (make block (center (- 0 (- xOS c)) 0) (size thinfilm sy) (material myAg)) ;; metal block for mask
  (make cone (center (- 0 (- xOS c)) 0) (radius D) (height (* 2 c)) (axis -1 0 0) (material air))
  (make cone (center (- 0 (- xOS c)) yOS) (radius D) (height (* 2 c)) (axis -1 0 0) (material air))
  (make cone (center (- 0 (- xOS c)) (- 0 yOS)) (radius D) (height (* 2 c)) (axis -1 0 0) (material air))
))

;; the code used to define OFFSET DISHES, dishes of two different depths.
((= shape 4)
  ;; Another specialised definition of f where two dishes are placed side by side, with depths c and c2
  (define (f p) (if (and (< (vector3-y p) (+ D (/ W 2))) (> (vector3-y p) (/ (+ D W) 2))))
    (> (vector3-y p) (/ (+ D W) 2))
    (if (> (/ (expt (- (vector3-y p) (/ (+ D W) 2)) 2) (* 4 Foc)) (- (+ (vector3-x p) xOS) (- c2 c))) Air Si)
      (if (and (< (vector3-y p) (- 0 (/ W 2))) (> (vector3-y p) (- 0 (+ D (/ W 2)))))
        (> (vector3-y p) (- 0 (+ D (/ W 2))))
        (if (> (/ (expt (+ (vector3-y p) (/ (+ D W) 2)) 2) (* 4 Foc2)) (+ (vector3-x p) xOS)) Air Si)
  ));; Another specialised definition of f where two dishes are placed side by side, with depths c and c2

(if (> c2 (+ (vector3-x p) xOS)) Air Si)
)

(setq default-material (make-material-function
(epsilon-func f)))

(setq geometry (list
;; PML clearing
(make-block (center 0 (+ (/ sy 2) (/ pml_y 2)))
(size sx pml_y infinity) (material air)) ; top pml
(make-block (center 0 (- 0 (+ (/ sy 2) (/ pml_y 2)))
(size sx pml_y infinity) (material air)) ; bottom pml
(make-block (center (- 0 (+ (/ sx 2) (/ pml_x 2))) 0)
(size pml_x Com_Domain_Y infinity) (material air)) ; right pml
(make-block (center (+ (/ sx 2) (/ pml_x 2)) 0)
(size pml_x Com_Domain_Y infinity) (material air)) ; left pml

;; Create a silver metal layer with apertures over the two troughs.
(make-block (center (- 0 (- xOS c2)) 0)
(size thinfilm sy) (material myAg)) ;; metal block for mask
(make-block (center (- 0 (- xOS c2)) (/ (+ D W) 2))
(size (* 1 thinfilm) D) (material air))
(make-block (center (- 0 (- xOS c2)) (- 0 (/ (+ D W) 2)))
(size (* 1 thinfilm) D) (material air))
))

;; Error checking to make sure shape was define correctly
(else (print "Error: no input shape\n") (exit))
)

;; END OF GEOMETRY ===============================

;; SOURCE CREATION ===============================
; creates the EM source

;; This conditional is used to set the SOURCE DIRECTION/POSITION.
; it either sets the source to begin on the LEFT (0) or the RIGHT (1) of the cell
(cond
((= sourcedirection 1) ;; Sets Source POSITION (and so
direction). the position is corrected to be Source_Pml_Dist
inside the physical domain, to not be inside the PML
(define-param Source_Center_X (- (/ sx 2)
Source_Pml_Dist))
(define-param Source_Center_Y 0)
) ; RIGHT TO LEFT
((= sourcedirection 0)
(define-param Source_Center_X (- 0 (- (/ sx 2)
Source_Pml_Dist)))
(define-param Source_Center_Y 0)
) ;; LEFT TO RIGHT
);

;; Define the source size. generally a 0 x size, and a y size of
the full computational cell width, minus PML
(define-param Source_Size_X 0)
(define-param Source_Size_Y (- sy (* 2 Source_Pml_Dist)))
;; Defines the component of light to us. Ususally used Z in 2D
(corresponding to out of the page on an image)
(define-param Source_Component Ez)
;; This final set actually creates the source from the defined
variables.
(set! sources (list
  (make source
    (src (make gaussian-src
      (frequency fcen) (fwidth fwth)))
      (component Source_Component)
      (center Source_Center_X Source_Center_Y)
      (size Source_Size_X Source_Size_Y)))
)
);

;; END OF SOURCES ============================

;; PML LAYER & RESOLUTION ====================
;; creates the pml border that should 100% absorb (but doesnt?)
(set! pml-layers (list
  (make pml (direction Y) (side High)
    (thickness pml_y) (pml-profile (lambda (u) (* u u)))))
  (make pml (direction Y) (side Low)
    (thickness pml_y) (pml-profile (lambda (u) (* u u)))))
  (make pml (direction X) (side High)
    (thickness pml_x) (pml-profile (lambda (u) (* u u))))
)}
(make pml (direction X) (side Low)
  (thickness pml_x) (pml-profile (lambda (u) (* u u))))
)
;; END OF PML & RESOLUTION ===============

;; PARAMETER OUTPUT ===============
;; Let us display the running parameters
;;(print "Computational domain center: " "[0,0], and size: [" Com_Domain_X "," Com_Domain_Y "]\n"
);
;;(print "PML layer[X,Y] size: [" pml_x "," pml_y "]\n"
);
;;(print "Physical domain center: " "[0,0], and size: [" sx "," sy "]\n"
);
;;(print "Source center: [" Source_Center_X "," Source_Center_Y "], and size: [" Source_Size_X "," Source_Size_Y "]\n"
);
;;(print "Detector T center: [" Detector_T_Center_X "," Detector_T_Center_Y "], and size: [" Detector_T_Size_X "," Detector_T_Size_Y "]\n"
);
;;(print "Detector R center: [" Detector_R_Center_X "," Detector_R_Center_Y "], and size: [" Detector_R_Size_X "," Detector_R_Size_Y "]\n"
)

; BEGINNING OF DATA OUTPUT===============

; Output of the data
;; Run until is the command that actually starts the computation running. It will "run until" a runtime has completed
(run-until runtime ;2007 timesteps at 514nm wavelength
;; These following lines set the output of the program
  (at-beginning output-epsilon) ;; outputs the dielectric geometry at simulation beginning
  (at-beginning output-mu) ;; outputs permittivity geometry at beginning, always all zero;
  (at-end (synchronized-magnetic output-poynting-x)) ;; code to output poynting X and Y;
  (at-end (synchronized-magnetic output-poynting-y)))
;
  (at-end (synchronized-magnetic output-dpwr)) ;outputs d-energy (wassat?)
  (at-end (synchronized-magnetic output-tot-pwr)))
; outputs total power at end of program
;; This is our main output line. This appends to file "energy" the total power at every (captureevery) timesteps.
Appendix

(to-appended "energy" (at-every captureevery
(synchronized-magnetic output-tot-pwr)))) ;; synchronisation
term is important when outputting E & B

;output-sfield-z

;;; END OF FILE ==============

A.3.2 matlab scripts

This first matlab script is used to extract the data from the .h5 file, compress it for
storage, as well as outputting the time integrated contourplot of the simulation.

% Clear all variables to begin the script. Just good practice.
clear all
% when running large numbers of models using a BASH script to run
 MEEP, we use COUNT.dat
% to determine where we are in the run process. count is used in
 several variables throughout the file
count = dlmread('count.dat');

% Variables to name what the model was (lens/dish) (refractive
test or pitch) etc.
type = 'lens';
run = 'para-fnum';

% Defines the most important MEEP input variables, the diameter
 and depth.
% These variables can be either 1 value, or an array if using
 count.dat for a large set of runs.
diam = 3
depth = 0.75
fnum = 1

% defines a number of the variables used in MEEP during these
 runs, used later when outputting.
spacing = 2.0 ;
wavelength = 500 ;
resolution = 40 ;
metal = 'none' ;
mthick = '0' ;
nref = 1.497 ;
\%nref2 = 1.0 ;
nfactor = 1; \% SET TO ! AS STANDARD (for different "output-every"
values in the MEEP file)
\%zlim = 3.0 ;

\%H5 file input \% Takes data from the .h5 files generates by meep
and creates a matrix
disp('Uploading H5 files now')
energy = h5read('lens-energy.h5','/energy');
eps = h5read('lens-eps-000000.0000.h5','/eps');
disp('Finished File Upload')

\% The first step is to compress the time-set of data into a
single xy dataset
\% This is done by simply summing over time. Effectively similar
to integrating over time.
disp('Calculations and Normalisation')
timesum = sum(energy);
[i,j,k]= size(timesum);
normalisem(1:j,1:k) = timesum(1,1:j,1:k); \% The data is
normalised to the maximum value
\% in the data set.
\% normalise to the maximum value.
nfactor = max(max(normalisem));
\% normalising
for g = 1:j
  for h = 1:k
    reverse(g,h) = normalisem(g,h)/nfactor;
  end
end

\% This creates two variables, used during outputting to give the
user an idea of the maximum value.
zmax = max(max(normalisem));
zlim = ceil(zmax); \% also used for creating a colorbar when
necessary

\%\% The following block is entirely for image output. imwrite is
the simplest method, but only creates
\%\% black and white images, hence getframe is sometimes used with
contourf.
disp('Setting up the Plot')
imwrite(reverse,sprintf('%s_%s_D%2.1f_c%1.2f_fnum%1.2f_wv%3.0f.png'
,type,run,diam(count),depth(count),fnnum(count),wavelength),'png')
%h = surf(reverse);
%set(h,'LineStyle','None')
%set(gca,'xlim',[0,j]);
%set(gca,'ylim',[0,k]);
%caxis([0,zlim]);
%view(0,90);
[C,h] = contourf(reverse)% creates the colour contourplot
%colorbar; % creates a colorbar to the side
%colormap(jet); % sets the colormap to "jet"
%print -dtiff -r1200 output % output method didn't work
%disp('Capturing plot to File contour.tif')
% captures a screenshot of the contourplot, then outputs to an
imagefile
%frame=getframe();
[x,map]=frame2im(frame);
%imwrite(x,'contour.tif','tif');

%%% Additional image writing for outputting a profile slice to
file. if used.
%slice = reverse(j/2,1:k);
%maxval = max(slice);
%plot(slice);
%set(gca,'xlim',[0,j]);
%set(gca,'ylim',[0,maxval]);
%frame=getframe();
[x,map]=frame2im(frame);
%imwrite(x,'profile.tif','tif');

% output the time-summed dataset to a file.
% The .h5 files are gigabytes in size, a timesum .csv is ~6mb
disp('outputting timesummed data to .csv file')
dlmwrite(sprintf('%s_%s_D%2.1f_fn%1.2f_wv%3.0f.csv',type,
run,diam(count),fnum(count),wavelength),normaliseme)
% output the MEEP input properties (defined earlier) to file
disp('Saving properties to file')
fid = fopen(sprintf('properties_D%1.2f_fn%1.2f.txt'
,diam(count),fnum(count)),'w+');

%%%%%
fprintf(fid,'Type: %s \n run-set: %s \n diameter:
%f \n depth: %f \n fnum: %f \n wavelength: %f \n resolution: %f \n refractive-index: %1.2f \n max-intensity: %d \n zscale-limit: %1.2f \n',
type,run,diam(count),depth(count),fnum(count),
wavelength,resolution,nref,nfactor,zmax);
fclose ( fid );
disp ( 'script is done!' )
display ( 'bye bye now!' )
exit

A.3.3 Bash scripts

This bash script was used to run one or more MEEP models in a single run.

#!/bin/bash
#
# A short bash script to run one or more MEEP models in a single command.
# The following unit can be copied multiple times to run more than one model.
# For each run you simply need to change the number output into count.dat, as well as the value for D and c throughout.

# outputs a number into count.dat used by makeimageserverbig to know the depth and diameter
echo '1' > count.dat
# begins MEEP running, D and c can be changed to any sensible value
# note that $1 is the name of the meep control file (without.ctl)
# which should be input to the command line when running this script.
meep D=3 c=3.0 $1.ctl
# These two lines create an image of the dielectric and permittivity geometry at the first timestep.
h5topng -S2 -t 0 -r -Zc dkbluered -a gray -A $1-energy.h5 -o D3c3.0-eps.png
h5topng -S2 -t 0 -r -Zc dkbluered -a gray -A $1-energy.h5 -o D3c3.0-mu.png
# This runs the matlab script makeimageserverbig to compress the .h5 files and create images
matlab -nodisplay -nosplash -nodesktop -r makeimageserverbig
# This is just tidying up, removing the several gigabyte .h5 files so a new one can take its place.
rm *.*
echo "done 1/24"
echo "Its Done!! HAHAA!!!
Bibliography


