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ABSTRACT

Metamaterials are artificially engineered materials for providing properties which are not readily available in nature. In the last decade, research activity in the field of metamaterials has led to diverse applications including remote sensing, lithography, communication, and biological imaging. For instance, researchers have shown that a class of metamaterials exhibit negative refraction and have also utilized this phenomenon to enable a super lens for beating the diffraction limit of light. Other fascinating developments include optical cloaking devices which involves bending of the electromagnetic waves completely around the objects. Therefore, metamaterials have become an important subject for study.

The central focus of this thesis is primarily on two applications of metamaterials: sub-wavelength imaging and laser beam manipulation. The proof-of-concept of sub-wavelength imaging has been demonstrated in the mid-infrared regime. A tapered array of step-index cylindrical waveguides is the basis for the magnifying infrared fiberscope. Optimized designs have been presented for the proposed infrared fiberscope by numerical modeling. The fabrication of the fiberscope is based on a high pressure chemical fluid deposition technique to deposit precisely defined periodic arrays of semiconductor waveguides within the holes of a microstructured optical fiber made of silica. The optical properties of various waveguides (germanium, silicon, zinc selenide, silicon nitride) fabricated by this method have been characterized in the infrared regime. The basic essential features of an imaging fiber bundle such as isolation between adjacent pixels, magnification, optical throughput and near-field image transfer characteristics
have been investigated. The imaging concept is demonstrated at 1.55 µm, 3.39 µm and 10.64 µm using appropriate materials for fabricating the tapered array of waveguides to maximize the optical throughput.

Manipulation of the laser beam has been demonstrated using patterned ferroelectric domains in lithium tantalate. The linear electro-optic effect in ferroelectrics was utilized to demonstrate the proof of concept of two dimensional dynamic focusing, optical switching and laser beam shaping. The beam propagation method was employed to design the required domain pattern. The domain pattern was fabricated by well established electric field assisted poling techniques. The performance of these devices is found to closely agree with theory.
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Chapter 1

Introduction

1.1 The fundamental diffraction limit

According to geometric optics, the image of a point source provided by an optical instrument is a perfectly sharp point. However, the image of a sharp point in focus is not a point, but a small fuzzy pattern of concentric rings, called the diffraction pattern. This led to the discovery of the fundamental ‘diffraction limit’ in optics by Abbe and Rayleigh. The best resolution achievable using a conventional lens in an optical system is limited by diffraction. The fine features of the object which are smaller than half the wavelength of light are permanently lost while imaging with a conventional lens. The underlying reason is that the information about the fine features in the object is contained in the evanescent component of the light emerging from it. These evanescent waves decay exponentially resulting in the loss of sub-wavelength (\(\lambda\)) features in the acquired image. In order to capture the evanescent components, the lens should be placed in the near-field within a distance of \(\sim\lambda/3\) from the specimen surface. Thus, the best theoretical resolution achievable in the far-field using conventional lenses is only \(\lambda/2n\), where ‘n’ is the refractive index of the imaging environment. Various methods which have been developed to overcome this limitation either by capturing the evanescent components in the near-field or by shrinking the wavelength of light by imaging in a medium with a higher refractive index. This phenomenon has also impeded the
progress of various fields ranging from nanolithography to endoscopy in biomedical imaging.
1.2 Overview of the contemporary imaging systems

Over the last decade, various methods have been proposed for sub-wavelength imaging. By exploiting plasmonic excitations at a sharp metallic tip, 10nm or less resolution can be achieved in the visible regime. There are other ideas to beat the diffraction limit of light, such as using a superlens, coupled-sphere waveguides, and resonantly excited surface plasmon-polaritons in arrays of metal wires\(^7\)\(^{-11}\). Metamaterials are also being actively pursued for sub-wavelength confinement of light\(^{12}\)\(^{-15}\). Near-field scanning optical microscopy (NSOM) is an established scanning probe technique that uses sub-wavelength apertures, including tapered optical fibers, to achieve 40nm resolution in the visible range. Researchers at the University of California, Berkeley have been successful in achieving sub-wavelength resolution up to 60 nm using a silver superlens as shown in Figure 1-1\(^{16}\). A vast majority of imaging by NSOM is in the visible range. However, using a SiC superlens layer with negative permittivity at \(\lambda\sim11\mu\text{m}\), in combination with an NSOM tip, Taubner et. al. demonstrated a resolution of \(\sim1\mu\text{m}\) at one specific wavelength\(^{17}\). NSOM can thus offer outstanding optical resolution. The major drawback with the NSOM is that the data collection is point-by-point, with long integration times. As a consequence, it is a serial and time intensive technique. The other commonly employed infrared imaging tools are predominantly based on solid immersion lenses (SIL), and coherent fiber-optic bundles\(^{18}\)\(^{-22}\). The techniques based on SIL and NSOM offer exceptional spatial resolution, but are extremely time intensive due to the serial scanning natures of these methods. They are also limited only to samples that can be brought to the microscope stage. Hence fiber-optic bundles are preferred for (a)
imaging specimens with restricted optical access in a minimally invasive manner, (b) characterizing the dynamic response and (c) time sensitive samples. A few specific applications include in-vivo cellular imaging\textsuperscript{23}, minimally invasive endoscopic medical imaging\textsuperscript{24}, and gas sensing inside chambers with limited optical access\textsuperscript{25}. The current technology for infrared fiber-optic imaging bundles is based on an array of either hollow/chalcogenide core waveguides. These imaging bundles have a modest pixel size of the order of 5 times the wavelength of light\textsuperscript{26}. The resolution of the imaging bundles seems to be predominantly limited by the inter-pixel cross talk. Therefore, there is a definite need for rapid imaging systems with sub-wavelength resolution.

![Chemical signature of various organic molecules in the infrared regime](image)

Figure 1-2: Chemical signature of various organic molecules in the infrared regime, Infrared fibers and their applications, SPIE Press, 2004, James Harrington
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http://www.electrophysicscorp.com
1.3 Challenges and opportunity in the mid-infrared regime

The mid-infrared ($\lambda \sim 3$-$8$µm) and long-infrared ($\lambda \sim 8$-$15$µm) wavelengths ranges are of tremendous importance for materials research as well as sensor technologies due to the vibration resonances that yield unique chemical signatures of materials, as can be gathered by a search on the relevant NIST database shown in Figure 1-2. Thermal imaging relies on infrared radiation since the black body radiation at room temperature peaks at $10.64$µm as shown in Figure 1-3. The wavelength range is also finding increasing importance in semiconductor inspection, sensors, night vision, automotive and airline industry, non-destructive testing and process monitoring, free-space communications, biology and health care such as in endoscopy and imaging. However, beyond the communication wavelength of $1.55$ µm, the overall research activity, as well as the choice and quality of the available optoelectronic components drop, with a concomitant increase in the cost. This applies to nearly every optical component, ranging from infrared sources, detectors, cameras, transmission media (fibers, waveguides), and even optics and coatings. For instance, commercial infrared detection range from cooled detectors and cameras based on semiconductor selenides, antimonides and tellurides such as HgCdTe, InSb, PbS, and others, to uncooled bolometers based on VO$_x$, amorphous silicon, and pyroelectric detection using ferroelectrics. Typical commercial infrared cameras today in the $\lambda = 3$-$5$µm have a resolution of $\sim 15$µm or more and this increases to $\sim 25$-$60$µm for cameras used for longer wavelengths. An infrared camera sold by Electrophysics© is illustrated in Figure 1-4 along with specifications.
Thus, working in the infrared regime clearly presents a unique challenge and an opportunity today.

1.4 Research objectives

The thesis work primarily focuses on two applications of metamaterials, namely: sub-wavelength broadband imaging and laser beam manipulation. Sub-wavelength imaging is demonstrated in the infrared regime (1-10 µm) by using tapered waveguides inside a silica based microstructured optical fiber. The optimum design for the imaging system (*fiberscope*) was developed by numerical simulations involving the beam propagation method and finite element modeling. In order to create the designed structure, a high pressure assisted chemical fluid deposition technique was employed to fabricate infrared waveguides (Ge, Si, and ZnSe) within the holes of a microstructured optical fiber. The processing conditions required for fabricating infrared waveguides with low propagation loss were determined by an iterative scheme. The proof-of-principle of imaging rudimentary objects in the near-field with sub-wavelength features were demonstrated at 1.55, 3.39 and 10.64 µm with built-in magnification and minimal cross-talk by using appropriate waveguide materials for maximum optical throughput.

Laser beam manipulation was demonstrated by using patterned ferroelectric domains in lithium tantalate. The linear electro-optic effect in ferroelectrics was exploited for the demonstration of two dimensional dynamic focusing, optical switching and laser beam shaping. The beam propagation method was employed to design the domain pattern required for the corresponding functionality. The numerically designed domain pattern
was fabricated by well established electric field assisted poling techniques. The performance of these devices was characterized at 633 nm and found to closely agree with theory.

1.5 Thesis organization

The thesis is comprised of 6 chapters. The potential of the high resolution infrared imaging is highlighted along with the fundamental limitations of the contemporary imaging systems in Chapter 1. A novel design for a magnifying fiberscope to achieve sub-wavelength resolutions in infrared imaging systems is proposed in Chapter 2. Numerical simulations for substantiating the essential features of the proposed design, such as inter-pixel isolation and image transfer characteristics are also presented in this chapter. In Chapter 3, the fabrication scheme employed to create the envisioned design is described. The high pressure assisted chemical fluid deposition technique for depositing various waveguide materials inside the pores of a microstructured optical fiber with high aspect ratio is discussed. The optical and material properties of the waveguides fabricated using this method is summarized in Chapter 4. An optimized geometry of an infra-red waveguide with low propagation loss is presented with potential applications in imaging. In Chapter 5, the details of fabrication, characterization, along with numerical simulations are presented for the magnifying infrared fiberscope using a tapered array of waveguides. The proof-of-concept of sub-wavelength imaging is demonstrated in this chapter. The future scope of the research work and outstanding issues are mentioned in Chapter 6.
Appendix A, B and C discuss the various ways of manipulating the characteristics of the laser beam using the electro-optic effect in ferroelectric lithium tantalate.
References


30. www.thorlabs.com

31. For an introductory review, see

    http://en.wikipedia.org/wiki/Thermographic_camera

32. For example, see FLIR cameras at www.flir.com/Thermography

Chapter 2
Design and numerical modeling

2.1 Conceptual schematic and essential features

The sub-wavelength imaging system demonstrated is based on a tapered microstructured optical fiber with an array of waveguides as illustrated in Figure 2-1. Each of these waveguides in the array operates as a pixel in the proposed imaging system - fiberscope. Potentially, thousands of such waveguides can be densely packed, and aid in the transport and confinement of photons from the object to the image plane. It can be visualized to be analogous to an array of NSOM tips. The primary advantage of the proposed fiberscope over contemporary imaging systems is the ability to capture an instantaneous snapshot of the specimen with a resolution well below the wavelength of light. This would facilitate the imaging of various time sensitive specimens such as biological cells and proteins which cannot be performed by serial imaging techniques. The basic features essential for demonstrating the proof-of-concept of the infrared fiberscope are (a) low loss infrared waveguides (b) isolation between adjacent pixels (waveguides) to minimize cross-talk and (c) magnification.

2.2 The building block: Step-index cylindrical waveguides

The invention of optical fibers was initially driven by the need for imaging fiber bundles to transmit images from a remote location¹. In a step-index waveguide, light is guided in the central region with a higher refractive called the core, surrounded by a
Figure 2: Schematic of the proposed infrared camera involving a combination of silicon/zinc selenide, germanium/zinc selenide: core (red) / cladding light grey) waveguides embedded in a silica (SiO$_2$) glass matrix. Absorption of the silica matrix, and (when required) additional coatings (not shown) around the waveguides, isolate the pixels.
cladding layer with a lower refractive index. The light is confined in the core by the principle of total internal reflection. The power transmitted through the waveguide is distributed into various guided modes allowed by the waveguide geometry. Each allowed mode has a unique transverse field distribution. The field distributions can be numerically derived by solving the wave equation in cylindrical co-ordinate system. The solutions for the guided mode profiles are represented by the modified forms of the Bessel function. The total number of allowed modes is a function of the core radius, wavelength of light and the difference in refractive index between the core and cladding. Typically, all other values remaining constant, the number of guided modes increase with increasing core diameter, decreasing wavelength and increasing index difference between the core and cladding. The lower order modes have lower propagation loss compared to the higher order modes. The fundamental HE_{11} mode has no cut-off in step-index waveguides and is of significance especially in imaging applications.

### 2.3 Material Selection for infrared waveguides

The first step is to design the geometry of a single cylindrical waveguide—the building block of the camera. The choice of materials for the core and cladding regions are based on the real and imaginary part of the refractive index. The imaginary part of the refractive index determines the propagation loss of the guided modes in these waveguides. Germanium (Ge) and zinc selenide (ZnSe) exhibit extremely low intrinsic absorption in the 4-13 μm wavelength regime and are good candidates for the waveguide as shown in Figure 2-2. The real part of the refractive index has a direct consequence on
Figure 2-2: The transmission spectra of SiO$_2$, Ge, Si, and ZnSe to be considered for the infrared fiberscope concept, Handbook of Optical constants of solids, Ed. Palik
the extent to which the guided mode in the waveguide can be confined. Hence, germanium is the most preferred material for the core as it has the maximum value of refractive index (~4) in the wavelength region considered. Silicon also has a large index (~3.5); however, it shows larger absorption in the 5-9µm range, but exhibits excellent transmission between 1.1-6µm. Si will also be considered for these wavelength regions.

To minimize the cross-talk between adjacent pixels, the cladding material has to be highly absorbing for providing the isolation required between adjacent waveguides. Thus, any light which is scattered outside the core gets absorbed in the cladding and does not leak into the adjacent waveguides. Silica has a relatively high coefficient of absorption in the 4-13 µm regime and is a good candidate for the cladding material.

To minimize the interaction of the guided mode with the silica claddings, an additional cladding layer of zinc selenide is introduced between the germanium core and silica cladding (Figure 2-3). Zinc selenide has a very low absorption coefficient and simulations predict that the optical transmission increases by more than an order of magnitude as shown Figure 2-4.
2.4 Numerical modeling of an imaging system with a resolution of $\lambda/3$

A fiberscope is defined as an imaging element with a resolution of $\sim\lambda/3-\lambda/4$ or higher for a typical wavelength of 10.6µm with a large array of pixels. As mentioned earlier, the basic features essential for the infrared fiberscope are:

(A) Low loss infrared waveguides as pixels
(B) Isolation between adjacent waveguides to minimize cross-talk
(C) Magnification of the image

Each of these design tasks is discussed next.

![Graphs showing losses of fiberscope designs](image)

Figure 2-4: Fiberscope pixel design in a silica matrix. (Left) A 1µm ZnSe cladding lowers the loss by up to an order of magnitude for small Ge core diameters. (Right) For an 8µm total waveguide diameter, an optimum ZnSe diameter for minimum loss is ~2.1µm and ~5.9µm Ge core.
A. Low-loss Ge/ZnSe Infrared Waveguides

Figure 2-3 shows a transverse cross-sectional schematic of a waveguide geometry that is envisioned. Numerical simulations using a combination of the beam propagation method (RSOFT©), and the finite element method (COMSOL Multiphysics©), have been used to confirm the feasibility of the proposed concept, to choose the combination of materials, and to determine the waveguide geometry for optimum performance. The representative simulations at a wavelength of 10.6\(\mu\)m were performed using BPM and FEM. The effect of the ZnSe buffer layer on the propagation loss was investigated. As illustrated in Figure 2-4 (a), as the Ge waveguide core gets smaller, the optical losses associated with the fundamental HE\(_{11}\) mode increase. However, the propagation loss is found to drastically decrease by up to an order of magnitude with the introduction of a zinc selenide (ZnSe) cladding layer. For a given total diameter of the hole in silica MOF, there is an optimum ratio of the thickness of the ZnSe layer to the thickness of the Ge (or Si) core. If the ZnSe cladding is too thin, the losses due to leakage into the SiO\(_2\) layer will be high. If the thickness is too large, the high index Si or Ge core diameter will be correspondingly smaller (for a fixed total diameter of the hole in silica fiber), and the mode localization decreases. For instance, for an 8\(\mu\)m capillary diameter in silica MOF, the minimum propagation loss of \(\sim 0.1\)dB/mm is predicted to be for a ZnSe cladding that is \(\sim 2.1\)\(\mu\)m thick, and \(\sim 5.9\)\(\mu\)m thick Ge core as shown in Figure 2-4 (b).

B. Optical Magnification through Ge/ZnSe Waveguides

The magnification is achieved by tapering the micro-structured optical fiber array. The taper angle and length determine the magnification of the fiberscope. Figure 2-5
Figure 2-5: A fiberscope with $R \approx \lambda/1.8$ resolution: BPM simulation of three tapered Ge(3µm)/ZnSe(0.5µm) core/cladding waveguides in SiO2 (pink) matrix, with a pitch (resolution) of 6µm, which is $\approx \lambda/1.8$ at $\lambda=10.6$µm. (A, B, C), show individual excitation of each waveguide by a Gaussian 3µm diameter beam at $\lambda=10.6$µm wavelength. (D) shows the excitation of all three waveguides, each with a Gaussian source with input intensity ratios of 1:0.73:0.64 (L to R). The output intensity ratios are 1:0.71:0.69 (L to R) in close agreement with input, as shown by the (blue line) intensity profiles (E). Magnification is 10X, for a 1mm long taper. Integrated propagation loss is <1dB as discussed in section 2.5.
shows a BPM simulation at a wavelength of 10.6\(\mu\)m, of three waveguides, each of Ge core (3\(\mu\)m) and ZnSe cladding (0.5\(\mu\)m) in a silica matrix. Note that the magnification of 10X over a 1mm long taper is easily achieved. The input intensity profile is also reproduced at the exit face, as shown in (E). The input was three Gaussian beams, each of 3\(\mu\)m diameters, and an intensity ratio of 1:0.73:0.64. The exit beams at the output closely seem to follow this intensity ratio (1:0.71:0.69). This simulation indicates that the silica matrix is effective for optical isolation at 10.6\(\mu\)m, when each waveguide is illuminated by its own source. This aspect of the isolation is discussed in detail in the next section. The image will be read out at the wider end of the taper by a conventional detector with a much lower resolution. Most of the conventional detectors in the 4-20\(\mu\)m wavelength range are based on thermal imaging and have a resolution of the order of 25-50\(\mu\)m\(^5\). Thus the conventional detectors can be hermetically sealed on to the backside of the proposed camera for capturing real-time images. Also note that the resolution is \(\sim\)6\(\mu\)m, as determined by the pitch length between two adjacent pixels. This demonstrates that sub-wavelength imaging is possible. We show in the following sections that a resolution down to \(\sim\)1.5\(\mu\)m is possible with low cross-talk.

**C. Optical Isolation between Pixels through Silica Matrix**

As demonstrated by Figure 2-5, one of the ways to achieve optical isolation between pixels is by the silica matrix that absorbs strongly in the infrared regime. The illumination condition in Figure 2-5, which was three Gaussian beams with diameters the same as the waveguides, exciting each waveguide individually. One way
Figure 2-6: Fiberscope with R-\( \lambda/2.7 \) resolution: A comparison of light propagation with and without a metal mask at the input. The BPM simulation of three tapered Ge(2\( \mu \)m)/ZnSe(0.5\( \mu \)m) core/cladding waveguides in SiO2 (pink) matrix, with a pitch (resolution) of 4\( \mu \)m. (a) and (b) show schematics without and with a gold metal mask, respectively. (c) When the center waveguide is excited by a Gaussian beam of 4\( \mu \)m diameter, the output excitation shows some intensity in the side waveguides as well. Over a 100\( \mu \)m taper length and a magnification of 10X, the cross-talk significantly decreases from \( \sim10\% \) (without a metal mask) to \( \sim1\% \) with a metal mask. (d) Imaging with a metal mask: Input Gaussian intensities in the ratio of 1:0.7:0.5 in the three pixels are reproduced at the output with ratio 1:0.67:0.49. Propagation loss is of the order of \( \sim1\)dB.
to achieve individual illumination is to use a metal mask at the input face, which exposes only the waveguides, and masks the silica around them from the incident light. This is important, because without the mask, the cross-talk would be significant particularly as the waveguides shrink down in scale, and are illuminated by a plane wave.

Figure 2-6 shows an example of three Ge/ZnSe waveguides with a Ge core diameter of 2µm each. The resolution as defined by the pitch is $R \sim 4\mu m$ which is $\sim \lambda/2.7$. The center waveguide is illuminated by a $\lambda=10.6\mu m$ Gaussian beam of 4µm diameter without a metal mask (2-7(c), red curve). The output light intensity profile shows a majority of light in the center waveguide. However, $\sim 10\%$ of the intensity also resides in each of the side waveguides, which represents a cross-talk. When a metal mask is introduced at the input, the cross-talk is reduced to $\sim 1\%$ (2-7(c), blue curve). With three different input intensities and a metal mask at the input, the output reproduces the input with minimal cross-talk.

These above simulations clearly indicate that a combination of the highly absorbing silica matrix, and a metal mask at the input would allow a pitch (resolution) down to $\sim \lambda/2.7$ at $\lambda=10.6\mu m$ with pixel sizes of $\sim 2\mu m$, magnification of $\sim 10X$, optical propagation losses of the order of $\sim 1dB$ and cross-talk limited to $\sim 1\%$ or less in the adjacent waveguides.

One of the important aspects of the fiberscope design is that the optical propagation loss can be significantly minimized as compared with the pure metal-based plasmonic waveguides. Broadly, we have two categories of waveguides in a SiO$_2$ matrix: the Ge/ZnSe wave-guides, and the Ge/ZnSe/metal waveguides. Despite the lossy silica
and metal media involved, we show below that for a ~200μm taper length, the integrated taper loss can be <1 dB for Ge/ZnSe, and ~20dB for Ge/ZnSe/metal waveguides.

Figure 2-7 a, b, c, respectively show a 2-D Finite Element Modeling (FEM) mode solution (using COMSOL™) for 10.6μm laser light of a single pixel comprised of Ge/SiO₂, Ge/Pt/SiO₂ and Ge/ZnSe/Pt/SiO₂ waveguides for a Ge core of 1.6μm. It is clear that the introduction of a Pt layer has spatially confined the mode strongly within the Ge. However, the simulation also indicates that if the waveguide was infinite in extent in the
third dimension (fiber length) with a uniform waveguide thickness, a loss of 400dB/mm is expected for Ge/SiO$_2$, which significantly increases to ~5500dB/mm for Ge/Pt/SiO$_2$. The introduction of a ZnSe interlayer between the Ge and Pt however reduces this loss back to ~1090 dB/mm, which is a reduction of nearly 5 times. Thus ZnSe interlayer can play a significant role in the reduction of waveguide losses by up to 5 times for the smallest of pixels possible using this concept. These losses can be dramatically reduced by another factor of 10 simply by the tapering out of the waveguide to larger sizes towards the output end of the camera.

Figure 2-7 (d) shows the influence of the tapering on the optical propagation losses as the light propagated through the waveguides. The losses drop exponentially as core diameter of the Ge waveguide increases. For a magnification of 20X and a taper length of 200µm, the integrated taper loss for the Ge/ZnSe/metal waveguide taper is ~19.2dB. Similarly, for a magnification of 10X and a taper length of 100µm, the integrated taper loss for the Ge/ZnSe taper (no metal) is <1dB. These optical losses are significantly superior to the losses in pure metal based sub-wavelength imaging concepts, with a corresponding tradeoff in the resolution.

2.6 An alternative approach using an array of photo-detectors

The electronic approach is very similar to the one described using an array of tapered waveguides. The only difference is that, in the electronic approach, doped semiconductor junctions (P-I-N) or metal-semiconductor schottky junctions can be fabricated within an array of sub-wavelength holes. The junction within each capillary
behaves like a photo-detector / pixel and transmits the image from the input to the output plane. One of the major hurdles in this approach has been overcome by developing the capability for fabricating doped semiconductor junctions inside the capillary by Dr. Rongrui He. The primary advantage of this approach is that the wavelength regime of operation is not limited to the transmission window of the waveguides, but can now be extended to the visible region where most of the semiconductors are favorably opaque. Additionally, the proof-of-concept of a high speed photo-detector in a single capillary hole has been demonstrated in the visible regime by Dr. Rongrui He. The details are discussed in Chapter 4. The same concept can be extended to an array of holes.

2.7 Discussion and conclusions

Below is a summary of the unique aspects of the infrared fiberscope designed by numerical simulations.

- **Sub-wavelength Broadband Imaging** down to $\lambda/4$ resolution (2-2.5$\mu$m pitch at $\lambda$~10$\mu$m): Each wire inside the fiber will act as a waveguide pixel. In contrast, the best commercial infrared cameras today have 15-60$\mu$m pitch, which is $\sim3\lambda$-20$\lambda$. The proposed camera can operate between 4-13$\mu$m, limited only by the choice of the proposed materials.

- **Magnification** of 15X: Tapering of the fiber naturally allows such magnification. The current commercial cameras will image the output on the wider end of the taper, but with sub-wavelength resolution of the image transferred from the narrower end of the taper.
• **Optical Isolation:** Cross-talk between the pixels is naturally limited in our designs by the silica matrix which is highly absorbing in the 4-13μm wavelength range. Additional isolation, when required, can be achieved by introducing a silicon carbide or metal layer.

• **Optical throughput:** Unlike high losses (10,000dB/mm or more) in plasmonic waveguides, our pixel design such as cladding/core (ZnSe/Ge) waveguide structures can lower loss by up to 5 times, with a resulting tradeoff in resolution. Further, the tapering out of the waveguide allows for another order of magnitude lowering of the integrated optical losses: For example, for ~200μm taper length, ~1dB integrated taper loss without metal cladding, and ~20 dB with metal cladding is possible at λ=10.6μm and a resolution of ~λ/4.

• **Number of Pixels:** Since silica fiber diameters are easily scalable from centimeters to nanometers, thousands of pixels per fiber are envisioned.
References


Chapter 3

High pressure chemical fluid deposition of waveguides within microstructured optical fibers

In most of the proposed ideas for sub-wavelength imaging in literature, the fabrication of the device is the most challenging step. In order to realize the structure proposed in Chapter 2, one has to be able to deposit germanium, silicon, and zinc selenide inside the capillary hole of a tapered silica fiber.

The idea is to start out with a silica micro-structured optical fiber (MOF) template as per the design specifications. The silica MOFs are fabricated at the Southampton Optoelectronics Research Centre (ORC) with dense arrays of capillaries with diameters down to nanoscale dimensions (10 to 100 nm) and lengths of meters to kilometers that can be engineered to have virtually any desired periodic or aperiodic spatial configuration. Silica MOFs are thus versatile nanotemplates, which unlike mesoporous silica or anodic alumina nanotemplates, have extreme aspect ratio, are highly scalable; possess engineered geometries combined with unparalleled optical transparency, tensile strength ten times greater than that of steel, and the capability to deform elastically by 5 to 10% under mechanical tension. Thus wire materials deposited within the templates can be organized themselves in virtually any desired 2-D spatial configuration. Prior to fabrication of waveguides, the fiber will be tapered using a standard fusion splicer. The taper angle defines the magnification of the camera and can be controlled by varying the
temperature (arc current) to which the fiber is locally heated and the force with which the fiber pulled. After tapering the MOF to design specifications, the waveguides will be fabricated inside each of the capillary holes by a high pressure assisted chemical fluid deposition technique as described next.

3.1 High pressure chemical fluid deposition technique

Chemical vapor deposition (CVD) is a widely adopted processing technique to deposit crystalline and amorphous materials with ultra high-purity\(^4\). The process is often used in the semiconductor industry to produce thin films. Microfabrication techniques widely use CVD to deposit materials in various forms, including: monocrystalline, polycrystalline, amorphous, and epitaxial. These materials include: silicon, carbon fiber, filaments, carbon nanotubes, SiO\(_2\), silicon-germanium, tungsten, silicon carbide, silicon nitride, silicon oxynitride, titanium nitride, and various high-k dielectrics\(^5-6\). The CVD process is also used to produce synthetic diamonds.

It is a process in which the precursor molecules in the vapor state undergo a chemical reaction to form the desired materials. The knowledge base of the CVD process has accrued over decades because of its wide spread use in industrial applications, especially electronics and optics. In early 1970’s, low loss optical fibers were fabricated by employing the CVD of fused silica. Additionally, the advent of fields such as nanotechnology and photovoltaics, the research activities related to CVD are gaining more prominence.

Variations of the chemical vapor deposition have been developed to deposit
various classes of materials. Some of the common, widely known methods include plasma enhanced CVD, hot wire CVD, laser assisted CVD, thermal CVD, and low pressure CVD\(^7\text{-}^9\). However, in some extreme cases it is not feasible to employ these methods. For instance, in the case of deposition within the pores of a microstructured optical fiber, the physical geometry may be a limitation due to the extreme aspect ratio of the substrate. Therefore, techniques such as plasma enhanced and hot-wire CVD cannot be employed for the deposition.

The extreme aspect ratio of the microstructured capillaries within the fibers presents a challenge for introducing materials. Typically CVD is carried out at low or reduced pressures\(^10\). Low pressures are good for typical CVD geometries however; it would not be possible to deposit within extreme aspect ratio capillaries over significant lengths at low pressures. High-pressures are thus necessary to achieve sufficient mass transport of precursor molecules into the capillaries. Inert gasses such as helium, argon, and carbon dioxide are used as high-pressure carrier gasses to transport precursor molecules through the microstructured capillaries.

High-pressure reservoirs are charged with precursors then pressurized with an inert gas to between 5 and 40 MPa. At these pressures the gasses employed are typically above their critical temperature and pressures making them supercritical fluids. The fibers are attached to a valve on the high-pressure reservoir using a high-pressure compression style fitting similar to the schematic illustrated in Figure 3-1. When the valve is opened the high-pressure fluid mixture is forced through the capillaries. The fiber itself is placed in a furnace and heated as the fluid flows through the capillary. The precursor molecules
thermally decompose depositing the desired materials onto the capillary walls creating waveguides within the MOFs.

The high-pressure chemical deposition is a relatively amateur field as most chemical deposition techniques are carried out at atmospheric or reduced pressures\textsuperscript{11}. However, there is a great advantage presented by the decade’s long CVD knowledge base for the introduction of a wide range of materials and structures. The chemistries of many materials and compositions have been thoroughly investigated in the low-pressure regime. This knowledge can be used as a starting point and aide in adaptation to high-pressure in-fiber deposition. There are various modifications to the high pressure assisted chemical fluid deposition technique for fabricating a wide range of materials. Some of the variables which can be tuned to obtain desirable properties are by controlling the flow rate of the precursors, pressure of the precursors and the carrier gas mixture flowing
<table>
<thead>
<tr>
<th>Material</th>
<th>Precursor (Carrier Gas, Pressure MPa)</th>
<th>Temperature (degrees C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ge</td>
<td>GeH₄(He, 35)</td>
<td>300</td>
</tr>
<tr>
<td>ZnSe</td>
<td>Zn(CH₃)₂₂+Se(CH₃)₂ +H₂ (H₂, 30)</td>
<td>350</td>
</tr>
<tr>
<td>Si</td>
<td>SiH₄(He, 35)</td>
<td>400-500</td>
</tr>
<tr>
<td>Pt</td>
<td>PtMe₂(cod), (CO₂, 10)</td>
<td>300</td>
</tr>
<tr>
<td>Ag</td>
<td>C₁₃H₁₃AgF₆O₂ (dissolved in supercritical CO₂, 10)</td>
<td>200</td>
</tr>
<tr>
<td>W</td>
<td>WF₆(He/H₂, 30)</td>
<td>350</td>
</tr>
<tr>
<td>SiC</td>
<td>(CH₃)₂CHSiH(CH₃)₂ (He, 35)</td>
<td>750-875</td>
</tr>
</tbody>
</table>

Table 3-1: Summary of the precursors and deposition temperatures for various materials fabricated via the high pressure chemical fluid deposition technique.
down the silica fibers, temperature of the deposition furnace, the rate at which the temperature is ramped to the set-point, choice of precursor and carrier gases, concentration of the precursors, and the reaction time interval. In some cases, more than one precursor is used for achieving doping in silicon, or for the fabrication of compound semiconductors such as ZnSe. Table 3-1 summarizes some of the conditions to be used for the deposition of different materials. The various advantages of this deposition technique are: highly conformal deposition resulting in smooth films, ability to fill nano-sized capillaries with materials, and sequential deposition can lead to the fabrication of multi-layer structures. These prominent features are illustrated in Figure 3-2. The details of the deposition conditions for a few of the materials used in the fabrication of the infrared fiberscope are discussed below.

3.2 Silicon

Chemical vapor deposition of silicon is typically carried out at low pressures however the extremely narrow and long pores of the MOFs would make low pressure deposition infeasible. Therefore, high pressures are utilized to achieve sufficient mass transport of the silane precursor molecules into the capillaries. In a typical deposition 5% partial pressure gas mixture of silane in helium with a total pressure of 34 MPa is forced through the 5.6 μm diameter capillary of a MOF. As the reactive gas mixture passed through the pore, the MOF is heated to 500 °C, resulting in the deposition of silicon onto the walls of the capillary. Deposition under such extreme conditions onto the walls of the capillary leads to highly conformal silicon layer growth continuing down to sub 10 nm
Figure 3-3: SEM images of fiber cross-sections displaying a capillary completely filled with silicon at 3cm (A), 4cm (B), 6cm (C), and 7cm (D) inside the deposition furnace (SEM captured by Dr. Neil Baril).

Figure 3-4: a-Si:H deposition profile within a 6.2 µm diameter capillary at 500°C from 5% silane:He gas mixture at 5000 psi total pressure, (a, b) SEM and (c) deposition profile experiments were done by Dr. Neil Baril.
Figure 3-4: Micro Raman Spectra of a silicon wire deposited within a 5.6 µm capillary of a MOF (top) and a Si reference sample (bottom). The lines through the data are Voigt fits.
pore size. In this manner it is possible to deposit solid silicon microwires several centimeters in length. The low deposition temperature leads to the formation of amorphous silicon wires within the capillaries of the MOF. After deposition the amorphous Si wire can be crystallized in a second step by annealing the fibers at 725 °C for one hour, followed by dwelling at 1325 °C for ten minutes. High temperatures are necessary to achieve large grain growth, however extended times at high temperatures weaken the silica of the optical fibers. The short ten minute anneal at 1325 °C leads to large grain growth without compromising the integrity of the silica. This is confirmed by characterizing the materials properties, discussed in Chapter 4. A scanning electron micrograph of silicon deposited within a 6 μm capillary is shown in Figure 3-3.

3.3 Amorphous hydrogenated silicon

Standard thermal chemical vapor deposition can enable the infiltration of higher aspect ratio templates. However plasma assisted chemical vapor deposition of silane under the standard conditions of low pressure and high temperature, results in the deposition of amorphous films with hydrogen content less than 1% with poor optical and electrical properties. It has been reported in literature that inclusion of hydrogen in amorphous silicon is found to greatly improve the optical and electrical properties. Therefore, to achieve hydrogenation of the silicon samples, the high-pressure chemical fluid deposition technique was slightly modified. In the previous section, the deposition of silicon occurs within open-ended capillaries, where a high-pressure carrier gas transports precursor molecules into the capillaries of MOFs for thermal decomposition.
Figure 3-4: Raman spectra collected with 633 nm excitation left, displaying Si-H stretch near 2000 cm$^{-1}$ (inset), and IR spectrum right
Under those conditions it is possible to deposit amorphous silicon, however by sealing the exit end of the capillary it is possible to deposit solid microwires of a-Si:H several centimeters in length. With one end sealed, the precursors are no longer flowing at high rates and the deposition chemistry is altered. The transport of precursor molecules into the capillary arises due to diffusion of helium and H$_2$ gas through the fused silica walls of the MOF, instead of flow driven by the pressure drop along an open ended capillary.

In larger volume reactors, pyrolysis of silane under similar high pressure conditions leads to the formation of micron sized particles, referred as ‘fines’ henceforth, are produced in the gas phase$^{14-16}$. Fines can contribute to surface roughness in deposited films as shown in Figure 3-4(b). To avoid fines low pressures and or low concentrations of silane are typically employed. We have found that high pressure deposition within capillaries that have a large surface area to volume ratio leads to highly conformal deposition. A representative cross section of a 5.6 µm ID fiber can be seen in Figure 3-4(a) displaying the smooth conformal deposition. The conformal deposition is further illustrated by the fact that solid wires only microns in diameter and several centimeters in length can be formed. Any deviation from conformal smooth deposition, such as the formation of fines, would quickly plug the capillary and halt the deposition beyond that point. We believe that it is the large surface area to volume ratio that enables the conformal deposition at high pressures, because the gas phase agglomerates do not have sufficient time to grow before sticking to the capillary walls. This is further evidenced by the fact that non-conformal deposition is observed in larger capillaries greater than 50 µm in diameter Figure 3-4(b).
Hydrogenation of the deposited material likely arises due to reduced deposition temperatures and an increased contribution of higher order silanes and agglomerates, produced by homogeneous gas phase reactions. Increasing silane pressure and reducing the deposition temperature has been shown to increase hydrogen concentration in amorphous silicon. In the case of deposition within a sealed capillary the solid wire of a-Si:H forms in a region where the temperature of the deposition furnace is as low as 300°C, compared to typical deposition temperatures greater than 500°C used in low pressure CVD from silane.

Helium carrier gas and hydrogen produced as a byproduct of silane decomposition must exit the capillary by diffusing through the silica walls to allow more precursors into the capillary. Even though the deposition is diffusion limited in this way, the Si deposits surprisingly fast. In fact the initial deposition rate is higher than that observed for flowing depositions in which the silane would be transported much more rapidly through the capillary. As the deposition proceeds, the film eventually pinches off the capillary and extends toward the attached silane reservoir forming a solid wire Figure 3-4(c). The material properties are summarized in Chapter 4. The processing conditions and the recipe for the deposition of silicon were developed by Dr. Neil Baril in the Department of Chemistry at Pennsylvania State University.

3.4 Zinc Selenide

The fabrication of ZnSe optical fibers faces even greater challenges in materials processing. The conventional fiber drawing technique has stringent requirements on
material properties and is so far restricted to glasses, polymers, and low melting point metals and semiconductors \(^{18-20}\). Microstructured optical fibers \(^{21, 22}\) with air holes running through their lengths provide unique templates to incorporate new materials and shape them into high aspect ratio, cylindrical fiber geometries.

The reaction using dimethylzinc (DMZn), dimethylselenide (DMSe) and hydrogen is particularly suitable for ZnSe deposition in the pores of MOFs under high pressures. The liquid DMZn and DMSe have appreciable vapor pressures at room temperature, and their molar ratio can be tuned to optimize the stoichiometry and
morphology in deposited materials. On the other hand, this reaction does not suffer the room temperature precursor pre-reaction issue that is common in the DMZn - H₂Se reaction typically used for conventional thin film deposition. Therefore, premature plugging in small capillaries is avoided. Experimentally, the deposition is carried out by pressurizing a mixture of DMZn, DMSe, and H₂ to a total pressure of 35–70 MPa and allowing the mixture to flow through MOFs heated by a furnace. The as deposited material is confirmed to be polycrystalline ZnSe in the cubic zinc-blende phase by x-ray diffraction. The radial conformal deposition on the capillary wall from outside in eventually leads to nearly completely filled cylindrical ZnSe wires with 2–50 µm in diameter, as shown in the cross section scanning electron image (Figure 3-5). The deposition can be quite uniform along the length as well, for example, a wire can have a uniform cross section for as long as 4 cm when fabricated inside a 10 cm long furnace. With the refractive index of ~2.4 for ZnSe and ~1.4 for silica, the obtained structures form unique ZnSe core/silica cladding optical fiber waveguides.

The high pressure deposition conditions have been controlled to achieve high surface quality in order to reduce surface scattering, one of the most significant loss mechanisms for a waveguide. The outer surfaces of these ZnSe wires could be nearly atomic smooth as the conformal coating duplicates the smoothness of the MOF capillary walls that have only 0.1 nm rms in roughness. The challenge for the in-fiber ZnSe waveguides is thereby to reduce the inner pore diameter and improve the smoothness of the inner surfaces. We have found that this can be achieved by increasing the precursor mixture total pressure, as the flow rate has significant influence on the deposition rate and material morphology. As the deposition progresses and the inner pore shrinks, the flow
Figure 3-5: Structural characterization. a) A XRD pattern collected from a 50 µm diameter ZnSe waveguide with the zinc blende structure. The small angle background is from both the silica template and the film used to mount the sample. b) Raman spectra of both the ZnSe optical fiber (solid line) and an optical grade ZnSe reference (dashed line)
rate decreases and the reaction becomes more diffusion limited, causing the growth rate to drop\textsuperscript{25}. With a slower growth rate, reaction intermediates have more time to move to the low energy sites on the deposition surface and larger grain growth is promoted as a consequence. These large grains grow towards the inner cavity and form rough inner surfaces when they meet. It is thereby desirable to have a high flow rate when the central hole is less than 1 µm to maintain an appreciable growth rate and allow small grains to develop. An effective way to do so is to increase the total pressure as we have found that the flow rate is proportional to the square of the pressure. Experimentally, inner holes with diameter as small as 300 nm have been obtained when a total pressure of 70 MPa is used. If the holes are small enough, their scattering effect could become insignificant to the overall optical transmission by noting that commercial silica single mode optical fibers can have an approximately 100 nm diameter “central dip” of refractive index that has only a small influence on the fiber performance\textsuperscript{26}. Our loss measurements support this argument and will be discussed later.

Electronic grade DMSe and DMSe were purchased from SAFC Hitech. The partial pressures of DMSe and DMZn are about 150-300 torr and their molar ratio is optimized at 2:1 (Se:Zn) to achieve the desired stoichiometry, morphology, and material quality. A typical deposition is carried out at 450 °C and 70 MPa for 24 hours. The processing conditions and the recipe for fabrication of ZnSe were developed by Justin Sparks in the Department of Chemistry at Pennsylvania State University.
3.5 Germanium

Much of the recent progress in semiconductor photonics has leveraged off the highly developed silicon-on-insulator (SOI) based technologies. Following the development of the silicon Raman laser\textsuperscript{27}, a number of important optoelectronic devices have been demonstrated including switches\textsuperscript{28}, modulators\textsuperscript{29} and an ultrafast optical oscilloscope\textsuperscript{30}. More recently, lasing from the direct-gap transition in a germanium-on-silicon waveguide has been demonstrated\textsuperscript{31}. Importantly, relative to silicon, germanium

Figure 3-6: (a) SEM of the 5.6 $\mu$m diameter core germanium fiber; scale bar 1 $\mu$m was captured by Justin Sparks. (b) Optical image of the polished 5.6 $\mu$m core fiber mounted inside thicker silica capillary; scale bar 20 $\mu$m. Inset shows the polished 30 $\mu$m core fiber; scale bar 30 $\mu$m
Figure 3-7: Raman spectrum of the as deposited germanium waveguides showing a broad peak which is a characteristic of amorphous samples, and the Ge-H vibrations as a result of hydrogenation in the inset.

has an extended mid-infrared transparency entailing a transmission window from 2 – 14\( \mu \)m, a higher nonlinearity and superior electronic properties making it an advantageous material choice for a wide range of active waveguide devices\(^{32,33}\).

The germanium fibers are fabricated using a microfluidic chemical deposition process in which a mixture of germane and helium (GeH\(_4\)/He) is forced to flow through the central hole of a fused silica capillary at high pressures \( \sim 35\text{MPa} \). The deposition of semiconductor materials at such high pressures is permitted owing to the robust
mechanical strength of fused silica capillaries. Germanium is deposited under no-flow conditions by sealing the exit end of the silica capillary. The deposition furnace is set at a relatively low temperature of ~300 C so that the material grows in an amorphous state. This is important as it allows for the interfacial layer of germanium to assume the pristine smoothness of the silica substrate (0.1 nm root mean square surface roughness) to minimize potential scattering losses at the core-cladding interface. An SEM and optical micrograph of the Germanium deposited inside a 6 µm silica capillary is shown in Figure 3-6. Furthermore, the low deposition temperature also suppresses the outward diffusion of hydrogen, which occurs through the silica cladding, so that a percentage will remain in the germanium core to neutralize the dangling bonds. Similar hydrogenation of amorphous silicon core fibers has resulted in a clear reduction of absorption losses as discussed in the previous section. The processing conditions and the recipe for the deposition of germanium were developed by Dr. Neil Baril in the Department of Chemistry at Pennsylvania State University.
3.6 Optical grade polishing

The quality of the input and output waveguide facets has a significant effect on the insertion loss of these waveguides fabricated within the silica capillaries. In order to obtain a smooth finish, the facets are polished using 50 nm sized colloidal silica slurry resulting in excellent surface quality. To start with, the fully filled regions of the fabricated waveguides are cleaved and inserted inside slightly longer silica tube with an outer diameter of ~ 6mm, and an inner diameter such that it closely fits the fabricated waveguide. This also enhances the handling and reduces the risk of damaging/breaking the sample during optical characterization. Epoxy or wax is used to bind the waveguide securely to the silica capillary. The constituents of the epoxy are mixed thoroughly in the ratio of 1:10 by weight and filled into the silica tube by capillary action. The epoxy is cured at 120°C for 30 minutes in an oven to ensure that the sample is securely held inside the bigger silica tube. The epoxy is found to shrink on curing, and hence it is advisable to use minimal amounts by using a silica tube, whose inner diameter closely matches that of the outer diameter of the sample. After curing the epoxy, the waveguide is inspected under an optical microscope to ensure that there are no thermally induced cracks present along its length. The region of interest in the silica tube with the waveguide embedded inside the bore is then diced using a diamond saw perpendicular to its length. The diced portion is then mounted on a homemade aluminum holder for coarse polishing with alumina. The recipe for coarse polishing is as follows: grinding for 3-5 minutes with 30 µm alumina to make sure that there are no major cracks on the surface of the waveguide. After grinding, it is inspected under the optical microscope to ensure that the pits which
are formed on the surface are uniform. The sample is then thoroughly washed under running tap water, and ground for 3 minutes with 15 µm, 9 µm and 5 µm sized alumina in sequence. After each step, it is inspected to make sure that the pit diameter reduces and is uniform. This completes the coarse polishing of the sample. After which, the sample along with the holder is sonicated to make sure that it is free of any alumina particles. The sample is then polished using the slurry containing 50nm colloidal silica with the Logitech automatic polishing machine as illustrated in Figure 3-7.
3.7 Discussion and Conclusions

The high pressure chemical fluid deposition technique has been employed to fabricate smooth waveguides inside the pores of silica based microstructured optical fiber. In addition to silicon, germanium and zinc selenide, other technologically important materials such as silicon nitride, platinum, tungsten, gold and doped semiconductors could be fabricated by employing different precursors. The deposition recipe was tuned to obtain the best material properties required for the waveguide applications. The material properties of the waveguides fabricated by this method are discussed in chapter 4. A similar recipe was used for deposition of an array of tapered waveguides required for sub-wavelength infrared imaging.
References

Chapter 4
Waveguide characterization

4.1 Contribution to loss in waveguides

Losses in optical waveguides originate from three different sources: scattering, absorption and radiation\(^1\). The contribution of each of these sources is dependent on the material quality and the waveguide design. Scattering can arise due to either volume or interfacial scattering. Volume scattering occurs due to the defects in the bulk of the waveguide such as voids, presence of impurity phases and grain boundaries. Interfacial scattering is primarily due to the roughness of the interface between the core and cladding. The contribution of volume scattering to optical loss depends on the number of defects and the size of the defect with respect to the wavelength. In bulk waveguides, the Rayleigh scattering is the dominant loss mechanism which exhibits \(\lambda^{-4}\) dependence. The interfacial scattering in waveguides fabricated by HPCFD within the pores of MOF templates are negligible due to the atomically smooth silica walls over which the deposition occurs. There are various mathematical models to quantify the loss due to the presence of an interface, and the values are negligible for an interface with rms roughness of the order of \(\sim 0.1\) nm. The higher order modes are more prone to interfacial scattering than the fundamental modes.
Absorption of photons in semiconductor waveguides originates primarily due to free-carriers and inter-band transitions. Inter-band absorption occurs when the energy of the incident photon is higher than the band gap. This excites electrons from the valence band to the conduction band. Therefore, wavelengths longer than the absorption edge of the material are employed in most applications of semiconductor waveguides. At wavelengths below the absorption edge, it can be utilized for photo-detector applications.

Free-carrier absorption is significant in semiconductor waveguides. The presence of electrons and holes are found to change both the real and imaginary parts of refractive indices of the waveguide material. The imaginary part of the refractive index is directly related to absorption in the waveguide and has been described by the Drude-Lorenz equation. The losses are found to dramatically increase with increased concentration of free-carriers (doping). However, it is not a significant source of absorption in un-doped semiconductor waveguides.

Radiation loss in straight waveguides is negligible. They are significant in bent waveguides due to the variation in the angle of incidence which may result is radiation. They may also be significant in tapered waveguides due to the conversion of the energy from one mode to the other if the new mode is leaky.

### 4.2 Coupling to optical waveguides

Coupling of light into waveguides for various applications is conceptually simple, however, in practice it is a non-trivial problem. The numerical aperture of the semiconductor waveguides is also very high due to the large magnitude of refractive
index compared to silica. It is a highly challenging problem especially when the cross-sectional dimension of the waveguides is of the order of a few micrometers. The commonly used techniques for coupling light into waveguides are butt coupling, end-fire coupling, prism coupling and grating coupling. Butt coupling and end-fire coupling are the simplest of these methods which involve shining of light onto the input facet of the

Figure 5-1: Schematic of the various ways of coupling light into the optical waveguides, image adapted from Silicon Photonics, G Reed and A Knights, 2004.
waveguide. In butt coupling, the two waveguides are placed very close to each other (butted) in order to facilitate the overlap of the mode profile of the transmitting waveguide onto the input face of the receiving waveguide. In the end-fire coupling technique, a lens is used to focus the input beam onto the input facet of the receiving waveguide. If the numerical aperture of the focusing lens is matched with that of the receiving waveguide, the end-fire coupling method can be used to excite all the allowed modes in the waveguide. The prism coupling and grating coupling methods are fundamentally different since the light is launched into the waveguides from the surface at a specific angle with a well defined propagation constant. This restricts the excitation of fewer modes within the waveguide. The schematic of the different coupling schemes is illustrated in Figure 4-1. For semiconductor waveguides, the prism coupling method is not widely applicable since typically the prism should have a higher refractive index than the waveguide itself, which is very high (3.5 in the case of silicon, and 4 for Germanium). Coupling using gratings is a powerful technique for coupling into specific modes, however it is not applicable for cylindrical waveguides, since fabrication of a grating is a planar process. This leaves us with the option of end-fire or butt coupling which are more fiber-friendly methods for coupling light into waveguides.

The coupling efficiency in these two methods is dependent on the extent of the field overlap between the excitation and the waveguide modes; the fraction of light reflected at the input facet of the waveguide; the surface quality of the input facet; the numerical aperture mismatch between the waveguide and excitation fields; and the spatial alignment of the excitation and waveguide modes. The overlap² of excitation and waveguide fields is evaluated by the overlap integral between the excitation field and all
the allowed modes in the waveguide. The overlap integral between any two fields $A$ and $B$ is given by

$$I = \frac{\int dy \int dx A(x)B(x)}{\sqrt{\int dy \int dx A^2(x) \int dy \int dx B^2(x)}}.$$ 

The denominator is the normalizing factor and the value of the integral varies between 0 and 1 where 1 represents 100% coupling efficiency. In most cases, the value of the overlap integral is approximately calculated by evaluating it between the fundamental mode and the excitation field instead of all the allowed modes in the waveguide.

The losses due to reflection from the input facet of the waveguide are determined by using the Fresnel equations. For normal incidence, it reduces to

$$\frac{n_1 - n_2}{n_1 + n_2},$$

where $n_1$ and $n_2$ are the refractive indices of the waveguide and the surrounding medium respectively. This is an approximate estimate of the losses due to reflection at each interface. The percentage of light reflected from the input facet of the waveguide increases with increasing refractive index. In order to minimize this, anti-reflection coatings can be used. However, for optical loss measurements, the reflection losses at each interface are accounted while making the calculations. The losses at the interface are also dependent on the quality of the facets. It needs to be polished to optical grade without any surface imperfections to reduce the loss at the interface. Therefore, the waveguide facets are polished using colloidal silica to achieve an rms surface roughness of ~ 50 nm as discussed in Chapter 3.
4.3 Measurement of propagation loss

The insertion loss is the total loss associated with the insertion of the waveguide into an optical system. This includes both the propagation loss and the coupling loss. The propagation loss is a direct indication of the material quality. The commonly used methods for the measurement of propagation loss are the cut-back method\textsuperscript{4}, Fabry-Perot resonance method\textsuperscript{5}, and the scattered light measurement technique\textsuperscript{6}. The Fabry-Perot loss measurement method is accurate only for ultra-low loss waveguides since it is dependent on the interference of multiple reflections within the waveguide which acts like a cavity. The scattered light from the surface of the waveguide can also be used to measure the propagation loss. The assumption is that the amount of light scattered is directly proportional to the power contained in the propagating light. This is tricky for fiber waveguides, especially since the MOF is enclosed in wax/epoxy in a larger capillary which acts as an absorbing layer for the scattered light. Hence, of the three methods, the cut-back method is the simplest one, and has been adopted in conjunction with end-fire coupling to make all measurements.

In the cut-back method, the insertion loss (-10*\log (output power/input power)) of the waveguide is recorded as a function of the length of the waveguide by cutting the length down by a small fraction after each measurement. The insertion loss (Y axis) is plotted as a function of the length (X axis) of the waveguide. The slope of this plot gives the propagation loss of the waveguide, and the intercept along the Y axis gives the coupling loss in the system. This method is a destructive method and the accuracy of the method increases as the number of measurements is increased. The inherent assumption
in this method is that the propagation loss is uniform throughout the length of the waveguide. In most of our samples, this assumption is not valid, since the deposition temperature varies along the length of the waveguide resulting in variation in material properties. Therefore, a variation of the cut-back method is used for all the measurement by carrying out the insertion loss measurement for a single waveguide length and accounting for coupling losses by calculating the Fresnel reflection coefficient, and the overlap integral assuming ideal spatial alignment. This is a non-destructive method and is best suited for waveguides which may not have uniform material properties along the length.
4.4 Experimental set-up

The end-fire coupling method is adopted for all the measurements of propagation loss at the telecommunications wavelength of 1.55 µm. The laser output from a continuous wave tunable diode laser (Agilent) is coupled into the waveguide by using a single mode lensed optical fiber (OZ optics). Lensed fibers with varying spot sizes and numerical apertures can be commercially purchased to increase the coupling efficiency. The two lensed fiber used have a numerical aperture of 0.15 and a spot size of wither ~5 µm or ~ 2.5 µm at the focal plane. The lensed fiber is mounted on a fiber chuck using magnetic holders to secure the fiber within the grooves. The spatial position of the lensed fiber is varied by using a piezo electrically controlled translation stage for optimizing the coupling efficiency. The polished semiconductor waveguide is encased in a silica capillary of~ 6 mm diameter and is mounted using a home-made holder on a mechanically controlled translation stage. The light exiting out of the waveguide is collected and focused on to an infrared camera (Hamamatsu) using a 60 X Newport objective lens with a numerical aperture of 0.85. The numerical apertures of the semiconductor waveguides are very high due to the high value of their refractive index. Therefore, it is advisable to use an objective with a high numerical aperture. The magnified image of the guided mode is also observed on a CCD monitor and the intensity profile is acquired using a frame grabber installed on a computer. A systematic approach is adopted while coupling the light into the waveguides. First, using while light illumination from an arc-lamp, the output facet of the waveguide is focused onto the infrared camera. After this step, the position of the collection objective and the sample are not altered. The lensed fiber is visually brought close to the input facet of the
waveguide and the intensity pattern of the guided mode is simultaneously monitored on the CCD camera. As the lensed fiber is brought very close to the input facet, the gain and sensitivity settings on the infrared camera are adjusted to obtain a good contrast. The piezo-electric controllers are then used to optimize the coupling efficiency by looking at the intensity pattern on the camera. After visual optimization, a flip mirror is introduced in the path of the collimated output beam and is focused onto a power meter. The coupling efficiency is further optimized by fine-tuning the position of the lensed fiber using the piezo-electric controllers. A photograph of the lensed fiber, sample and the collecting objective are shown in Figure 4-2. After optimization, the output power and the intensity profile of the guided mode are recorded. For estimating the input power, the sample is removed, and the light from the lensed fiber is collimated using the same 60 X objective on to a power meter. By doing this, we account for the losses due to the reflectivity of the flip mirror and transmission of the objective. The reflection losses at each facet are calculated by using the Fresnel equations under normal incidence and accounted for in the measurements. The coupling efficiency (overlap integral) is assumed to be 100% to get the upper estimate of the propagation loss using simple calculations. The optical properties of various materials such as silicon, germanium, zinc Selenide, doped semiconductor junctions and silicon nitride have been characterized using this approach.
4.5 Silicon

The push for higher bandwidth in optical communications is a driving force for advancement in silicon photonics. Recently, silicon has been proving its worth as an optical material for various passive and active optoelectronic devices such as waveguides, high speed modulators, lasers, amplifiers, and photo-detectors. Because of the vast body of research in silicon on chip fabrication, much of the research in silicon photonics to date has been carried out on chip-based platforms. However the introduction of silicon into the pores of a microstructured optical fiber (MOF) would enable the seamless integration of numerous applications with light transmission. Optical modulation of guided light and in-fiber field effect transistors have already been demonstrated in silicon filled MOFs. However, realization of low optical transmission loss is crucial to the further development of in-fiber optoelectronic devices. Optical transmission loss is a major hurdle for polycrystalline silicon photonics. One of the primary loss mechanisms in polycrystalline silicon waveguides is scattering due to surface roughness. The attenuation of the guided light over long distances is appreciably low in MOFs due to the extremely smooth interface at the core/cladding. The capillary walls have a surface roughness of less than 1 Å rms. This could be great advantage if the material properties of silicon deposited within the silica capillary could be improved. The best amorphous silicon deposited by HPCFD is found to have a propagation loss of around 10dB/cm. In order to improve the material quality, the amorphous silicon waveguides are annealed at 1300 C for 10 minutes to induce crystallization. The silica capillary becomes soft at this elevated temperature, and hence the annealing cannot be performed over a long time.
overall morphology is well maintained after annealing, as shown in an SEM in Figure 4-3. It also showcases the ultra-smooth nature of the waveguide walls, which is an inherent advantage in HPCFD. The crystalline nature of the wire is investigated by Raman spectroscopy, which is a fast and nondestructive technique, sensitive to the crystalline properties of silicon. The T$_{2g}$ Raman mode of crystalline silicon at 521 cm$^{-1}$ is sensitive to the crystalline fraction and grain size of the material. Asymmetry in the Raman peak would indicate the presence of nano-crystalline and amorphous phases, and broadening of the T$_{2g}$ peak indicates a distribution of smaller and larger crystallites. Raman spectra were collected with a Dilor XY Raman spectrometer.
spectrometer using 633nm excitation, a 1200gr/mm grating, and a 600mm dispersion path length. The laser power was set to 46 µW to ensure that there was no broadening or shifting of the Raman mode due to heating. The crystalline peak was fit with a Voigt profile to separate the Lorentzian Raman component from the Gaussian instrumental component, giving FWHM values of 2.83 cm\(^{-1}\) and 2.97 cm\(^{-1}\) for the silicon wafer and the deposited wire respectively (Figure 4-3). Our Si wires match the Raman shift of the single crystal Si reference, at 521 cm\(^{-1}\), and demonstrate broadening as small as 0.14 cm\(^{-1}\) with no asymmetry. This result indicates that the deposited material is high quality polycrystalline silicon with large grains and no apparent amorphous fraction.

Figure 4-4: (A) Infrared image of 1.55 µm light being guided through a silicon wire within a MOF. (B) An optical image of light being reflected from the surface of the same silicon filled MOF. The bright spot at the center is the 5.6 µm silicon core that is more reflective than the surrounding silica
Optical transmission loss measurements were carried out on the Si wires using the cutback method. In order to reduce the extrinsic optical loss at the input and output face of the fiber, the ends of the fiber were chemo-mechanically polished to optical grade. An optical micrograph of the polished silicon core is shown in Figure 4-4. The polished Si core in the center reflects the light the most intensely. A 40x objective was used to couple laser light at 1.55 µm light from a continuous wave laser source into the 5.6 µm diameter Si core. The 1.55µm light exiting the waveguide was collected and collimated onto a camera/power head using a 60x objective. The magnified image of this light exiting the fiber from the Si core recorded on the camera is displayed below in Figure 4-4. The high brightness of the Si core on the infrared image indicates its excellent guiding performance at 1.55 µm. The loss is further quantitatively determined by the cutback measurements. The loss was calculated to be 7dB/cm using the cutback method. This is on par with the best polycrystalline waveguides reported in literature.

Optical losses in polycrystalline silicon waveguides arise primarily from surface losses and scattering at grain boundaries. The extremely smooth inner surface of the capillary walls of the MOFs, which have a rms roughness of less than 1 angstrom, aids in eliminating losses due to surface scattering. High temperature annealing produces large micron diameter crystalline grains, reducing the number of grain boundaries that the light must cross in propagating through the fiber. We believe that it will be possible to further reduce the losses within the MOFs due to their extreme aspect ratio geometry.

One of the strategies adopted to improve the optical transmission of silicon waveguides is by tuning the annealing parameters. In order to understand the effect of annealing parameters, two silicon waveguides with a 6 µm core diameter were annealed
at 530 C and 560 C respectively. After measuring the propagation loss of these samples, both these samples were annealed at 1300 C for 15 minutes and propagation loss was re-measured. The propagation loss values are illustrated in Figure 4-5. The sample annealed at 530 C is found to have a higher propagation loss in compared to the sample annealed at 560 C. However, after annealing at 1300C, the trend is reversed as the sample which was initially annealed at 530C has a lower propagation loss of ~ 9dB/cm. The underlying reason for this observation is that the sample annealed at 530C has larger grains, hence more defects compared to the one annealed at 560C. This higher concentration of defects

Figure 4-5: The effect of annealing parameters on the propagation loss of silicon. Data plotted by Justin Sparks.
results in a higher loss. However, after annealing at 1300C, the defects are annealed, and the main contribution to loss is from the grain boundaries. Since the sample initially annealed at 530C has larger grains, hence lower grain boundary scattering, it has a lower loss. This investigation reveals how the propagation loss is highly sensitive to the annealing parameters and calls for detailed investigation. The other strategy that has been adopted to lower the propagation loss is by incorporation of hydrogen to satisfy the dangling bonds in amorphous silicon. The details will be discussed in the next section.

4.6 Amorphous hydrogenated Silicon

Amorphous hydrogenated silicon (a-Si:H) has found extensive application in large area thin film transistors for liquid crystal displays and solar cells. Low temperature and low cost production are the primary motivation for pursuing a-Si:H technology. Improved optical properties due to hydrogen passivation of dangling bonds also make it a useful material for optoelectronic integrated circuits and waveguide devices such as modulators, switches, and light emitting devices. High quality films and structures of a-Si:H are generally produced via PECVD, or hot wire CVD. However, because these techniques are based on remote generation of reactive radicals for deposition they are not suitable for deep high aspect ratio structures. Template organization of materials is a powerful tool for developing new and exciting functionalities and interactions. Infiltration of a-Si:H into high aspect ratio structures would enable the production of waveguides capable of long, intense light matter interactions. Such interactions could be exploited for the production of new in-fiber
photonic and optoelectronic devices that would, for example, allow manipulation of light in transmission.

Figure 4-6: Raman spectra collected with 633 nm excitation left, displaying Si-H stretch near 2000 cm\(^{-1}\) (inset), and IR spectrum right. Raman spectrum acquired by Dr. Neil Baril

The deposition recipe adopted for fabricated hydrogenated amorphous silicon waveguides has been discussed in Chapter 3. In order to characterize the material properties, both Raman and infrared spectroscopy are sensitive techniques for gaining information about the microstructural and chemical nature of the wires. The Raman and IR spectra illustrated in Figure 4-6 indicate the presence of both S-H and SiH\(_2\) bonded hydrogen. The Raman active Si-H peaks are located between 600-900 cm\(^{-1}\) and near 2000 cm\(^{-1}\) \(^{19}\). The peaks in the 600-900 cm\(^{-1}\) region are convoluted with 2nd order Si peak. However, the peak at 2000 cm\(^{-1}\) confirms the presence of hydrogen. The width and position of the TO peak near 480 cm\(^{-1}\) suggests high quality silicon.
Deposition temp: 500 deg C, Sample 1

At ~ 2 cm inside the furnace

Sample Length: 21 mm

10.6 dB/cm

Length: 9.9 mm

6.2 dB/cm

Length: 10.1 mm

19.1 dB/cm

Direction of flow

at 500 C

Deposition Temp: 500 deg C, sample 2

Sample Length: 21 mm

11.5 dB/cm

Length: 9.9 mm

7.5 dB/cm

Length: 10.1 mm

21.2 dB/cm

Direction of flow

at 500 C
Deposition temperature: 450 C

7.3 dB/cm

Sample: Length: 22.8 mm

3.5 dB/cm

Length: 11.2 mm

11.8 dB/cm

Length: 10.8 mm

Direction of flow

d at 450 C

Deposition temperature: 400 C

3.5 dB/cm

Sample designation: 1A

Sample length: 25.4 mm

3.1 dB/cm

Length: 12.3 mm

Sample designation: 2A

5.8 dB/cm

Length: 12.3 mm

Sample designation: 2B

Direction of flow

d at 400 C
In order to determine the deposition conditions for obtaining the material with best optical transmission, an iterative scheme was followed. Decreasing the deposition temperature has been reported to increase the hydrogen concentration. This should thereby have a direct consequence on improving the optical transmission. A systematic study of the optical propagation loss, as a function of temperature was performed. The summary of the measurements at 500C, 450 C and 400C are illustrated in Figures 4-7, 4-8, 4-9, 4-10 respectively. The optical transmission properties of the waveguides were observed to be non-uniform along the length of waveguide. This could be partially attributed to the temperature profile along the furnace used for deposition. The propagation loss is the lowest at the end closest to the reservoir, and increases steadily at regions farther away from the reservoir. Another important observation is that with decreasing deposition temperature of the furnace, the propagation loss tends to decrease. This could be attributed to the dependence on the diffusion rate of hydrogen on temperature. At higher temperatures, the diffusion rates are higher resulting in the loss of hydrogen content in the waveguide. The lowest value for propagation loss was found to be as low as ~ 3dB/cm in the samples deposited at 400C. The optical transmission of the hydrogenated waveguides is approximately 20 times higher than amorphous silicon deposited under the same conditions with gas flow. Due to the low loss in these waveguides, the Fabry-Perot effect is observed when the propagation loss is measured as a function of the power in the waveguide. The propagation loss seems to oscillate due to the resonant cavity formed between the input and output facets of the silicon waveguide, which act like partially reflecting mirrors due to their high refractive index. One such
representative plot is shown in Figure 4-11. The material properties could be improved further by fine tuning the deposition parameters.

4.7 Zinc Selenide

Low loss optical waveguides and high quality laser sources in the mid-infrared region are of great interest for thermal imaging, chemical sensing, and medical surgeries\textsuperscript{20}. Among a number of IR transparent fluorides and chalcogenides, crystalline ZnSe has emerged as an excellent material candidate because of its transparency over a
wide range from 1−20 µm$^{21}$ and its supreme performance as a tunable IR laser (1.9−3.1 µm) when doped with Cr. Indeed, very low loss, high efficiency lasers have been demonstrated in Cr$^{2+}$: ZnSe bulk crystals$^{22}$. However, it is essential to develop high quality, compact ZnSe waveguides for advanced performance and applications such as precision delivery of high IR power in surgery. Effort has been made on fabricating microscale planar waveguides of many materials including ZnSe, but the success has been limited, primarily due to the roughness introduced during microfabrication$^{23}$. Free standing optical fiber waveguides are advantageous over wafer based planar waveguides, as they are flexible, transmit light over long distances, and act as symmetric, long cavities for laser generation with high output power and high optical quality$^{24}$. The deposition recipe developed by Justin Sparks, described in Chapter 3 is followed to fabricate ultra-smooth crystalline, cylindrical ZnSe waveguides within the pores of the silica capillary.
Besides the surface morphology and geometry, the material quality also has to be optimized to obtain low material loss for a waveguide. Raman spectroscopy reveals sharp peaks at 204.9 cm\(^{-1}\) and 251.2 cm\(^{-1}\), associated with the transverse optical (TO) and longitudinal optical (LO) optical phonons as shown in Figure 4-12. Their Lorentzian peak widths, 4.7 cm\(^{-1}\) (TO) and 6.1 cm\(^{-1}\) (LO), are comparable to the values of 4.1 cm\(^{-1}\) and 5.9 cm\(^{-1}\) obtained from an optical grade ZnSe reference (Alfa Aesar), indicating that the ZnSe is highly crystalline. This is further demonstrated by the symmetric shape of the LO peak, as asymmetry can be caused by a high concentration of crystalline defects\(^{25}\). The crystalline nature of the deposited material is verified by analyzing the X-ray diffraction pattern.

<table>
<thead>
<tr>
<th>Wavelength ((\mu m))</th>
<th>Core diameter ((\mu m))</th>
<th>Propagation Loss (dB/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.55</td>
<td>5</td>
<td>17</td>
</tr>
<tr>
<td>1.55</td>
<td>11</td>
<td>1</td>
</tr>
<tr>
<td>1.55</td>
<td>50</td>
<td>&lt;1</td>
</tr>
<tr>
<td>3.39</td>
<td>50</td>
<td>6.5</td>
</tr>
<tr>
<td>10.64</td>
<td>50</td>
<td>16</td>
</tr>
</tbody>
</table>

ZnSe
Figure 4-13: Optical propagation loss of ZnSe waveguides with two different core diameters as a function of the input power.

<table>
<thead>
<tr>
<th>Output Face</th>
<th>Mode 1</th>
<th>Mode 2</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="5 um core" /></td>
<td><img src="image2.png" alt="Mode 1" /></td>
<td></td>
</tr>
<tr>
<td><img src="image3.png" alt="11 um core" /></td>
<td><img src="image4.png" alt="Mode 2" /></td>
<td></td>
</tr>
</tbody>
</table>

Figure 4-14: Intensity profile of the different guided modes recorded on the infrared camera at 1.55 µm wavelength.
The optical transmission characteristics of ZnSe optical fiber waveguides are summarized in Table 4-1. The propagation loss is found to increase with decreasing core diameter due to the interaction of the guided mode with the cladding. The effect of the miniscule center hole is also dominant at smaller core diameters. The propagation loss seems to be constant as a function of the input power as shown in Figure 4-13. The transverse intensity profile of the guided modes recorded on the infrared camera is illustrated in Figure 4-14. The lowest propagation loss is observed to be less than 1dB at the telecommunications wavelength of 1.55 µm. This represents a significant advance in low loss infrared waveguides. The sub-1 dB·cm⁻¹ loss is to our knowledge the lowest value reported for microscale ZnSe waveguides in the mid-IR region and could immediately enable various practical applications. The unique fiber geometry of these waveguides makes them even more advantageous in power delivery and laser generation. Moreover, crystalline ZnSe holds promises beyond a simple dielectric optical waveguide. It is a direct band gap semiconductor and has pronounced nonlinear optical properties, which may be harvested to realize unprecedented optoelectronic functions in fibers such as electrically driven light emission.

4.8 Germanium

Optical loss measurements of the germanium fibers are presented in the mid-infrared regime for two different core diameters. Micro-Raman measurements have been used to establish the nature of the amorphous core which has been found to have a small percentage of hydrogen. Significantly, the incorporation of hydrogen into amorphous
materials is known to saturate the dangling bonds so that losses due to absorption are reduced\textsuperscript{26}. The extended mid-infrared transmission range measured in the germanium waveguides demonstrates their potential for use in areas ranging from broadband data transmission to biology and medicine. We investigate two germanium fibers with core diameters of 50 µm and 30 µm. There was no light guiding observed in germanium waveguides with a core diameter of 5.6 µm. To facilitate efficient free-space coupling of light into and out of the germanium fibers we first mount them inside thicker silica capillary tubes and then use a standard polishing technique to finish the end faces. Figure 3-16 shows an SEM and optical microscope image of the polished facet illustrating
complete filling of the capillary. The quality of the core materials was determined using micro-Raman spectroscopy conducted on a Renishaw inVia system with a 633 nm HeNe laser source. A 100X objective was used to focus 250 µW of power in a 0.4 µm spot directly onto the polished germanium cores and a typical spectrum of the backscattered radiation, as recorded on an air-cooled “RenCam” CCD detector, is shown in Figure 4-15.

The Raman spectrum shows a broad peak, indicative of an amorphous material, around 280 cm\(^{-1}\), which is slightly shifted from the transverse optical Raman resonance of 278 cm\(^{-1}\) for amorphous germanium\(^2\). However, this result is not unexpected as silica exhibits a different thermal expansion to germanium so that cooling from the elevated deposition temperatures will induce thermal mismatch stresses that can account for the slight shift in the resonant peak. Evidence of hydrogenation is provided by the peak at ~1900 cm\(^{-1}\), as shown in the inset of Figure 4-15, which is associated with the Ge-H stretching mode. Although this stretching mode is typically split with a second vibration appearing at ~2000 cm\(^{-1}\), our observation of only a single peak suggests that the hydrogen content is low. The optical transmission properties of the germanium fibers were measured using a continuous wave carbon dioxide laser at 10.64 µm and a Helium-Neon laser at 3.39 µm. In both cases, the single pass measurement technique was performed on fibers of length ~5mm to determine the average loss value at a given wavelength. This was deemed to be the most appropriate technique owing to there being
a variation in the hydrogen content over the fiber lengths which led to significant deviations in the losses calculated via the cutback method. Thus it is clear that the process of hydrogen incorporation via this method requires further investigation if uniform low loss fibers are to be obtained.

The laser sources used at these wavelengths were both continuous wave (CW), where at 3.39 µm a HeNe laser was used with an input power of ~12mW and at 10.6 µm a CO2 laser was used with an input of ~30mW. In both cases the light was launched into the fiber’s core with a 25.4mm focal length ZnSe lens and the output was focused onto a Laser Probe Inc. RK-5720 thermal power meter, with a RKP-575 detector head, using a 52X Ealing silver coated reflecting objective. Coupling into the core was facilitated by using a pyroelectric Electrophysics PV 320 camera to image the guided output. The losses measured at these two wavelengths are summarized in Table 4-3.

<table>
<thead>
<tr>
<th>Wavelength (µm)</th>
<th>Core diameter (µm)</th>
<th>Propagation Loss (dB/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.39</td>
<td>30</td>
<td>1.73</td>
</tr>
<tr>
<td>3.39</td>
<td>50</td>
<td>0.51</td>
</tr>
<tr>
<td>10.64</td>
<td>30</td>
<td>0.48</td>
</tr>
<tr>
<td>10.64</td>
<td>50</td>
<td>0.53</td>
</tr>
</tbody>
</table>
This observed wavelength dependent trend is consistent with our previous measurements for the losses in hydrogenated amorphous silicon fibers which we primarily attributed to scattering effects. By optimizing the deposition procedure to improve the germanium material quality and better understand the process of hydrogen incorporation, we anticipate that the losses in these fibers could be reduced over this entire wavelength range, as similarly been achieved within the silicon core fibers. The low losses measured at the longer wavelengths indicate that efficient device performance should be possible in this wavelength regime, opening up the potential for germanium fibers to find use in important applications in medicine and spectroscopy.

4.9 Germanium core, Zinc Selenide cladding for imaging applications

Novel waveguide geometry for low loss infrared waveguides with sub-wavelength core dimensions has been proposed in Chapter 2. As the core diameter of germanium is reduced, the guided mode tends to leak into the silica cladding resulting in a high propagation loss. By introducing a layer of ZnSe as the cladding, the interaction of the guided mode with silica can be minimized with an associated reduction in the propagation loss. This concept has been numerically verified. By adopting the deposition strategy for ZnSe and Ge discussed in chapter 3, core-cladding structures were fabricated with varying ZnSe cladding thicknesses inside a silica capillary as shown in Figure 4-16.

A continuous wave CO₂ laser at 10.6µm is employed to characterize the waveguides illustrated in Figure 4-16 with sub-wavelength dimensions. The guided mode recorded on the pyroelectric camera is illustrated in Figure 4-17. The resolution of the
Table: Waveguide Geometries with Experimental and Numerically Simulated Propagation Loss Values

<table>
<thead>
<tr>
<th>Geometries</th>
<th>Experimental</th>
<th>Numerical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geometric</td>
<td>21 dB/mm</td>
<td>17 dB/mm</td>
</tr>
<tr>
<td>Geometric</td>
<td>0.25 µm</td>
<td>0.73 µm</td>
</tr>
</tbody>
</table>

Profile of the guided mode (c, e) Optical micrographs of the masks fabricated at the input face to test for cross talk and experimentally simulated propagation loss values is presented to highlight the common trend.

Optical micrograph of a Ge under plane wave illumination before and after fabrication.
guided mode is limited by the point spread function of the reflecting objective which was measured to be ~21 µm. The variation in waveguide transmission as a function of the cladding layer thickness is investigated. From the experimental data, the optimum thickness of the ZnSe layer required for maximum waveguide transmission was estimated to be ~ 2 µm. The observed trend seems to agree reasonably well with numerical simulations shown in Figure 4-16. The discrepancy in the absolute values can be attributed to the multi-mode waveguide behavior and material impurities. These low loss infrared waveguides can be used for imaging applications by fabricating an array of these in a microstructured optical fiber by adopting a similar deposition recipe for ZnSe and Ge. An array of Ge-ZnSe waveguides is fabricated within the pores of silica based MOF as shown in Figure 4-18.

![Fabricated array of waveguides](image)

Figure 4-18: Optical micrograph of the polished facet of the array of Ge-ZnSe waveguides.
In order to characterize the inter-pixel isolation in the array of waveguides, a uniform layer of metal (gold) was coated on the input face. Two waveguides (pixels) were exposed by focused ion beam (FIB) assisted milling of the metal at their input face as shown in Figure 4-17. Under illumination, the intensity distribution of the light at the output did not reveal any cross-talk between adjacent waveguides. Furthermore, to demonstrate rudimentary imaging capabilities, the waveguides forming the letters “P S U” were exposed by FIB milling as shown in Figure 4-17. Under illumination, the pattern “P S U” at the input was found to be transferred to the output plane. The variation in the intensity profile of each waveguide/pixel can be attributed to the variation in waveguide transmission arising due to the varying cladding thicknesses. The same experiment performed at a wavelength of 3.39 µm demonstrating near field image transfer is illustrated in Figure 4-19. The guided mode through each individual waveguide is very well resolved at this wavelength since its dimension is larger than the point spread function of the imaging system. The same structure is fabricated in a tapered MOF template for demonstrating the concept of magnification in the next chapter.

![Intensity pattern recorded on the infrared camera at 3.39 µm demonstrating the near field image transfer characteristics of the Ge-ZnSe array](image)
4.10 Doped semiconductor junctions

In the electronic approach to sub-wavelength imaging, doped semiconductor junctions (P-I-N) or metal-semiconductor schottky junctions can be fabricated within an array of sub-wavelength holes. The junction within each capillary behaves like a photo-detector / pixel. The advantage of this approach is that it can be used for imaging in the visible regime where silicon and germanium are highly absorbing. One of the major hurdles in this approach has been overcome by Dr. Rongrui He, who developed the capability of depositing N-type, P-type and intrinsic silicon inside the capillary. Raman spectroscopic analysis was employed to study the doping profile in the P-I-N junctions. The Raman spectrum of doped Silicon exhibits a shift in the center frequency, broadening and asymmetry in the first order Raman peak centered at around 520 cm\(^{-1}\). The presence of holes and electrons in doped Silicon, produces an inter-valence-band, electronic and broad scattering background which overlaps with the one-phonon Raman line. This overlap results in a Fano type resonance which has been well established in the literature.

The Raman intensity according to the Fano’s theory\(^{27}\) proportional to:

\[
\frac{(q\Gamma / 2 + \omega - \omega_p)^2}{(\omega - \omega_p)^2 + (\Gamma/2)^2};
\]

where \(\Gamma\) is the peak width, \(\omega\) is the frequency, \(\omega_p\) is the center frequency, and \(q\) is the doping parameter inducing asymmetry in the peak. The \(q\) parameter is positive when the doping is P type and negative when it is N-type. The 2-D spatial variation of the peak width, center frequency, and \(q\) factor across the P-I-N junction are extracted from the Raman spectra recorded using a confocal scanning system. Figure 4-20 (a), (b) and (c) illustrates the variation of peak position, peak width and the \(q\)-parameter. The innermost
Figure 4-20: Two dimensional R and (c) correspond to the variation of the peak position, peak width and the q-parameter across the annular junction respectively.

Figure 4-21: Left panels illustrated the experimentally guided modes in a p-i-n junction, whereas the right panels show the corresponding numerical simulations.
p-type region, the middle I layer and the outermost N-type region can be easily recognized from these plots.

Light guiding experiments at 1.55 um have been carried out in the P-I-N junctions deposited inside the glass capillary. The spatial intensity distribution of various guided modes shown in Fig.15 was recorded using a CCD camera. They seem to match well with the mode structure calculated by finite element modeling as shown in Figure 4-21. The propagation loss in these structures has been estimated to be ~ 25dB/mm. However, efforts are being made to reduce the propagation loss by incorporating hydrogen in amorphous Silicon. The lowest value measured until now is 3 dB/cm. The propagation loss associated with various metal contacts was simulated by finite element modeling. The same fabrication can be extended to an array of holes.

4.12 Discussion and Conclusions

The best optical transmission characteristics of various materials fabricated within the silica capillary measured have been summarized in Table 4-4. Crystalline ZnSe has been found to exhibit the best optical transmission (<1 dB/cm), followed by amorphous hydrogenated silicon (3dB/cm) in the near infrared regime. Infrared waveguides with sub-wavelength dimensions have also been fabricated for imaging applications. The contribution to propagation loss in most of these waveguides seems to be materials quality. In order to improve the properties, the processing conditions can be fine-tuned and the fabrication could be carried out in a cleaner environment under ultra-high vacuum similar to the conditions being adopted in semiconductor industries.
<table>
<thead>
<tr>
<th>Material</th>
<th>Wavelength (micron)</th>
<th>Core diameter (micron)</th>
<th>Optical propagation Loss (dB/cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amorphous Silicon</td>
<td>1.55</td>
<td>5.8</td>
<td>12</td>
</tr>
<tr>
<td>Polycrystalline Silicon</td>
<td>1.55</td>
<td>5.8</td>
<td>7</td>
</tr>
<tr>
<td>Amorphous hydrogenated Silicon</td>
<td>1.55</td>
<td>6.2</td>
<td>3</td>
</tr>
<tr>
<td>Zinc Selenide</td>
<td>1.55</td>
<td>50</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Silicon Nitride</td>
<td>1.55</td>
<td>6.2</td>
<td>11</td>
</tr>
<tr>
<td>Germanium</td>
<td>10.64</td>
<td>8</td>
<td>160</td>
</tr>
<tr>
<td>Ge/ZnSe</td>
<td>10.64</td>
<td>8</td>
<td>135</td>
</tr>
<tr>
<td>Silicon pin Junction</td>
<td>1.55</td>
<td>15</td>
<td>250</td>
</tr>
</tbody>
</table>
References


27. U Fano, Phys Rev, 124, 1866 (1961)
Chapter 5

A magnifying fiberscope for sub-wavelength imaging

5.1 Introduction

Infrared imaging is critically important to wide-ranging fields including defense, medicine, chemical sensing, semiconductor inspection, thermal imaging, and scientific research\textsuperscript{1-3}. Commonly employed infrared imaging tools are predominantly based on solid immersion lenses (SIL)\textsuperscript{4-9}, near-field scanning optical microscopy (NSOM) and coherent fiber-optic bundles\textsuperscript{5}. The techniques based on SIL and NSOM offer exceptional spatial resolution, but are extremely time intensive due to the serial scanning natures of these methods. They are also limited only to samples that can be brought to the microscope stage. Hence fiber-optic bundles are a preferred candidate for (a) imaging specimens with restricted optical access in a minimally invasive manner, (b) characterizing the dynamic response and (c) time sensitive samples. A few specific applications include in-vivo cellular imaging, minimally invasive endoscopic medical imaging, and gas sensing inside chambers with limited optical access\textsuperscript{11-13}. The current technology for infrared fiber-optic imaging bundles is based on an array of either hollow/chalcogenide core waveguides. These imaging bundles with the best resolution so far have a modest pixel size of the order of 50 µm (approximately 5 times the wavelength of light)\textsuperscript{5}. The resolution of the imaging bundles seems to be predominantly limited by the inter-pixel cross talk. Therefore, aimed at improving the resolution, we demonstrate
the proof-of-concept of an infrared *fiberscope* based on a microstructured optical fiber with sub-wavelength resolution.
<table>
<thead>
<tr>
<th>Technique</th>
<th>λ</th>
<th>Resolution</th>
<th>Notes</th>
<th>Reference</th>
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</thead>
<tbody>
<tr>
<td>SIL</td>
<td>1.3 µm</td>
<td>0.15 µm</td>
<td>Silicon Lens</td>
<td>Hammamatsu NanoLens spec sheet</td>
</tr>
<tr>
<td>Fiber array</td>
<td>Visible</td>
<td>8 - 20 µm</td>
<td>Glass fibers</td>
<td>Donald M Chiarulli, White paper, Schott fiber optics</td>
</tr>
<tr>
<td>Fiber array</td>
<td>0.8 µm</td>
<td>600 µm</td>
<td>10000-30000 fibers</td>
<td>Development of fiber image bundle, Tokyo Electronics, Japan</td>
</tr>
<tr>
<td>Detector array</td>
<td>0.2 µm</td>
<td>30 λ</td>
<td>Array of electron multipliers, used for X Rays</td>
<td>Nuclear Instr. and Methods, 162, 587 (1979)</td>
</tr>
<tr>
<td>SIL</td>
<td>0.44 µm</td>
<td>0.1 µm</td>
<td>Using a glass lens of high refractive index (2)</td>
<td>Appl. Phys. Lett, 57, 2615 (1990)</td>
</tr>
<tr>
<td>SIL</td>
<td>0.405 µm</td>
<td>0.1 µm</td>
<td>Using a Lens with a high refractive index (2)</td>
<td>G S Kino et al, SPIE Proc, 1556, 2 (1991)</td>
</tr>
<tr>
<td>SIL</td>
<td>0.7 µm</td>
<td>λ/4</td>
<td>Used for high density recording</td>
<td>G S Kino et al, Opt. Lett, 18, 305 (1993)</td>
</tr>
<tr>
<td>Fiber array</td>
<td>3-5 µm</td>
<td>45 µm</td>
<td>Chalcogenide fibers, 1000-4000 pixels, &lt; 1dB/m</td>
<td>J. Non-cryst. Solids, 184, 40 (1995)</td>
</tr>
<tr>
<td>SIL</td>
<td>0.44 µm</td>
<td>0.19</td>
<td>Lens mounted on the cantilever probe</td>
<td>G S Kino et al, Appl. Phys. Lett, 74, 501 (1999)</td>
</tr>
<tr>
<td>Scanning SIL</td>
<td>0.442 µm</td>
<td>0.15</td>
<td>Superspherical lens mounted on cantilever probe</td>
<td>Appl. Phys. Lett, 72, 2779 (1998)</td>
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<tr>
<td>Confocal SIL</td>
<td>0.633 µm</td>
<td>0.19</td>
<td></td>
<td>Appl. Phys. Lett., 77, 3459 (1999)</td>
</tr>
<tr>
<td>SIL</td>
<td>0.56 µm</td>
<td>0.13</td>
<td>Gallium Phosphide lens, high refractive index, NA=2</td>
<td>Appl. Phys. Lett, 75, 4064 (1999)</td>
</tr>
<tr>
<td>Fiber array</td>
<td>4-10 µm</td>
<td>700</td>
<td>Silver halide fiber bundle for chemical sensing</td>
<td>Appl. Opt, 39, 1112 (2000)</td>
</tr>
<tr>
<td>SIL + 0.5 µm</td>
<td>9.3 µm</td>
<td>λ/10</td>
<td>10-1000 times better throughput than tapered fiber</td>
<td>Optical MEMS IEEE/LEOS International Conference, 133, ‘Microfabricated SIL with metal aperture’, DA Fletcher et al (2000)</td>
</tr>
<tr>
<td>SIL + cantilever</td>
<td>0.4 µm</td>
<td>0.133</td>
<td>Silicon Nitride lens, NA=1.8</td>
<td>J. MEMS, 11, 470 (2002)</td>
</tr>
<tr>
<td>Fiber array</td>
<td>2-10 µm</td>
<td>5-35</td>
<td>Chalcogenide fiber</td>
<td>US Patent 6175678B1</td>
</tr>
<tr>
<td>Fiber array</td>
<td>2-12 µm</td>
<td>50</td>
<td>Hollow metallic waveguides</td>
<td>Opt Eng, 43, 1195 (2004)</td>
</tr>
<tr>
<td>Fiber array</td>
<td>2-12 µm</td>
<td>40</td>
<td>Ag/AgI waveguides</td>
<td>Optical Fibers and Sensors for Medical Applications IV, Proc of SPIE, 5317, 94 (2004)</td>
</tr>
<tr>
<td>SIL array</td>
<td></td>
<td></td>
<td>Details on fabrication of GaP SIL arrays</td>
<td>US Patent 2004/0085644 A1</td>
</tr>
<tr>
<td>SIL</td>
<td>1.2 µm</td>
<td>0.3</td>
<td>Semiconductor inspection</td>
<td>JJAP, 44, 3385 (2005)</td>
</tr>
<tr>
<td>SIL</td>
<td>1.2 µm</td>
<td>0.23</td>
<td>Subsurface microscopy</td>
<td>APL, 78, 4071 (2001)</td>
</tr>
</tbody>
</table>

Table 5-1: Summary of the research work in the area of imaging using solid immersion lenses, near field scanning microscopy and coherent imaging fiber bundles.
5.2 Conceptual Design

A silica based microstructured optical fiber (MOF) similar to the one illustrated in Figure 5-1 (a) is used as a starting template to fabricate an array of infrared waveguides within each of the capillaries. The size and the spatial arrangement of the capillaries in the MOFs can be precisely engineered. Within each of the capillaries in the MOF, infrared waveguides are fabricated by the high pressure assisted chemical fluid deposition technique discussed in Chapter 3. As explained previously, the highly versatile deposition technique can be employed to fabricate a wide gamut of materials including crystalline, amorphous, hydrogenated & doped semiconductors, metals and compound semiconductors. This gives us a wide range of materials to choose from for designing the fiberscope. Each of these cylindrical waveguides fabricated by this method acts like a pixel. The resolution of the fiberscope is defined by the diameter of the individual waveguides (pixels). In order to attain sub-wavelength resolution, the waveguides fabricated inside each of the capillaries of the MOF can be tapered down to have sub-wavelength dimensions at the input as illustrated in Figure 5-1 (b). In this design, a magnified image will be transmitted from the input end with sub-wavelength resolution to the un-tapered output end of the MOF. The diameter of each waveguide at the un-tapered output end can be chosen to match the pixel size of commercially available photo-detectors or focal plane detector arrays for recording the magnified image. Tapering the waveguide down to sub-wavelength dimensions is conceptually feasible since the fundamental HE_{11} mode in symmetric cylindrical waveguides have no cut-off diameter in contrast to the hollow metallic waveguides which are currently used in
imaging bundles. The proof-of-concept of the proposed design has been demonstrated at 1.55\(\mu\m\), 3.39\(\mu\m\) and 10.64\(\mu\m\) wavelengths.

![Graphs illustrating the effect of broadening with decreasing wavelength.](image)

The real (n, solid line) and imaginary (k, dotted line) values of refractive index for (a) Germanium and (b) Silica in the infrared wavelength regime have been demonstrated at wavelengths.

![Graphs illustrating the effect of broadening with decreasing wavelength.](image)
5.3 Material Selection

The choice of materials for designing each of the pixels in the MOF are based on the real \((n)\) and imaginary \((k)\) part of the refractive indices, and the ease of fabrication. For the core: a high \(n\) and a low \(k\) are preferred. Higher \(n\) leads to a strong confinement of the guided mode resulting in reduced cross-talk between pixels. This increases the potential for the maximum achievable resolution. Lower \(k\) results in reduction of waveguide propagation losses, thereby resulting in enhanced sensitivity. Considering these factors, Germanium is a good candidate for the core with high \(n\) and very low \(k\) in the infrared regime. The refractive index of Germanium is illustrated in Figure 5-2(a)\(^{16}\).

The silica based MOF template, which is the surrounding matrix, is transparent in the visible regime, but has a strong absorption in the infrared as illustrated in Figure 5-2(b). As a consequence, it is an ideal choice for minimizing the cross-talk between pixels while maintaining the superior mechanical properties at the same time. However, with decreasing waveguide diameter, the guided mode profile tends to expand into the absorptive silica matrix resulting in reduction of waveguide transmission. Figure 5-3 illustrates the effect of decreasing waveguide diameter on the intensity profile of the guided mode.

Therefore, to reduce the interaction of the guided mode with the silica, a cladding layer of Zinc Selenide (low \(k\)) is introduced between the Germanium core and the Silica matrix as shown in Figure 5-1(c). This has been verified by finite element modeling and is found to increase the waveguide transmission as discussed in Chapter 4. Since Germanium does not transmit below 1.9\(\mu\)m wavelength, Silicon can be used instead in
the near infrared regime. Silicon, similar to Germanium, has a high real and a very low imaginary part of refractive index. Hence, a tapered array of silicon waveguides is used for demonstrating the proposed concept at 1.55 µm wavelength instead of a tapered array of Germanium core, Zinc Selenide cladding waveguides embedded in a silica matrix is used for demonstration at 3.39 µm wavelength.

5.4 An array of tapered Germanium waveguides

The fabrication of the tapered array of waveguides involves a series of complicated steps. In order to simplify the problem, a single tapered waveguide was fabricated in a silica capillary. After determining the process parameters for fabricating a single tapered waveguide with the desired geometry, the same recipe was extended to fabricate an array of tapered waveguides. The high pressure chemical fluid deposition technique discussed in Chapter 3 is employed for fabrication of waveguides within the pores of the MOF. Tapered waveguides can be fabricated by either depositing them in un-tapered silica capillaries, followed by a tapering routine, or by tapering the silica capillary template followed by a deposition routine. The latter approach is preferable due to the mismatch in the mechanical properties of Germanium (Ge), Zinc Selenide (ZnSe) and Silica which might lead to interfacial cracking while tapering the array of Ge-ZnSe waveguides in a silica matrix. Therefore, the silica capillary is tapered to the desired geometry and used as a template for deposition of the waveguides. For tapering the silica templates, a commercially available Ericsson fusion splicer shown in Figure 5-4 is employed to taper the silica MOF template. An electric arc is used to locally heat the
region to be tapered, and the fiber is simultaneously pulled with uniform longitudinal force in opposite directions. The arc current and the pull time can be varied to obtain the desired tapered geometry.

To start with, the two magnetic chucks which pull the fiber in opposite directions are aligned using two cleaved, standard single mode fibers. This ensures that the tapered region is symmetric and reproducible. Cleaning the electrodes before tapering improves the reproducibility of the taper parameters. A silica fiber with a single capillary with 30 µm diameter was used as the template. The polymer coating covering the silica fiber is removed prior to tapering to avoid contamination of the CCD sensor array in the in-built

Figure 5- 4: An Ericsson fusion splicer which can also be used for tapering silica fibers, http://www.surpluseq.com/
camera which enables real-time video while the fiber is being tapered. The two magnetic chucks are securely fastened to hold the fiber in tension such that there is no slack. The arc current was varied and pulled for 5 s to get the desired taper geometry. As illustrated in Figure 5-5 (a-d), the taper waist tends to decrease with increasing arc current.

After tapering the fiber, it was thoroughly inspected under the microscope to ensure that the desired taper geometry is obtained, and is also free from randomly occurring defects as shown in Figure 5-5 (e, f). Extra care is taken to ensure a dry environment while cleaving the ends of the silica fiber to prevent any water/acetone/methanol from getting into the capillary rendering it unusable for deposition.
Figure 5-6: An Ericsson fusion splicer which can also be used for tapering silica fibers.

Figure 5-7: Optical micrograph of tapered silica capillary (a) before and (b) after Ge deposition, scale bar 30 µm.

A) Before deposition

B) After Ge deposition

1) before
Figure 5-8: Optical micrographs of the polished input (narrow) and output (wider) facet of a tapered Germanium waveguide at 20 x and 100 x magnification.

Figure 5-9: Scanning electron micrograph of a silica based microstructured optical fiber template used for fabricating an array of tapered Ge waveguides, SEM by Rongrui He.
To start with, a single tapered Ge waveguide was fabricated using the high pressure chemical fluid deposition technique discussed in detail in chapter 3. The most critical parameter to be determined in the fabrication of tapered waveguides is the position of the tapered region inside the furnace to ensure complete filling at the narrow end of the taper (waist). In order to determine this, the deposition profile of Ge in an untapered silica fiber with a 30 µm diameter is studied as illustrated in Figure 5-6. Thus, from the deposition profile, it is clear that the taper waist should be positioned in the 6-7 cm inside the furnace for complete filling. Having determined the position, a silica fiber with a 30 µm capillary was tapered in the fusion splicer at an arc current of 11.9 mA and pulled for 5 s before using it as the template as shown in Figure 5-7. The tapered section of the fiber is polished to optical grade using colloidal silica as shown in Figure 5-8. The length of the tapered region is ~ 300 µm, diameter of the narrow (input) end is ~ 4 µm and the wider (output) end is ~31 µm resulting in a magnification of ~ 8 X. The tapered waveguide is fully filled at the input, however only partially filled at the output with ~ 10 µm hole in the middle. The hole in the middle can be fully filled by either increasing the duration or temperature of the deposition. This tapered waveguide can be used as a near field probe for sub-wavelength imaging applications at 10.64 µm. Having determined the processing conditions for a single tapered waveguide, the same recipe was extended to fabricate an array of tapered Ge waveguides. A microstructured optical fiber shown Figure 5-9 was used as the starting template.

It has 168 holes in a hexagonal arrangement, with a center-center spacing of 15 µm between each other. The diameter of each of the holes is ~ 13 µm and the overall outer diameter of the MOF silica fiber is ~ 285 µm. In order to determine the location at
which the tapered region should be positioned inside the furnace, the deposition profile of Ge inside and un-tapered MOF template was monitored under similar deposition conditions (no-flow). The deposition temperature inside the furnace was increased to 525°C for reducing the deposition time, and ensure complete filling of the holes unlike the single tapered waveguide. After allowing the deposition for 2 days, the deposition profile inside the furnace was revealed by cross-sectional scanning electron microscopy as a function of the position inside the furnace as illustrated in Figure 5-10.

Figure 5-10: Cross-sectional scanning electron micrographs of the MOF array after Ge deposition revealing the deposition profile, SEM by Justin Sparks
On increasing the deposition temperature, the fully filled region shifted closer to the entrance of the furnace (3-4 cm). In addition, the time required for deposition was observed to drastically reduce. Therefore, the position at which the tapered region needs to place was determined to be in between 3 to 4 cm inside the furnace since they are fully filled. This can be seen clearly from Figure 5-11. Next, the MOF template was tapered using the Ericsson fusion splicer. The arc current and the pull time were iteratively varied as described previously to obtain the desired taper geometry as shown in Figure 5-12.

![Cross-sectional SEM of the MOF at 3, 4 cm inside the deposition furnace](image)

**Figure 5-11**: Cross-sectional SEM of the MOF at 3, 4 cm inside the deposition furnace, SEM by Justin Sparks

The arc current was set at 14.8 mA and pulled for 5 s to produce the taper shown below. Alignment of the two magnetic chucks is of extreme significance to achieve uniform tapering of all the 168 holes/pixels. When the chucks are not aligned properly, the extent of tapering is found to increase along the radius from the center. The fusion
Splicer is also used to seal off the end of the MOF template for carrying out the deposition under no-flow conditions. The front end of the fiber is cleaved using a standard fiber cleaver shown in Figure 5-13.
After cleaving at the front end, the facet is inspected under an optical microscope to ensure that all the holes unplugged with debris/dirt. This is important for the deposition to occur uniformly in all the holes. The cleaved end is then attached to the reservoir and the Ge deposition is carried out at 525°C for 2 days under no-flow conditions. After deposition is complete, the tapered region of interest is polished using colloidal silica as described in Chapter 3. The wider end (output) of the taper was polished down until the waveguides were fully filled unlike the ones illustrated in Figure 5-14 for improving the transmission through each waveguide. The optical micrographs of the input (narrow) and output (wider) ends of the array of tapered waveguides are illustrated in Figure 5-15.
The diameter of each individual Ge waveguides at the input varies between 3.5-6 µm, the center-center spacing between Ge waveguides is ~ 10 µm and outer diameter of the overall silica fiber at the input is ~ 185 µm. At the output facet, the diameter of each of the individual Ge waveguides is ~ 13 µm, center-center spacing is 15 µm and the diameter of the MOF silica fiber is 285 µm. The length of the tapered region is 300 µm. This geometry results in an inherent pixel magnification of ~ 3X and a pitch magnification of 1.3 X. For instance, this structure can be used for imaging at 10.64 µm wavelength with a resolution of ~ 5 µm (λ/2). The magnification can be improved further by tuning the taper parameters. The fabricated fiberscope can be used in conjunction with commercially available imaging systems or butt-coupled to fiber-optic bundles for achieving sub-wavelength resolution.

The resolution, inter-pixel cross-talk and rudimentary imaging capabilities of the fiberscope are characterized using the experimental set-up illustrated in Figure 5-16. The
fiberscope is employed to transmit the magnified image of an object in the near-field with sub-wavelength features. In order to mimic an object with sub-wavelength feature size, a metal mask representing the letters “P S U” was fabricated at the input (narrow) end of the fiberscope as depicted in Figure 5-17.

fore and after ricated gold mask
In order to fabricate the metal mask, a uniform layer of 100 nm Gold + 10 nm
Chromium (adhesion layer) was deposited on the input facet by a Kurt. J. Lesker electron beam assisted evaporator shown in Figure 5-18.

Prior to mask fabrication, the silica surrounding the input facet was etched in buffered HF for 30 s to generate a topographical contrast between the Ge waveguides and the surrounding matrix. This is of paramount importance for mask fabrication, since the contrast is very weak in the ion image, which is primarily used as an aid for milling the metal off to make the P S U pattern. The chromium layer is hard to mill off, and needs long exposures to high energy ion beams. Therefore, its thickness is minimized as much as possible. Coupling of light into the input facet becomes very challenging when the entire region is covered with metal. Therefore, to aid in coupling of light into the P S U pattern, some regions of the silica capillary which act like a housing for the fiberscope are left uncovered without any metal coating. This is achieved by masking it with polyimide tape. This was found to greatly help in getting feedback while coupling light into the P S U pattern which is not possible if the input facet is completely covered with metal.

The metal layer covering the waveguides forming the letters ‘P S U’ was selectively milled by a focused ion beam (FIB) assisted milling technique using a FEI Quanta 200 3D Dual beam system. The ion current used for milling was between 0.1 nA and 0.5 nA. The milling parameters (ion current and time) were determined by monitoring the specimen current while milling the metal layer from a similarly sized dummy region far away from the input facet. The image transfer capability of the fiberscope is characterized using the experimental set-up described in Figure 5-16.

A continuous wave carbon-dioxide laser at 10.64 µm and a Helium-Neon laser at 3.39 µm were both used to uniformly illuminate the near-field ‘P S U’ pattern. Two gold
mirrors (M1, M2) are iteratively adjusted to align the laser beam along the desired direction. An anti-reflection coated Silicon lens (L) and a Zinc Selenide lens with a focal length of 25.4 mm was used for focusing the 3.39 µm and 10.64 µm laser beam onto the P S U pattern respectively. The fiberscope (F) was mounted on an XYZ translation stage to ensure that the P S U pattern on the input facet of the fiberscope is positioned at the focal plane of the focusing lens. A 52x reflection objective (Ealing) with 0.65 NA was used for collecting the magnified image transmitted through the fiberscope. The magnified image is focused onto a broadband pyroelectric camera (Electrophysics PV 320). A thermal power head (Laserprobe Inc. RK 575, RK5720) is used to measure the output power. Under uniform illumination, the pixels in the P S U pattern at the input were magnified 3.5 times and found to be transmitted to the output plane. The intensity pattern recorded on the pyroelectric camera at both 3.39 and 10.64 µm wavelengths are shown in Figure 5-19. The variation in the intensity profile of each of the exposed waveguide/pixel can be attributed to the variation in germanium core diameters. The uniformity can be improved by further optimization of the processing conditions and also while tapering the MOF template.
In addition, a numerical simulation based on finite element modeling (COMSOL) of the proposed design at 10.64 µm under localized illumination of the pixels forming the pattern PSU is illustrated in Figure 5-19 (f). It can be clearly observed from numerical simulations that the interpixel cross-talk is minimal in the proposed design. The experimentally measured cross-talk between pixels is found to be ~ 11% in comparison to the 2% predicted by numerical modeling. In theory, the metal coating is supposed to improve the cross-talk as discussed in Chapter 2. The transferred image looks fuzzier at longer wavelengths due to the resolving capability of the reflection objective being used.
for characterization. The point spread function of the reflection objective was experimentally measured to be $\sim 2\lambda$.

The proof-of-concept of infrared imaging at sub-wavelength resolution using a fiberscope was demonstrated. A near field image with a resolution of less than 5 µm is transmitted with an in-built magnification of 3X at a wavelength of 3.39 µm and 10.64 µm. The resolution can be further improved by tuning the parameters for aggressively tapering the MOF template followed by a similar approach in fabrication. The fiberscope can be used for various imaging applications anywhere in the broad transmission window of Germanium between 2 – 15 µm. The design can be extended for imaging in the visible and near infrared regime by appropriate choice of materials for the waveguide core. The optical throughput through the germanium waveguides can also be improved in the infrared regime by introducing a ZnSe cladding layer to minimize the interaction of the guided mode with the lossy silica matrix. In order to demonstrate the same concept at 1.55 µm wavelength, tapered silicon waveguides can be used instead of Germanium.

5.5 An array of tapered silicon waveguides

Amorphous hydrogenated silicon is found to have the best optical properties at 1.55 µm as discussed in Chapter 4. The recipe for fabricating a single tapered silicon waveguide is first established and extended to an array similar to the strategy adopted for fabricating an array of tapered Ge waveguides. The deposition recipe for amorphous hydrogenated silicon has been described in detail in Chapter 3. The deposition was carried out under no-flow condition. The deposition profile in the furnace at a set point of
450 C was characterized and the fully filled region was found to range in-between the 3-6 cm inside the furnace. Therefore, the tapered region of the silica capillary template needs to be placed at 4 cm inside the furnace to achieve complete filling. To start with, a silica fiber with a capillary diameter of 30 µm was tapered using the Ericsson fusion splicer at an arc current of 11.9 mA and pulled for 5 s. For restricting the flow through the capillary, the output end was sealed by tapering the fiber at 15 mA. The deposition was allowed to proceed for 4 days in an unrestricted manner. The optical micrograph of the tapered silicon waveguide deposited inside a tapered silica capillary is illustrated in Figure 5-20. The relevant portion of the tapered waveguide was cleaved and polished using colloidal silica as shown in Figure 5-20.
The diameter of the tapered silicon waveguide at the input is \( \sim 2 \, \mu m \) and \( \sim 32 \, \mu m \) at the output end. This leads to an in-built pixel magnification of 16X. The length of the tapered region is \( \sim 300 \, \mu m \). The output end of the tapered waveguide is not fully filled, and has a \( \sim 10 \, \mu m \) hole in the middle. This problem can be solved by increasing the deposition temperature and allowing the deposition to occur over longer duration. However, presence of a hole at the output is not detrimental to the resolution achievable while imaging. The overall transmission of the waveguide might slightly reduce due to the presence of a hole thereby lowering the sensitivity of the probe.
Having established the deposition conditions for a single tapered silicon waveguide, the same recipe was extended to fabricate an array of silicon waveguides. The microstructured optical fiber template shown in Figure 5-21 was tapered down using the Ericsson fusion splicer. The MOF template has 167 capillaries with a diameter of 9 µm; center-center spacing of 13 µm; and an overall silica diameter is 260 µm. The MOF silica fiber was tapered at an arc current of 14.8 mA and pulled for 5 s. This results in a 300 µm tapered section as shown in Figure 5-22. Alignment of the two magnetic chucks in the splicer is of extreme significance to achieve uniform tapering of all the 167 holes/pixels. When the chucks are not aligned properly, the extent of tapering is found to increase along the radius from the center. The fusion splicer is also used to seal off the end of the MOF template for carrying out the deposition under no-flow condition. After cleaving at the front end, the facet is inspected under an optical microscope to ensure that all the holes unplugged without any debris/dirt. This is important for the deposition to
occur uniformly in all the holes. The cleaved end is then attached to the reservoir and the Si deposition is carried out at 350°C for 7 days in Furnace E under no-flow conditions. The tapered region is placed 6.5 cm inside the furnace. After deposition is complete, the tapered region of interest is polished using colloidal silica as described in Chapter 3. The optical micrographs of the input (narrow) and output (wider) ends of the array of tapered waveguides are illustrated in Figure 5-23.

The tapered array of silicon waveguides are not fully filled under the deposition conditions similar to the one used for the single tapered silicon waveguides. Even though the silicon tubes are not ideal for imaging applications, they can be used for the demonstrating the proof-of-concept of imaging at 1.55 μm. The diameter of each the individual Si waveguides at the input facet (narrow) varies between 2-2.5 μm, the center-center spacing between Si waveguide is ~ 9.5 μm and outer diameter of the overall silica
fiber at the input is ~ 195 µm. The thickness of silicon is ~ 1 µm. At the output facet, the
diameter of each of the individual Si waveguides is ~ 8.5 µm, center-center spacing is
13.5 µm and the diameter of the MOF silica fiber is 260 µm. The thickness of silicon is ~
1.5 µm. The length of the tapered region is 300 µm. This geometry results in an inherent
pixel magnification of ~ 3.5 X and a pitch magnification of ~1.4 X. This structure can be
used for imaging anywhere in the broad transmission window of silicon. For instance, at
a wavelength of 1.55 µm, an image with a resolution of ~ 2.5 µm can be transmitted
using this structure without any appreciable cross-talk. The built-in magnification can be
improved by tuning the taper parameters. The fabricated fiberscope can be used in
conjunction with commercially available imaging systems or butt-coupled to fiber-optic
bundles for achieving sub-wavelength resolution.

The resolution, inter-pixel isolation and preliminary imaging transfer capabilities
of the fiberscope are characterized using the experimental set-up similar to the one

illustrated earlier in Figure 5-15 at a wavelength of 1.55 µm. The tapered array of silicon waveguides is employed to transmit the magnified image of an object in the near-field with sub-wavelength features. In order to mimic a near-field object, a metal mask representing the letter ‘P’, the shape ‘→’ were fabricated at the input (narrow) end of the fiberscope as depicted in Figure 5-24.

The metal mask (100 nm Au + 100nm Cr) was fabricated on the input facet of the polished sample as discussed previously in Chapter 3. A uniform layer of metal was deposited by electron-beam assisted thermal evaporation and the desired pattern was fabricated by focused ion beam assisted milling. Prior to metal deposition, the sample was etched in buffered HF for 30 s to etch the surrounding silica and induce topographical contrast which acts as an aid while fabricating the metal mask. The image transfer capability of the fiberscope is characterized using the experimental set-up similar to the one shown Figure 5-16.

A continuous wave tunable diode laser (Agilent) at 1.55 µm was used to uniformly illuminate the near-field pattern. The laser beam exiting the single mode fiber from the Agilent diode laser was collimated using a collimating lens from Thorlabs. Two silver mirrors (M1, M2) are iteratively adjusted to align the laser beam along the desired direction. A plano-convex lens made of BK7 glass with a focal length of 25.4 mm was used for focusing the 1.55 µm laser beam onto the metal mask at the input facet of the fiberscope. The fiberscope (F) was mounted on a piezo-electrically controlled XYZ translation stage to ensure that the metal pattern on the input facet of the fiberscope is positioned at the focal plane of the focusing lens. A 60x objective (Newport) with 0.85 NA was used for collecting the magnified image transmitted through the fiberscope. The
magnified image is focused onto a broadband near infrared camera (Hammamatsu).

Under localized illumination of a single pixel, the cross-talk is measured to be less than 5% as shown in Figure 5-25 (b).
Under uniform illumination of the pixels in the ‘P’ and ‘\rightarrow’ pattern at the input were magnified 3.5 times and found to be transmitted to the output plane with minimal cross-talk between pixels. The intensity pattern recorded on the infrared camera at 1.55 µm is shown in Figure 5-25 (c, d). The variation in the intensity profile of each of the exposed waveguide/pixel can be attributed to the variation in silicon core diameters. The uniformity can be improved by further optimization of the processing conditions and also while tapering the MOF template. In addition, a numerical simulation based on finite element modeling (COMSOL) of the proposed design at 1.55 µm under uniform illumination of the pixels forming the pattern \rightarrow is illustrated in Figure 5-26. It can be clearly observed from numerical simulations that the inter pixel cross-talk is minimal in the proposed design. The experimentally measured cross-talk between pixels is found to be \sim 5 \% in comparison to the 2% predicted by numerical modeling.
5.6 An array of tapered Germanium core- ZnSe cladding waveguides

In order to improve the optical transmission through the silicon waveguides by minimizing the interaction of the guided mode with silica, an attempt was made to introduce a layer of ZnSe between the silicon and silica. The deposition was attempted in a fiber with a single capillary with 8 µm diameter. However, it resulted in a dendrite like growth of Si over ZnSe as shown in Figure 5-27.

The underlying reason for this observation is not fully understood. The processing conditions need to be optimized further in order to achieve uniform growth of silicon over ZnSe.
One possible reason for this wire like growth could be due to relatively high deposition temperature of silicon (400 C) resulting in the precipitation of zinc rich islands out of the ZnSe layer. These zinc rich islands may act as catalysts for the wire type growth of silicon analogous to the VLS growth. Therefore, germanium, which has a lower deposition temperature (300C) than silicon, and has also been deposited successfully over ZnSe (Chapter 4), might be a better choice for the core material while ZnSe is the cladding. The conditions for depositing germanium and Zinc Selenide inside the pores of the silica fiber were similar to those discussed in Chapter 3 and 4. The fabrication of the Ge-ZnSe waveguide geometry is a two-step serial process. The deposition of ZnSe is under flowing conditions, followed by germanium under no-flow conditions. After ZnSe deposition, the silica fiber template is detached from the reservoir. The output end is sealed by tapering at a high arc current using the fusion splicer and reattached to a different reservoir filled with a mixture of germane and helium. ZnSe deposition is done at 450 C whereas the Ge deposition is at a much lower temperature of 300 C. One critical parameter to be determined is the position of the tapered region inside the furnace for both depositions since the deposition rates are different. To determine this, the ZnSe and Ge deposition was carried out in a silica fiber with a single silica capillary with a 12 µm diameter. The deposition profile of Ge and ZnSe inside the furnace was determined by cross-section SEM as a function of the position inside the furnace as illustrated in Figure 5-28.
For ZnSe deposition at 450°C, the ideal position of the taper was determined to be 4 cm inside the furnace, whereas for Ge deposition at 300°C, the ideal position was determined to be 6.5 cm inside furnace. The deposition sequence was attempted in a silica fiber with a single capillary diameter of 30 μm illustrated in Figure 5-29. The silica fiber template is tapered using the fusion splicer at an arc current of 12.1 mA and pulled for 5 s. A thin layer of ZnSe is deposited first to act like a cladding, followed by Ge which acts as the core in the waveguide. The ZnSe deposition is carried out at 450°C for ~4 hours, followed by Ge for ~7 days. After deposition, the tapered region is polished.
with colloidal silica to optical grade. Optical micrographs of the silica fiber template at each stage of the fabrication sequence have been illustrated in Figure 5-29.

Figure 5-29: Optical micrograph of the tapered silica fiber at each step of the deposition process (d) is an optical micrograph with crossed polarizer for inspecting the uniformity of the ZnSe layer.

The waveguide geometry was monitored as the tapered region was polished. The deposition profile was observed to be asymmetric with respect to the taper. The ZnSe deposition, which is carried out under flowing conditions, is the underlying reason behind the asymmetry. The direction of the flow of precursors during the ZnSe deposition is
indicated by the red arrow in Figure 5-29(b). The ZnSe film is found to be darker in the region to the right of the taper compared to the left. On further inspection at higher magnification, illustrated in Figure 5-30, it is clear that the deposition profile of ZnSe is higher near the entrance of the furnace compared to the regions away from the entrance.

Figure 5-30: Optical micrograph of the deposition profile of ZnSe in a tapered silica template

The observation was reproducible even on varying the geometry of the tapered silica template as shown Figure 5-30 (a, b). This can be attributed to the tapered waist acting as a nozzle resulting in supersonic flow of precursors. Higher flow rates results in limited interaction time between the precursor molecules and the silica wall thereby resulting in lower deposition rates. This effect is accentuated by the fact that the
precursors are also depleted in concentration as we move away from the entrance of the furnace. The asymmetry in the deposition profile of ZnSe also results in its own tapered geometry. Therefore, the core diameter is not the smallest at the waist of the tapered silica template. It is observed to be off-centered towards the entrance of the furnace and occurs at the region where the rate of ZnSe deposition is the highest as illustrated in Figures 5-30, 5-31 and 5-32.

Figure 5-32: Optical micrographs of the deposition profiles of ZnSe and Ge at a higher magnification
The ZnSe film is also observed to be thicker on the side of the tapered region closer to the reservoir. In addition to this, the germanium deposition over the ZnSe layer was found to have a high deposition rate only in the region closer to the entrance of the furnace (right side of the taper). The waveguide geometry was investigated by carefully polishing the tapered region and observing it under an optical microscope in regular intervals. Figure 5-33 shows the input and output facets of the waveguide away from the furnace entrance / reservoir (left side of the taper). This could be due to the formation of a plug at the waist of the taper, after which there is no source of germanium precursor to the other side of the taper.

![Figure 5-33: Optical micrograph of the polished facet of the Ge-ZnSe tapered waveguide on the region away from the reservoir illustrating minimal germanium deposition](image)

The diameter of the tapered capillary is 3.5 µm at the input and 32 µm at the output resulting in a magnification of 8 X. At the output, the ZnSe cladding thickness is 4 µm and the Ge layer is only 1 µm thick. At the input facet, the individual thicknesses of the Ge and ZnSe layers are not resolvable and the combined thickness is 1.15 µm. The
length of the tapered region is 300 µm. Therefore, the useful part of the taper was the region closer to the reservoir/furnace entrance.

The first successful Ge-ZnSe tapered waveguide was fabricated after going through a dozen iterations. The primary reason for the failures were the randomly occurring defects during deposition, cracking under thermal stress, sample loss during polishing were the main reasons for the non-ideal samples. A sampling of the representative defects observed is summarized in Figure 5-34 and 5-35.

Figure 5-34: Optical micrographs of the two sources of defects occurring during fabrication
Figure 5-35: DIC microscopy of a randomly occurring defect
After 11 trials, a tapered germanium waveguide with ZnSe cladding with desired waveguide geometry was fabricated. The tapered region closer to the reservoir/furnace entrance was cleaved and polished using colloidal silica to reveal the waveguide geometry as shown in Figure 5-36.

Figure 5-37: Differential interference contrast microscopy of the polished input and output facets revealing the imperfections and surface roughness

The germanium core diameter at the input (narrow) facet was 3 µm with a ZnSe cladding thickness of 2 µm. At the output, the Ge core diameter was 28 µm and the ZnSe cladding thickness was 3 micron. This resulted in a magnification of 9 X. The length of the tapered region was 300 µm. The output end of the tapered waveguide is not fully filled, and has a ~9 µm hole in the middle. This problem can be solved by increasing the deposition temperature and allowing the deposition to occur over longer duration. However, presence of a hole at the output is not detrimental to the resolution achievable while imaging. The overall transmission of the waveguide might slightly reduce due to the
presence of a hole thereby lowering the sensitivity of the probe. The other issue with the ZnSe cladding layer is its surface roughness due to its crystalline nature. This is illustrated using differential interference contrast microscopy in Figure 5-37 to highlight the surface roughness and the imperfections arising during polishing of the facets.

Having established the deposition conditions for a single tapered Ge-ZnSe waveguide, the same recipe was extended to fabricate an array of Ge-ZnSe waveguides. In order to fabricate the structure proposed in the design, we start with the same silica based MOF template which was used for fabricating the tapered silicon waveguides shown in Figure 5-21. It has 168 capillaries (to-be-pixels) in a hexagonal arrangement. The diameter of each of these capillaries is 9 µm. The center-center spacing between adjacent capillaries is 13 µm. After fine tuning the process recipe for fabricating the waveguide geometry in a single capillary, the MOF silica template is tapered by using a standard fusion splicer. The region intended to be tapered is locally heated by an electric arc and pulled with uniform longitudinal force. The magnitude of the arc current and the time period for applying the longitudinal force are iteratively tuned until the desired taper geometry is obtained. The arc current is set at 14.8 mA and pulled for 5 s to taper each capillary in the MOF template to a diameter of 3µm. The length of the tapered section is 300 µm. The waveguides are then fabricated inside each of the capillary holes by adopting the same recipe developed for a single waveguide described above. The Zinc Selenide cladding is fabricated inside the MOF template followed by Germanium core. To deposit Zinc Selenide, Zinc and Selenium precursors are placed inside a high-pressure reservoir. The reservoir is then pressurized with hydrogen, which acts as a carrier gas and a reactant. This fluid mixture is then flowed through a heated MOF to carry out a high-
pressure chemical fluid deposition. A thin layer of crystalline Zinc Selenide is deposited at 450 C as shown in Figure 5-38.

Figure 5-38: Optical micrograph of the silica MOF template after each step during the fabrication
Following this, the output end of the fiber is sealed and amorphous hydrogenated Germanium is deposited at 300°C in a similar manner under no-flow conditions. Optical micrograph of the MOF template after each step is illustrated in Figure 5-38. After complete fabrication, the tapered region closer to the entrance of the furnace/reservoir is cleaved and chemo-mechanically polished to optical grade using colloidal silica. An optical micrograph of the polished facet of an array of tapered infrared waveguides with Germanium core, Zinc Selenide cladding infrared embedded within a silica matrix are shown in Fig 5-39.
The diameter of the Germanium core at the narrower (input) end is 2 \( \mu m \) and is 7 \( \mu m \) at the wider end (output). The Zinc Selenide cladding thickness at the input is < 1 \( \mu m \) and at the output is 1 \( \mu m \). The tapered geometry results in a resolution of 2 \( \mu m \) and an in-built magnification of 3.5 X. The uniformity in the thickness of the zinc Selenide cladding in each of the waveguides could be improved by tuning the taper parameters, and deposition conditions. The fabricated infrared fiberscope can be used in conjunction with commercially available imaging systems or butt-coupled to fiber-optic bundles for achieving sub-wavelength resolution.

![SEM images](image-url)
The rudimentary imaging capabilities of the fiberscope were characterized on the experimental set-up illustrated in Figure 5-16. The fiberscope is employed to transmit the magnified image of an object in the near-field with sub-wavelength features. In order to mimic an object with sub-wavelength feature size, a metal mask representing the figure ‘$\frac{\lambda}{N}$’ was fabricated at the input (narrow) end of the fiberscope as depicted in Figure 5-40.

In order to fabricate the metal mask, a uniform layer of 100 nm Gold + 10 nm Chromium (adhesion layer) was deposited on the input facet by a Kurt. J. Lesker electron beam assisted evaporator as discussed in Chapter 3. The metal layer covering the waveguides forming the figure ‘$\frac{\lambda}{N}$’ was selectively milled by a focused ion beam (FIB) assisted milling technique using a FEI Quanta 200 3D Dual beam system as shown in Figure 5-40.
A continuous wave Helium-Neon laser at 3.39 µm, and a carbon-dioxide laser at 10.64 µm were used to uniformly illuminate the near-field $\frac{\lambda}{N}$ pattern. Two gold mirrors (M1, M2) are iteratively adjusted to align the laser beam along the desired direction. An anti-reflection coated Silicon lens (L) with a focal length of 25.4 mm was used for focusing the laser beam onto the $\frac{\lambda}{N}$ pattern.
The fiberscope (F) was mounted on an XYZ translation stage to ensure that the \( \frac{\lambda}{N} \) pattern on the input facet of the fiberscope is positioned at the focal plane of the Silicon lens. A 52x reflection objective (Ealing) with 0.65 NA was used for collecting the magnified image transmitted through the fiberscope. The magnified image is focused onto a broadband pyroelectric camera (Electrophysics PV 320). A thermal power head (Laserprobe Inc. RK 575, RK5720) is used to measure the output power. Under uniform illumination, each of the pixels in the pattern \( \frac{\lambda}{N} \) at the input was magnified 3.5 times and found to be transmitted to the output plane without appreciable inter-pixel cross talk. The intensity patterns recorded on the pyroelectric camera at both 3.39 µm and 10.64 µm are in Figure 5-41 (a-d) and (e-f) respectively.

Figure 5-43: Numerical simulation (COMSOL) of the intensity pattern transmitted through the fabricated fiberscope
The transmitted image at 10.64 µm is not well resolved due to the limitation of the reflecting objective used. The output diameter of each individual pixel is only ~ 9 µm whereas the point spread function of the reflecting objective was measured to be ~ 2λ (20 µm). The variation in the intensity profile of each of the exposed waveguide/pixel can be attributed to the variation in germanium core diameters as shown in Figure 5-39. The uniformity can be improved by further optimization of the processing conditions. In addition, a numerical simulation based on finite element modeling of the proposed design under localized illumination of the pixels forming the pattern \( \frac{\lambda}{N} \) is illustrated in Figure 5-42. It can be clearly observed from numerical simulations that the inter pixel cross-talk is minimal in the proposed design and < 2%. From experiments, the cross talk is measured to be ~ 11%.

In summary, we have demonstrated the proof-of-concept of infrared imaging at sub-wavelength resolution using a fiberscope. A near field image with a resolution of less than 3 µm is transmitted with an in-built magnification of 3.5X at a wavelength of 3.39 µm. The resolution can be further improved by tuning the parameters for aggressively tapering the MOF template followed by a similar approach in fabrication. The fiberscope can be used for various imaging applications anywhere in the broad transmission window of Germanium between 2 – 15 µm. The design can be extended for imaging in the visible and near infrared regime by appropriate choice of materials for the waveguide core such as Zinc Selenide and Silicon respectively. The primary advantage of this imaging technique is the high information throughput (parallel process) with sub-wavelength
resolution. The fiberscope can also be extended to interesting applications such as optical
data storage and near-field photolithography.
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Chapter 6

Research summary and future directions

6.1 Summary of research work

- A magnifying (pixel magnification-3.5X) fiberscope with an array of tapered low-loss infrared (Ge core, ZnSe cladding) waveguides with sub-wavelength (3 µm) resolution at a wavelength of 3.39µm
- A magnifying (pixel magnification-3X) fiberscope with an array of tapered Ge waveguides with sub-wavelength (5 µm) resolution at a wavelength of 10.64 µm
- A magnifying (pixel magnification-3.5X) fiberscope with an array of tapered Si waveguides with a resolution of 2.5 µm at a wavelength of 1.55 µm was numerically designed, fabricated and characterized.
- Optical characterization of low loss amorphous hydrogenated Silicon (3dB/cm) and Zinc Selenide (<1dB/cm) waveguides in a silica fiber.
- Optical characterization of polycrystalline silicon, amorphous silicon, germanium and silicon nitride waveguides in a silica capillary fiber.
- Structural and optical characterization of annular semiconductor junctions inside a silica fiber for optoelectronic applications
- Numerical design of hollow cylindrical waveguides for transmission at 10.6 um
- Two dimensional dynamic focusing of the laser light by ferroelectric domain based electro-optic lenses
• M x N optical switching based on laser beam steering and focusing elements in ferroelectrics
• Gaussian to rectangular, elliptical and circular laser beam shaping by patterned ferroelectric domains

6.2 Future directions

The proof-of-principle of an infrared fiberscope with sub-wavelength resolution has been demonstrated in this thesis work. Basic essential features such as inter-pixel isolation, magnification and near-field image transfer characteristics have been demonstrated at three unique wavelengths. In-vivo imaging of biological samples and thermal imaging of integrated chips could be performed using the fabricated arrays. The proposed design could be pushed further to have an improved resolution comparable to that offered by the contemporary near-field imaging systems and reduced cross-talk. A highly absorbing metal or silicon carbide layer could be introduced between the core and the silica matrix to improve the isolation between the pixels. An array of semiconductor junctions could be fabricated in each of the pores of the MOF and the fiberscope could be used for imaging applications in the visible regime. The single tapered waveguides of germanium, silicon and Ge-ZnSe which have been fabricated in order to determine the deposition parameters can be used as the scanning tip for near-field scanning optical microscopy (NSOM) in the infrared regime. Preliminary NSOM measurements could be performed on standard metallic slits to determine the best resolution achievable using these probes. The single tapered waveguide could also be used for adiabatic mode
conversion. In addition to these, the germanium waveguides could also be used for modulation in the mid infrared regime by free-carrier induced absorption.
Appendix A

Two dimensional dynamic focusing of laser light by ferroelectric based electro-optic domain lenses

The current trend in optical data storage technology is towards improving data storage density and increasing data read/write rate. The ability to dynamically focus the laser beam has led to multi-layered storage systems resulting in higher storage density. Dynamic focusing in current technology has been accomplished by means of liquid crystals and electro-mechanical devices \(^1,2\). Such devices have a modest frequency response in the order of KHz. Therefore, aimed at improving the bandwidth, micro-optical devices based on ferroelectric materials like integrated high power electro-optic lenses and cascaded large-angle scanners \(^3\text{-}^8\) have been demonstrated earlier. The intrinsic electro-optic response of these ferroelectric materials enable high speed (GHz) operation, limited in practice by only the voltage requirements and the power supply. However the dynamic performance of these devices is limited to one dimension. The effectiveness of these devices in increasing data storage density could be improved further if their dynamic performance can be extended to two dimensions. In this letter, we successfully demonstrate the proof-of-concept of dynamic focusing of a laser beam along two dimensions with electro-optic lenses fabricated in ferroelectric lithium tantalate.

Lithium tantalate (LiTaO\(_3\)) is a uniaxial ferroelectric with 3m point group symmetry. It has two possible domain orientations with the spontaneous polarization (\(P_s\)) pointing along \(\pm Z\) axes. The direction of \(P_s\) in these domains can be switched by application of an external coercive field. Additionally, the application of an external field along the c-axis (\(E_3\)) can be used to create an electric-field tunable refractive index (\(\Delta n\))
across the domain wall, given by $\Delta n = n_e^3 r_{33} |E_3|$, where $n_e$ is the extraordinary index and $r_{33}$ is the corresponding electro-optic coefficient. Thus, by creating lens shaped ferroelectric domains, the variation in refractive index across the domain wall can be utilized to create an electro-optic lens with tunable optical power. In addition, power scaling can be achieved if a stack of several such lenses is fabricated in the device.

The optical power of a stack of cylindrical lenses is defined by

$$D = \frac{n_e^3 r_{33} V N}{t R}$$

(1)

where $N$ is the number of individual lenses, $R$ is the radius of curvature of the cylindrical surface, $V$ is the applied voltage and $t$ is the thickness of the crystal. Therefore, according to expression (1), the device can act either like a converging or diverging lens depending on the direction of the applied electric field. Figure 1(a) shows the simulation of the focusing performance of an electro-optic lens stack obtained by means of the beam propagation method $^{12}$. The lens stack is made up of 10 cylindrical domains with a radius of curvature of 407 $\mu$m. The spacing between each cylindrical lens is 10 $\mu$m. Other parameters used in the simulation were $V = 5kV$, $N = 10$, $t = 300 \mu$m, $n_e = 2.1806$, $r_{33} = 30.5 \text{ pm/V}$ $^{11}$. As observed, the beam diameter along the X axis can be modulated by placing a device along the X-Z plane. Similarly, if the device is placed along the Y-Z plane, the beam diameter along the Y axis can be modulated. As a result, if two devices are positioned at mutually orthogonal directions along the X-Z and Y-Z planes, we can dynamically focus the incident laser beam along both X and Y axes simultaneously.
Figure A-1: (a) Simulation showing the performance of an electro-optic lens stack obtained using the beam propagation method. (b) Images of lens shaped ferroelectric domains in LiTaO$_3$ obtained with polarized light microscopy. The spacing between individual lenses is 10 µm. (c) Schematic representation of the device testing experimental set up.
In order to demonstrate this concept, lens structures similar to Figure 1(a) were fabricated on commercially available z-cut LiTaO$_3$ wafers with thickness of 300 µm. To start with, the crystal was diced into rectangles of 11 mm x 7 mm. A tantalum electrode pattern similar to Figure 1(a) was lithographically defined. The lens-shaped domains were created by using the well established domain reversal techniques. An image of the domain pattern obtained using polarized light microscopy is shown in Figure 1(b). The devices were annealed to undo the effect of the internal field across the domain wall. Tantalum electrodes were deposited on +z and –z surfaces of the crystal to provide a pair of parallel plates in order to obtain uniform electric field across the thickness of the crystal. The input and output edges were polished to optical grade (rms value of 0.02 µm) to avoid losses due to reflection. An insulating layer of photoresist was spin coated on the +z and –z surfaces and baked to inhibit surface charge conduction. The contact to the electrodes was established with copper tape and the device was packaged using insulating silicone glue and rubber. Two such packaged devices were mounted on homemade device holders primarily designed to reduce the overall size of the assembly. The packaged devices were mounted on 3-D translation and rotational stages to ensure the following (a) input beam is parallel to, and passing through the centre of the cylindrical lens stack, (b) laser beam is not clipping the top and bottom surfaces of the crystal, and (c) the polarization of the incident laser beam is parallel to the spontaneous polarization in the crystal.

A Helium-Neon laser at 633 nm was used for testing the devices as shown in Fig. 1(c). Two high voltage power supplies V1 and V2 were employed to apply a uniform and stable electric field across first (D1) and second device (D2) respectively. While applying
the external field, precautionary measures were taken to not exceed the coercive field of LiTaO$_3$ (21 kV /mm) which could destroy the domain pattern in the crystals. In addition, a polarizer (P) and a half wave plate (W) were used to ensure that the polarization of the input beam was along the extraordinary axis of the crystal in both devices. The laser beam was confined within the thickness of the crystal in both first and the second device. This was achieved by using spherical lenses with focal lengths of 100 mm and 50 mm before device 1 and device 2 respectively. The focal length and position of both the lenses chosen were based on ABCD Matrix Theory for Gaussian beam propagation. The intensity profiles of the output beam were recorded by placing a CCD camera (COHU 4812) beyond the exit face of D2.

Figure A-2: Images of the far field spatial distribution of the output Gaussian beam at different applied voltages with both devices working simultaneously.
In order to highlight the combined performance of both devices, a representative sample of the far field spatial distribution of the incident beam as a function of the applied voltage on D1 and D2 is shown in Fig.2. As clearly observed, both horizontal and vertical diameters of the input beam can be modulated by varying the applied voltage on D1 and D2 respectively. The output beam diameter \((\omega_x, \omega_y)\) is found to vary between \((973 \, \mu\text{m}, 1039 \, \mu\text{m})\) and \((312 \, \mu\text{m}, 432 \, \mu\text{m})\) from \(-5\text{kV}\) to \(+5\text{kV}\) at an image plane 93 mm away from the exit face of D2. As expected, output beam diameters increase when the devices act as diverging lenses and decrease when they act as converging lenses. Furthermore, the output beam is found to maintain a Gaussian profile.

Figure A-3: Beam radius as a function of the propagation distance obtained at different values of the applied voltage. (a) For device #1. (b) For device #2. Solid lines correspond to the values predicted by ABCD analysis for Gaussian beam propagation.
In order to characterize the focusing properties of the device, the variation of intensity profile as a function of applied voltage was recorded at a different image planes. From these profiles, the Gaussian beam waist ($\omega$) was extracted and plotted as a function of propagation distance ($z$), at a fixed voltage as shown in Fig.s 3 (a) and (b). The focusing and defocusing effect of both devices can be clearly observed at positive and negative voltages respectively. In addition, theoretical analysis of this optical system based on the ABCD Matrix theory for Gaussian beam propagation was performed\textsuperscript{10, 11}. Each lens-stack was approximated to behave like a thin lens due to the small $\Delta n$ values. These theoretical predictions agree with the experimental values as shown in Fig.s 3(a) and 3(b). Thus, tunable optical power ranging continuously from: 129 m\textsuperscript{-1} to -129 m\textsuperscript{-1} can be obtained in two dimensions. The frequency response of the device was recorded at 20 kHz with applied voltage sinusoidally varying between +1kV and -1kV, is shown in Figure 4.

![Figure A-4](image.png)

Figure A-4: The performance of the device operated with a sinusoidally varying applied voltage between +1kV and -1kV at 20 kHz
A photodiode was employed to monitor the intensity of the laser beam at the output. The intensity variation of the laser beam results from the focusing and defocusing effect of the electro-optic lens with varying applied voltage. Nevertheless, the maximum modulation speed achievable is in the order of GHz and is chiefly limited by the time constant of the power supply in use.

In summary, we have demonstrated the concept of 2-D dynamic focusing accomplished by adopting an orthogonal arrangement of two electro-optic lenses fabricated on LiTaO$_3$ based on domain microengineering. The primary advantage of this device is the high frequency response and a possible extension of this concept to perform 2-D scanning. For commercial applications, the high values of the applied field could be reduced by decreasing the thickness of the crystal, decreasing the aperture of the cylindrical domain lenses, or by using materials with high electro-optic coefficients.
References

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Appendix B

Design and simulation of planar electro-optic switches in ferroelectrics

In optical communications, optical switches facilitate selective switching of the optical signal from M number of incoming fibers to N number of outgoing fibers. The primary desirable characteristics of an ideal optical switch are speed (kHz-GHz) and scalability (large number of input/output ports per switch). Past and current switch technologies include inkjet technology\(^1\), liquid crystals\(^2\), microelectromechanical systems (MEMS)\(^3,4\), photochromic effect \(^5\), and electro-optic effect \(^6,7\). Of these, the electro-optic mechanism promises the fastest switching speeds (up to \(10^{11}\) Hz), and is used as the preferred technology today for 10-40 GHz Lithium Niobate (LiNbO\(_3\)) based optical modulators \(^8\). Further advantages include small size, low operating power, and non-mechanical operation. The ferroelectric domain walls in LiNbO\(_3\) provide additional advantages when shaped as optical components, including high-speed switches, dynamic focusing lenses, laser beam steering elements, beam shaping, and integration of multiple such functions on the same chip have been demonstrated by us earlier \(^9-14\). Total internal reflection (TIR) at a ferroelectric domain wall has been used to demonstrate high-speed optical switches; however the operation is limited to 1 x 2 using a single domain wall, or 2 x 2 using a pair of walls, and device size scales with the number of ports.

In this letter, we present two alternate MXN electro-optic switch designs. These devices are designed using a combination of electro-optic prisms and lenses based on ferroelectrics lithium tantalate or niobate. **Design I** involves only electro-optic beam steering elements, and is a variant similar to the TIR switches described above, but
involves beam steering instead of reflection. **Design II** uses a combination of electro-optic steering and electro-optic focusing elements, which leads to significant advantages.

- **Figure 1**: (a) Numerical Simulation of the performance of the horn-shaped scanner employed in the optical switch design using the Beam Propagation Method at 1.55 µm. (b) Schematic of a 3 x 3 optical switch based on electro-optic beam steering elements. (c) Numerical simulation of the performance of the 3 x 3 optical switch using Beam propagation method at 1.55 µm.
We consider lithium tantalate as an illustrative material for our design calculations. In congruent lithium tantalate (LiTaO$_3$), the direction of spontaneous polarization ($P_s$) can be reoriented between equilibrium directions ($\pm z$ axes) by applying an external coercive field (21 kV/mm) along the $z$ direction. This phenomenon can be exploited to define an arbitrary domain pattern in the material by well-established domain reversal techniques. Furthermore, when the material is subjected to an external electric field ($E_z$) along the $z$ axis, there is an associated creation of a change in the refractive index ($2\Delta n$) across the domain wall, given by $\Delta n = -0.5* n_e^3 r_{33} E_z$, where $n_e$ is the extraordinary refractive index and $r_{33}$ is the corresponding electro-optic coefficient. This index change has been used by shaping ferroelectric domains as prisms and lenses for laser beam steering and focusing respectively $^9,^{14}$.

The first design is a 3 x 3 switch, whose schematic is illustrated in Fig. 1(a). It is composed of input (A, B, C) and output (P, Q, R) rows of electro-optic steering domain elements with corresponding rows of collimating ion-exchanged domain lenses $^{15}$. The shape optimized horn-shaped scanner (Reference) is used as the laser beam steering element in the design. The incident laser beam from the input fiber is butt-coupled into the device. The diverging incident beam is collimated along the $x$-axis by the passive double-ion-exchanged cylindrical lenses $^{16}$ at the input of each port. The beam is confined in the thickness direction using a planar waveguide formed by the standard annealed proton-exchanged or Ti-diffused waveguides $^{15}$, mode matched in dimensions to standard telecom fibers. The light is deflected by the first set of horn-shaped scanners (A/B/C) and is collected by the second set (P/Q/R) of scanners that re-straighten the path of the beam along the optic axis of the device. The light from the output ports is focused into the
output fiber by another stack of ion-exchanged cylindrical lenses in front of each output fiber as shown in Fig 1(a). Each of these scanners in the first and second column can be independently operated by depositing metal electrode on the ± z surface. Thus, the input beam from any of the three input ports can be switched to any of the three output ports (9 combinations) by varying the voltage across the scanners. The performance of this optical switch at 1.55 µm is simulated using the BPM. The performance of a horn-shaped scanner used in the design is numerically simulated using the Beam Propagation Method (BPM) as shown in Fig 1 (b). The material constants for LiTaO₃ used in the BPM simulation were $n_e = 2.1224$, $r_{33} = 27.4 \text{ pm/V}$ at 1.55 µm. The input aperture of the horn shaped scanner is 264 µm, the output aperture is 793 µm, and the device length is 10 mm. The wavelength of the laser beam is 1.55 µm and has a Gaussian beam diameter of 200 µm at the input aperture of the scanner. The distance between the end of the input port and input aperture of the output port is 28.5 mm. The total length and width of this optical switch are 48.5 mm and 5 mm respectively. All the nine possible combinations in a 3 x 3 switch are numerically simulated using the BPM and superimposed as shown in Fig. 1(c). The external electric field required on the input port and the output port ($E_{\text{input}}$, $E_{\text{output}}$) to deflect the incident beam from $A \rightarrow P$ is (0,0 V/µm), $A \rightarrow Q$ is (-3.3,3.3 V/µm), $A \rightarrow R$ is (-6.6,6.6 V/µm), $B \rightarrow P$ is (3.3,-3.3 V/µm), $B \rightarrow Q$ is (0,0 V/µm), $B \rightarrow R$ is (-3.3, 3.3 V/µm), $C \rightarrow P$ is (6.6,-6.6 V/µm), $C \rightarrow Q$ is (3.3,-3.3 V/µm) and $C \rightarrow R$ is (0,0 V/µm). To reduce the actual voltage requirements to less than 100 volts, thin slices (10-30 µm) from the pre-engineered domain pattern could be prepared by precision polishing or crystal ion slicing. More scanner ports can be appended to the input or output port to realize any M x N optical switch.
A key limitation of this design is its scalability, defined as $M$, the maximum
number of output ports, given by \( M = 1 + \frac{\theta_{\text{max}}}{\theta_{\text{div}}} \), where \( \theta_{\text{max}} \) is the maximum total deflection angle, and \( \theta_{\text{div}} \) is the divergence of the laser beam. Increasing \( M \) requires increasing \( \theta_{\text{max}} \) and decreasing \( \theta_{\text{div}} \). For increasing \( \theta_{\text{max}} \), horn shaped scanners are employed instead of rectangular scanners. The wider end of each horn shaped scanner (~0.79 mm here), limits the number of input ports that can be added adjacently. It also increases the deflection angle, hence the voltage required to address the neighboring scanners. The second limitation of this design is the divergence of the laser beam propagating from the input to the output ports within the plane of the device(x-y). Even if the focal point of the laser beam is at the center of the device, it will naturally diverge in the crystal plane, and will need to be accounted for in designing the passive ion-exchange lenses.

The schematic of a second design is shown in Fig. 2(a), for a 1 x N switch, which has significantly improved scalability, as well as alleviates the beam divergence issue. This design can be considered as half of an M x N switch, as described later. The design is a combination of dynamic only one (or two) electro-optic prisms and one (or two) set of electro-optic lenses. We exploit the fact that if an incident laser beam is offset from the principal axis of a lens, it is deflected towards the focal point.

Now let us look at the working principle of the 1 x N design. First, the angular position of the incident beam is controlled using a horn shaped scanner. Secondly, a lens re-deflects the laser beam and makes it parallel to the optic axis at the output port. This ensures that the coupling and insertion losses are minimized, as well as close packing of output ports is achieved. An additional key advantage of this design is that the lenses also compensate for the natural divergence of the laser beam inside the crystal and ensure that
the output beam diameter (225µm) is comparable to the input beam diameter (200µm). Thus, it plays a significant role in increasing the number of output ports.

The optical power of an electro-optic lens is defined as $D = \frac{\Delta n}{R}$, where $R$ is the radius of curvature of the lens. Due to the small values of $\Delta n$ ($\sim 10^{-4}$), the optical power of an electro-optic lens is lower in comparison to the conventional lens. Therefore, in order to increase the net optical power, a stack of 23 lenses with a radius of curvature of 3000 µm each, and separated by 5 µm gaps are cascaded in sequence as shown in Fig 2(a). The external field ($E_L$) applied across the stack of lenses, is kept constant in this particular design. The input aperture of the 8mm long scanner is 250 µm. The external field ($E_S$) applied across the horn shaped scanner is varied to deflect the beam to the corresponding output port. The performance of the switch is numerically simulated using the Beam Propagation Method at the following combinations of $[(E_S, E_L):$ Port A: (16, 16.6 V/µm); Port B: (8.3, 16.6 V/µm), Port C: (0, 16.6 V/µm), Port D: (-8.3, 16.6 V/µm), Port E: (16, 16.6 V/µm)] and superimposed as shown in Fig. 2(b). The device is 85 mm long and 10 mm wide. Based on the diameter of the output beam (~225 micron), the maximum number of resolvable spots/output ports that can be accommodated in this design is 4 per millimeter. The maximum number of output ports in the proposed design can be further increased by using a cascaded beam deflector, with an associated increase in the device size and complexity. The concept used in this design could also be extended to realize a quasi M x N switch shown in Fig 2 (c) where an M x1 and a 1 x N scanners can be combined on a single ferroelectric chip to give rise to an M x N switch. The proposed design is also comparable with the present-day technology\textsuperscript{18}, where the typical
spacing between adjacent optical fibers in a fiber optic array is 150 micron which would be able to accommodate a maximum of 6 ports per millimeter.

In summary, we have presented two designs for M x N optical switching using a combination of electro-optic prisms and lenses fabricated on a ferroelectric chip. The primary advantages of the M x N switch are high scalability, i.e. large number of output ports, high-speed electro-optic operation, design space over a wide wavelength range of transparency of the ferroelectric (0.35 – 4.5 µm), small size, low cost, and ease of fabrication. These advantages make it an ideal candidate for high speed optical switching applications over a wide wavelength range.
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Appendix C

Electro-Optic Laser Beam Shaping by Patterned Ferroelectric domains

Laser technology finds a wide range of applications such as photolithography, optical data storage, material processing, drilling, spectroscopy, printing and medical surgery \(^1,^2\). In many such applications, uniform power distribution of the laser beam over a specific region of interest is essential for ideal performance. However, the fundamental transverse electromagnetic (TEM\(_{00}\)) mode of the laser beam in most commercially available laser systems has a Gaussian intensity distribution which is not optimal for these applications. In order to circumvent this problem, various methods to transform the Gaussian intensity profile into any desired beam profile have been developed \(^3^-^6\). These methods based on geometric optics and diffraction theory are employed to determine the phase function of a phase modulating element (PME). The PME is used to modulate the phase of the incident beam in order to achieve the desired intensity distribution. Nevertheless, realization of the intricate phase function in a real optical system is a complicated problem involving multiple optical elements. It could be simplified further by utilizing the electro-optic effect in ferroelectric materials for the creation of the phase function. Recently, various electro-optic devices have been fabricated on ferroelectrics for laser beam steering, focusing and switching applications \(^7^-^9\). In this letter, we demonstrate the proof of concept of laser beam shaping using a compact phase modulating element fabricated on ferroelectric lithium tantalate.

Lithium tantalate (LiTaO\(_3\)) is a uniaxial ferroelectric crystal having two possible domain orientations, in which the spontaneous polarization is pointing along the
crystallographic +Z or -Z axes. The direction of spontaneous polarization in these domains can be reversed from a +Z to –Z state by the application of an external coercive electric field (~ 21 kV/mm) along the –Z axis (or vice versa). As a consequence, an 180° domain wall parallel to the Z-axis is formed at the boundary of the two domain states. This phenomenon can be utilized to define any arbitrary domain pattern in lithium tantalate by adopting well-established domain poling techniques\textsuperscript{10}. Furthermore, the linear electro-optic effect in ferroelectric materials can be favorably exploited to modulate the phase of the incident wavefront. For instance, a domain wall shaped in the form of a convex lens can be used to modulate the phase of the wave-front such that the laser beam is converging or diverging depending on the sign of the $E_z$. Hence, by calculating the required phase function and fabricating the corresponding domain wall pattern on lithium tantalate, a Gaussian intensity profile can be transformed to any (rectangular, circular, elliptical, etc) desired intensity profile.

In order to demonstrate this concept, we computed the phase function required to transform a Gaussian intensity profile to a flat-top profile. We used the well-established Gerchberg-Saxton (GS) and modified forms of GS algorithms\textsuperscript{1,2} for iteratively extracting the required phase function. The iterative scheme was implemented using Matlab\textsuperscript{®} 7.0.4 and optimized by the normalized mean square error and flatness of the intensity profile generated at the target plane. The phase function, $\varphi(x, y)$ required for transforming the laser beam at 633 nm wavelength ($\lambda$) from a Gaussian profile to a flat-top profile was computed. The corresponding ferroelectric domain pattern illustrated in Fig. 1 was calculated from the following analytical relationship:
\[ \Phi(w) = \frac{2\pi}{\lambda} \left[ n_e L + \frac{1}{2} n_e^3 r_{33} E_z (L - 2d(w)) \right] \]

Where ‘L’ is the length of the phase modulating device (10 mm), ‘w’ is the width of the PME fixed as 20 mm, d (w) is the function describing the domain wall pattern as a function of the width of the PME, E_z (1.45 kV/mm, calculated from simulation) is the external field strength required for achieving the required phase function. The material parameters of lithium tantalate used to determine the required domain pattern are \( n_e = 2.1809 \) and \( r_{33} = 30.5 \text{ pm/V} \).
To fabricate the proposed device, the domain wall pattern calculated from the phase function shown in Fig. 1 was replicated on commercially available z-cut lithium tantalate wafers with a thickness of 500 µm. An electrode pattern resembling required domain pattern as shown in Fig. 1 was defined on the +Z surface by photolithography. Fig. 2 (a)-(d) illustrates various regions of the photo-mask used during lithography.
The domain wall pattern was fabricated by employing well established electric field assisted domain poling techniques. In order to reveal the fabricated domain wall pattern, the samples were etched in a 1:2 solution of 49% hydrofluoric acid and 70% nitric acid for 24 hours at room temperature. The differential etching rate of the $+Z$ and $-Z$ domain result in an appreciable contrast at the domain wall. Various regions of the fabricated domain wall pattern were observed by cross polarized light microscopy. Fig.2 (e -h) are the polarized light micrographs of the fabricated domain wall pattern corresponding to Fig. 2 (a - d) on the photo-mask. It can be clearly observed that the fabricated domain wall pattern is similar to the simulated phase mask design. There are a few defects in the pattern which originate during fabrication and one of these is illustrated in Fig. 2(d, h). In order to facilitate real-time observation of nucleation and growth of the domains under an optical microscope, hard tap water (not de-ionized water which does not work) was used as an electrode on the top ($-Z$) surface. A uniform external coercive field (~ 21kV/mm) was applied across the sample to fabricate the domain pattern. The samples were wrapped in aluminum foil and annealed at 175 ° C for 12 hours. Annealing helps in relieving the stress and internal field across the freshly formed domain wall pattern. A uniform layer of tantalum (1 µm thickness) was sputtered over the 10 mm x 20 mm region on both the $+Z$ and $-Z$ surfaces. They act as a set of parallel electrodes required while testing the device. The input and output facets of the device were polished to optical grade using 0.02 µm colloidal silica. This reduces the optical insertion loss and prevents distortion of the laser beam due to surface scattering. After polishing, the device was packaged for testing.
A commercially available Helium-Neon laser at 633 nm wavelength was used to test the performance of the device. The schematic of the experimental set-up used is illustrated in Fig. 3.

The collimated laser beam is aligned to be parallel the crystallographic Z-axis (shown in Fig. 3) by iteratively adjusting the position of the two mirrors. A polarizer is utilized to align the laser polarization along the Z-axis. The device holder is mounted on a 3-axis rotation + 3 dimension translation stage to ensure the optical alignment of laser polarization along the spontaneous polarization of the ferroelectric domains in the device. This configuration maximizes the change in refractive index due to the linear electro-optic effect in lithium tantalate. A cylindrical lens with a focal length of 250 mm is used to couple the laser beam into the device. The focal length of the lens is chosen such that the waist of the focused laser beam lies at the centre of the device. The laser beam is confined within the thickness along the entire length to avoid any clipping or reflections from the top and bottom surfaces of the device. The spatial positioning and focal length...
of the required lens are calculated by modeling the optical system with ABCD theory for Gaussian beam propagation. The intensity profile of the laser beam exiting the output facet of the device is recorded in the far-field with a camera (COHU 4812).

\[ \text{Intensity (arb units)} \]

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<tr>
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<td>10</td>
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\[ \text{Intensity (arb units)} \]

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\[
\text{camera without flat-top profile.}
\]
g to design
The linear and angular position of the device is fine tuned until we obtain a Gaussian intensity profile of the outgoing beam on the camera. A Trek® high voltage power supply was used to apply a uniform external electric field required for converting the Gaussian into a flat-top profile. As per the simulations, external field strength of 1.4 kV/mm required for conversion is applied across the device. The conversion of the Gaussian intensity profile to a flat-top intensity profile under the application of external field is clearly observed as illustrated in Fig.4 (a). The ideal performance of the device computed using numerical simulations is shown in Fig. 4 (b). The minor variation between the experimental results and numerical simulation may be due to the presence of micro-domains and defects along the domain wall pattern as shown in Fig. 2 (d, h), and the power dissipation of the laser beam associated with reflection and scattering at the domain wall.

In summary, we have demonstrated the proof of concept of laser beam shaping from Gaussian to flat-top profiles by using compact ferroelectric domain wall patterns as the phase modulating element. The same device can be operated over a wide range of wavelengths in the transparency regime of lithium tantalate (0.35 – 4.5 µm) by calculating the external field strength required to induce the required phase shift at the desired wavelength. Similar concept can be adopted to achieve any desired output intensity profile including elliptical, circular, triangular, etc by calculating and fabricating the corresponding domain wall pattern required for conversion. Since any single pattern has a small footprint, many such patterns can be arranged on a single chip and operated in sequence to achieve multiple beam shapes from the same device. The idea can also be extended to perform laser beam shaping in two dimensions by means of two similar
devices placed in an orthogonal configuration. Additionally, the intrinsic electro-optic response (~ GHz) of ferroelectric materials promises the possibility of laser beam shaping at high speeds. The voltage requirements can be substantially lowered by decreasing the thickness of the device and also by using ferroelectric materials with higher electro-optic coefficients. In view of the aforementioned advantages, the proposed concept can greatly simplify the process of realizing the intricate phase functions required for laser beam shaping.
References


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